Metallurgy
Materials Engineering

THE INDIAN INSTITUTE OF METALS

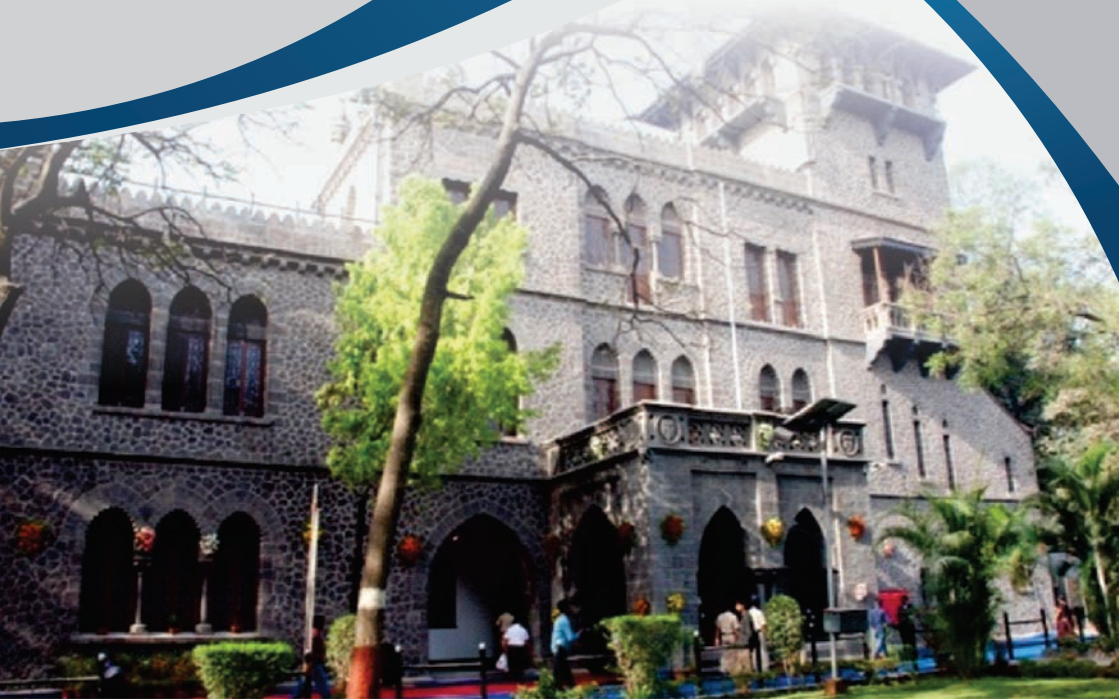
NMD ATM 2014

Redefining the Horizons of Metallurgy/Materials
Focus on Automotive, Aerospace, Defense and Energy

Book of Abstracts

52nd National Metallurgists' Day
(Under the aegis of Ministry of Steel, Government of India)
and
68th Annual Technical Meeting of
The Indian Institute of Metals

12-15 November, 2014
College of Engineering, Pune



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Message from the Technical Committee

The IIM-NMD ATM 2014 technical committee was focused on quality of the sessions and high coverage with respect to topics, presenters and delegates. The team has creatively structured the conference with 30 parallel theme symposia as well as four thematic panel discussions. We have received a tremendous response to our conference this year: more than 850 abstracts have been submitted for our annual technical meetings. We would like to attribute this to the charm of Pune City, the cultural capital of Maharashtra and the hard work put in by the organizing team.

We are co-organizing few theme symposia on various topics this year with other important professional bodies such as TMS, Electron Microscopy Society of India, Powder Metallurgy Association of India, Sponge Iron Manufacturers Association, and Society for Failure Analysis and Sheet Metal Forming Research Association. This will bring synergy and strengthen the quality of these sessions.

We have introduced several new industry-specific Theme Symposia at this year's Annual Technical Meeting. This includes Additive Manufacturing, IIM-TMS's Symposium on Industrial Realization of ICME, IT-enabled Manufacturing, Predictive Data Analytics for Manufacturing, Materials for Automotive Industries, Energy Sectors, Strategic Sectors such as Defense, Space and Nuclear and many others. We hope that this will enhance industry participation and will further the partnership between industries and the Indian Institute of Metals.

While we have added new theme symposia this year, we have paid equal attention to our mainstream themes such as mineral processing, iron and steelmaking, mechanical behavior of materials, and so on. We are organizing Nijhawan Memorial Symposium on Mineral Processing this year, in honor of Dr. B. R. Nijhawan, the first Indian director of CSIR-NML and former President of Indian Institute of Metals.

The technical committee has recommended introducing the technical posters in the main technical session in the form of short oral presentation, just before the respective poster sessions. We are pleased to present the technical program for IIM-NMD ATM 2014 consisting of 5 Plenary Lectures, more than 165Invited Lectures, 365 Contributory Oral Presentations, 432 Short Oral Presentations-cum- Posters.

We would like to acknowledge the contributions of Dr. Gerald Tennyson and Mr. Rishabh Shukla in compiling this "Book of Abstracts" and Dr. Venkat Runkana, Dr. Beena Rai, Dr. Ajay Karokoti and Dr. Satya Kuchibhatla for their assistance in editing and proof-reading at a short notice. We are grateful to all our Conveners for organizing excellent technical sessions and for their time, support and participation. We are looking forward to exciting technical sessions for learning and sharing at the 160 years heritage campus of College of Engineering Pune.

Dr. Amarendra Singh & Dr. Satyam S. Sahay

Co-Chair Technical Committee, IIM-NMD ATM 2014

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Bamane, Sushant

TECHNICAL PROGRAM

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PLENARY LECTURES

Nov 12, 2014: [11:30 - 12:30]

Titanium: Science, Technology and Application

Dipankar Banerjee, Indian Institute of Science, Bangalore

With some of the largest ore reserves in the world found on the beach sands of India, titanium has the potential to becoming a truly Indian metal. We briefly outline the basic framework of the metallurgy of titanium alloys and then describe the application of this understanding in the extraction, processing and manufacturing of titanium alloys with a focus on the indigenous capability built over several decades by the strategic sectors and academia.

Materials challenges for the Indian Atomic Energy programme

S. Banerjee, Bhabha Atomic Research Centre, Mumbai

The success of the Indian nuclear programme has been strongly linked with the development of indegeneous technologies related to nuclear materials- fuels, structural materials and their processing and characterisation techniques.India can proudly claim to be one of the very few nations who have the mastery of the entire fuel cycle. The first part of the presntation will address some of the important materials development issues which had significant impacts. At the present juncture when India has embarked upon a large scale growth of the nuclear programme involving heavy water reactors, light water reactors, fast reactors, high temperature reactors and thorium fuelled reactors fresh challenges are being posed in developing new materials, improving their performances and extending the lives of components. Some specific topics to be discussed are in-reactor creep of the pressure tube material, low alloy steels for reactor pressure vessels, metallic fuels for fast reactors with short doubling time, oxide dispersed stainless steels with higher tolerance for radiation damage, ceramic and refractory materials for high temperature reactors and corrosion resistant material for molten salt reactors.Separation and materials issues connected with nuclear waste management will also be covered.

Nov 13, 2014: [11:30 - 12:30]

The Digital Era: Driving Transformations in Minerals, Metals, Materials, and Manufacturing

K Ananth Krishnan, Tata Consultancy Services, Chennai

Globally, the digital revolution has impacted businesses on an unprecedented scale. The digital five forces ofMobility and Pervasive Computing, Big Data and Analytics, Social Media, Cloud Computing, and Artificial Intelligence (AI) and Robotics are coming together to changing processes in every industry. In this talk, I will highlight the power of digital technologies in the materials and metallurgy space, both from research and industry perspectives. We will look at trends and challenges in this sector and highlight how IT can help businesses look at new materials, speed time to market, reduce risk, drive efficiencies and enhance

sustainability. I will also provide a vision for the ecosystem that is required to accelerate progress in this space.

Industrial Realization of ICME: Status and Opportunities

Richard LeSar, Department of Materials Science and Engineering, Iowa State University, Ames, IA 50011 USA

Integrated Computational Materials Engineering (ICME) has become an accepted part of materials engineering in the past decade, with regular conferences (e.g., the biennial TMS-sponsored World Congress on ICME) and many new projects from around the world (e.g., the ICMEg project in Europe). There has also been an increasing interest expressed by industry in adopting ICME since the 2008 report from the US National Academy of Engineering. Finally, the notion that ICME should become a regular part of the materials curriculum has achieved widespread acceptance, although perhaps with less agreement on what should be taught. The goal of this talk is to give a snapshot of current activities in ICME, with a focus on the industry. We will also discuss impediments to ICME, mainly from computational and application perspectives. Our focus will be on answering a series of questions, including what is needed for a wider application of ICME in industry and what kind of educational and computational resources will be required.

Nov 15, 2014: [10:30 - 11:00]

New Steels Development - A Changing Game

Debashish Bhattacharjee, Tata Steel, Europe

This presentation shows that conventional way of steel development through chemistry and tensile strength properties is inadequate to address modern application needs. Current trend in development of steels shows the following key aspects:

(i) Multiple properties are simultaneous developed to address specific applications. Eg, automotive components needing simultaneously, high strength, good bendability, weldability, hole expansion, corrosion resistance and paint appearance.

(ii) Development of functional surfaces is integral part of steel development. Eg; Steels for facades with integral photovoltaic coatings is essentially development of steels for construction.

(iii) Development of new processing technology both at the steel manufacturer and the user is inseparable part of new product development.

ADVANCED COMPOSITES

Invited Talks

Session 1: Nov 13, 2014: [09.00 -11.00]

Design and development of Al-Cu-Fe based quasicrystals and composites

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The bulk quasicrystalline (QC) materials and their composites have attracted considerable interest due to their promising mechanical properties. The stable Al-Cu-Fe based icosahedral quasicrystal (IQC) phase and composites have been produced by conventional casting and rapid solidification techniques. Spray atomisation and deposition process has been used to synthesise bulk single phase icosahedral quasicrystals based on $Al_{62}Cu_{25.5}Fe_{12.5}$ system as well as quaternary system containing Sn and other elements and their composites. The elemental materials were iznduction melted under nitrogen cover and a billet of 250 mm in diameter and 350 mm in height was spray formed. The x-ray diffraction analysis of over-sprayed powder particles from Al-Fe-Cu alloy showed the presence of θ-Al2Cu, β-AlFe(Cu) and λ-Al13Fe4, in addition to IQC. It was interesting to point out that due to Sn addition the volume fraction of β-phase was found to increase. Single phase quasicrystalline particles were used to synthesise Al-based IQC composites by mechanical alloying in high energy ball milling. The alloyed powder were compacted and sintered to develop the compact product. The particles of quasicrystals were found to disperse in the Al matrix. The strength of the composites was found to increase to a greater amount but the ductility was reduced with the volume percentage as high as 20%. However there was an optimum level of the amount where the strength can be increased with reasonable ductility. A model considering the combined strengthening contributions consisting of load bearing, dislocation strengthening and matrix ligament size effects are used to describe the strength of the Al based metal matrix composites containing different amounts of quasicrystalline phases. Attempts will be made to discuss the various models in the context of quasicrystalline reinforcements as well as other intermetallics on mechanical properties of the composite materials.

Session 2: Nov 13, 2014: [13.30 -15.30]

Flexible ceramic filled PTFE laminates for microwave circuit applications

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Flexible microwave substrates are extensively used for variety of high end microwave circuit applications such as high power solid state amplifiers, missile guidance, satellite communication etc. In the commercial wireless communication sector also, access to wireless high speed data is quickly becoming a necessity in multiple markets like mobile networks, aerospace etc. Increased densities, increased functionality and

overall cost reduction need to be addressed and hence there is a perceived need to develop novel high frequency printed circuit boards. Dielectric properties of packaging materials significantly influence the performance of high-speed microwave devices. High dielectric constant reduces the circuit size since the wavelength traveling through the medium is inversely proportional to the square root of dielectric constant whereas extremely low loss tangent improves the signal integrity and avoids cross talks. Cu-cladded ceramic filled PTFE planar laminates are the ideal choice for high end microwave circuit fabrication. Understanding the need for indigenous technology, C-MET has developed a proprietary process methodology comprising of Sigma Mixing, Extrusion, Calendering followed by Hot pressing (SMECH process) to fabricate dimensionally stable, planar and isotropic PTFE/ceramic composite laminates. Silane coating has been done on particulate ceramic fillers to preclude moisture absorption. Waveguide cavity perturbation technique has been employed to accurately determine the dielectric constant and loss tangent of PTFE laminates in the X-band region. C-MET has successfully developed wide variety of filled PTFE substrates with dielectric constant ranging from 2.2 to 15.2 together with ultra low loss tangent (0.0018 at X-band), which can be commercially exploited for wireless communication applications. Pilot plant production facility has been established at C-MET to produce these strategically important materials. Variety of microwave devices such as high power solid state amplifiers, band pass filters, miniaturized patch antennas, Wilkinson Power dividers etc. have been fabricated using indigenously developed microwave substrates and extensive reliability tests were performed to ascertain their efficacy for practical applications.

Session 3: Nov 13, 2014: [15.30 -17.30]

Aluminum matrix micro- and nano-composites by powder metallurgy

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Among metal matrix composites, discontinuouslyreinforcedaluminum matrix composites (AMCs) account for the largest share. Liquid metallurgy (LM) processing techniques like stir casting andsqueeze casting have been a dominating the fabrication of AMCs .But powder metallurgy (P/M) route offersthe following advantages over LM: (i) Minimization of undesirable interfacial reactions due to lower processing temperatures (ii) Fine ceramic particles (<5ìm) can be easily dispersed uniformly in a refined microstructure (iii) Wettability of the reinforcement is unimportant. (iv) Near net shaped parts resulting in greater utilization of materials. The main challenges in P/M of AMCs are: To disrupt the Al_2O_3 film on Al which acts as a sintering barrier and (ii) to reduce porosity in the composite product, because the presence of the ceramic particles hinders densification.

The presence of liquid phase during sintering helps in densification.Further, the presences of Mg in the matrix powder mix and a N_2 sintering atmosphere have been known to disrupt Al_2O_3 film on Al particles. Keeping this in mind, a precipitation hardenable Al matrix containing 5.0 Cu and 1.0-1.5Mg (wt.%) was chosen in the present work. Two types of reinforcements were incorporated: (a) micro-SiC_p (~5ìm) and (b) nano-Si_3N_4 (~35 nm).

(Al-5Cu,1Mg)-SiC_p micro-composites**:** The aim was to determine the extent to which precipitation hardening and sliding wear resistance against steel counter-surface can be influenced by introducing the mechanical alloying (MA) step to refine the microstructure. It has been found that : (i) In comparison to

manual blending(HM) of the powder mix ,MA step in P/M processing accelerated ageing kinetics at 150°C and resulted inhigher peak hardness(maximum HB =201 kg/mm^2). (ii) The improvement in the wear resistance due to MA processing over HM has been found to be 2.2 for the matrix alloy and 4.5 for the 10% SiC_p composite in the peak-aged (PA) condition. (iii)The effect of SiC_p in reducing the coefficient of thermal expansion was more pronounced for MA processed composites.(Al-5Cu,1.5Mg)-Si_3N_4nano-composites**:** In this system, MA was successfully employed to achieve ultrafine and uniform dispersion of Si_3N_4nanoparticles (up to7.48vol.%) resulting in a remarkable improvement in properties viz.:(i) Ageing response of the composites at 150°C showed that thepeak ageing time reduced from 12 to 9 h and the peak hardness increased from 129 to 236HB(kg/mm^2) as the Si_3N_4 was increased from 0 to7.48vol.% . (ii) The PA composite containing 7.48 vol.% Si_3N_4 exhibited6.2 and 10.5 times higher wear resistance as compared to that of the matrix and wrought AA6061 alloy respectively.In both the composite systems,the presence of $CuAl_2$,Al_2MgCu,Al_2O_3 and AlN has been detected by XRD in the over-aged condition.

Contributory papers

Session 1: Nov 13, 2014: [09.00 -11.00]

Preparation of pure Ni-coated Al_2O_3 composite powders by electroless plating

Jayakumar Eravelly*, P. Pavan Kumar, Vishwanatha H M, Sudipto Ghosh
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Ceramic particles are a good choice of reinforcement to develop metal based composite materials owing to its high strength to weight ratio, corrosion resistance and wear resistance properties for automotive and aerospace applications. However, poor wettability of ceramic particles with the metal matrix is the hindrance for attaining better mechanical properties in Metal Matrix Composites (MMCs). To overcome this problem, continuous and uniform core shell Al2O3/pure Ni composite powders as precursors for MMCs were synthesized via electro less plating technique. The coating process was carried out in four steps: Soak cleaning of Al2O3 powder by acetone, sensitization by acidic SnCl2 solution, activation in acidic PdCl2 solution and finally deposition of nickel films on the activated Al2O3. Deposition was carried out using alkaline bath containing nickel sulphate (NiSO4.6H2O) as source of nickel, hydrazine hydrate ((N2H4.H2O) as reducing agent. At the end of each step, thorough cleaning with distilled water and drying of the powder was carried out. During the process, temperature and pH of the bath were maintained in the range 92°C-95˚C and 11-12pH respectively. Chemical composition of coated particles was investigated using Energy Dispersive Spectrometer (EDS) and X-ray diffraction (XRD) techniques. Further, uniformity of coating was studied using Field Emission Scanning Electron Microscope (FESEM). The results indicated that concentration variation of bath components significantly influences chemical composition of coated particles and uniformity of the coating. A complete core-shell structure of Al2O3/Ni has been achieved using this method.

Microstructure and tensile behaviour of Al-Mg/Al_3Zr in-situ composites

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The aluminium matrix composites (AMCs) with varying volume fractions (10-15%) of Al_3Zr particulates were fabricated via direct melt reaction (DMR) between Al- Mg alloy and inorganic salt K_2ZrF_6 at a temperature of 850°C.The composites were characterized by XRD, OM, SEM with EDS, TEM and tensile testing. The results indicate the successful in-situ formation of Al_3Zr particulates in the Al-Mg matrix. The Al_3Zr particulates were found in polyhedron and rectangular shapes. Grain refinement of aluminium-rich grains was also observed which could have resulted due to the presence of Al_3Zr particulates. The tensile tests were conducted at room temperature to evaluate the tensile properties. The tensile properties of composites were observed to improve continuously with Al_3Zr volume in a significant way as compare to base alloy and correlated with the microstructure. However, ductility decreases with increasing volume percent of Al_3Zr but while comparing with matrix alloy, ductility has improved.

Development of mixed ionic and electronic conductor based Ni-$La_{2-x}Ca_xZr_2O_{7-\delta}$ membrane for hydrogen separation

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Global warming has increased the demand for development of fossil fuel power plants with carbon capture and storage. An attractive alternative to conventional power generation processes is the pre-combustion CO_2 capture/separation option, where pure H_2 is separated from a mixture of H_2 and CO_2 before final combustion. It is important to identify potential high-temperature proton conductors possessing both high permeation property and good chemical stability in a concentrated CO_2 environment. The alkaline earth metals doped lanthanum zirconatepyrochlore have been investigated over past few years for variety of applications, especially as membrane. It is reported that $La_2Zr_2O_7$ has excellent thermal stability, low chemical resistance and low sintering rate. In the present work, attempt has been made to obtain an optimized Ca-doped lanthanum zirconate proton conductor and develop a hydrogen separation membrane using nickel and the optimized proton conductor. Single phase $La_{2-x}Ca_xZr_2O_{7-\delta}$, $0.3 \leq x \leq 0.7$, [LCZ1 (x=0.3), LCZ2 (x=0.5), LCZ3 (x=0.7)] compositions have been synthesized by combustion method. The electrical conductivity of the sintered specimens have been studied in air, and both moist and dry reducing atmosphere (5% H_2 in Ar) in the temperature range between 550°C and 850°C. Maximum electrical conductivity obtained at 850°C is 0.013 S/cm for $La_{1.95}Ca_{0.05}Zr_2O_{7-\delta}$ under moist reducing atmosphere. The overall electrical conductivity of the cermet material having 40 vol % Ni and 60 vol % $La_{1.95}Ca_{0.05}Zr_2O_{7-\delta}$ and sintered at 1400°C (99.7% density) is ~ 400 S/cm at 750°C in moist mixture gas atmosphere. Highest H_2 permeation flux of 3.52×10^{-4} ml/cm^2/s is achieved at a pressure difference of 2 bar between feed and permeate side with H_2 and N_2 flow rate of 500 sccm each at 800°C.

Fabrication of TiO_2 dispersed hydroxyapatite nano composite

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The paper explore TiO_2 dispersed Hydroxyapatite nano composite fabrication by conventional and microwave method of sintering at 1400 ^{o}C and then characterization using XRD, SEM, and TEM. Hydroxyapatite based Nano composites were prepared by dispersion of Titania (TiO_2) particles by milling and were studied in comparison with coarse particle reinforced composites. Mechanical mixing is a noble way to homogenize dispersion. The high rate of heating provided by the usage of microwave sintering enables faster, more efficient and better sintering. The Titania nanoparticles reinforced composite showed much higher hardness at lower concentrations of filler materials (5 % TiO_2 nanoparticles in HA and 5 % coarse TiO_2 particles in HA.) and both the composites were considerably harder than the pure HA hardness (obtained from literature). This fact can be attributed to the suppression of dislocation movement due to the reinforcements and also to the better sintering due to the enhanced diffusivity of nanoparticles .The result of paper shows that Microwave route is more beneficial fabrication route over conventional for developing Nano TiO_2 dispersed Hap Composite.

Microstructure of Ni_3Al particulate reinforced $AlSi_{10}Mg$ matrix composites prepared by direct metal laser sintering

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Metal matrix composites are becoming promising materials for aerospace, automotive and defense sectors as they possess superior mechanical properties. However, conventional processing routes such as powder metallurgy, casting etc. are not able to meet the demand of cost effective intricate components. Additive manufacturing (AM) technology is found to be an efficient route to produce metal matrix composites of such components. Direct Metal Laser Sintering (DMLS) is a laser based AM technology, gaining widespread popularity in manufacturing industry due to its ability to fabricate complex shaped 3D metal and multifunctional composite tools and functional end products directly from 3D CAD models. In this paper, the effect of laser power, scan speed on microstructure and densification of the direct metal laser sintered Ni_3Al particulate reinforced AlSi10Mg composites were investigated. Test samples were produced by EOS M270 DMLS machine equipped with 200W Yb-fiber laser, operated under argon atmosphere. Microstructure studies were conducted using optical and electron Microscopy. Composites produced using the processing parameters of laser power ≥170 W and scan speed ≥1000 mm/sec resulted uniform dispersion of Ni_3Al particles and less than 10% porosity. High rate of cooling resulted formation of heat affected zone (HAZ) around the melt pool and fine dendritic microstructure inside the melt pool.

Utilization of waste plastic and tyres as composite sleeper

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Economic growth, increasing population, urbanization and changing consumption resulting into rapid increase in generation of waste plastic and tyres in the world. Waste plastic are non-biodegradable, non-eco friendly and causes air and water pollution. Waste tyres causes increase in cost of raw material, resource constraints, air pollution associated with open burning. The present paper reviews the utilization of waste tyres and plastics in the formation of environmental friendly composite sleepers. Research work is in progress for development and industrial level fabrication of these sleepers. Composite sleepers can be used as an alternative railroad sleeper that is superior in performance, environmentally responsible, and provides a significant value to rail customers. It has high strength having a life of 40 to 50 years, low weight, high corrosion resistance, fire resistant, low thermal and electrical conductivity, non magnetic properties, good attenuation of sound and vibration, excellent design flexibility, low life cycle cost, recyclable and reuse of waste resources. This paper reviews the manufacturing process used by TieTek Company and comparison with traditional sleepers. The results achieved in lab tests and field trials in India have been discussed in the paper.Survey, test results and performance of the sleeper abroad as well as in India revels that composite sleepers may be an alternative for traditional sleepers. The use of composite sleepers in India can make a huge impact on the rail industry.

Thermal shock effect on hydrothermally immersed GFRP composites

A. P. Chakraverty[1*], U. K. Mohanty[1], B. B. Biswal[2] and S. C. Mishra[1]
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The degradation of layered GFRP composite immersed in distilled water is investigated at length. The composite samples are immersed in distilled water for 120 days in steps of 20 days each and exposed to both up and down-thermal shocks. The ingress of moisture increased with increased period of immersion and the inter-laminar shear strength (ILSS) values are more and more adversely affected with the increase in the immersion period. The samples with longest immersion and with exposure to up-thermal shock showed the worst degradation in the way of exhibiting the least glass transition temperature, the T_g. This may be due to the combined effect of greater moisture ingression and up-thermal shock responsible for enhancing the process of plasticization of the polymer matrix.

In-situ intermetallic Al_3Ti particles reinforced aluminium alloy composite

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In this study, in-situ Titanium-tri-aluminide (Al3Ti) particles were formed by the reaction of potassium hexafluorotitanate (K2TiF6) with 6061 aluminium alloy and ultrasonication was done to know its effect on the morphology of Al3Ti particles and its dispersion into the matrix. Melting experiments were performed by employing three different conditions. These were (1) K2TiF6 addition, (2) K2TiF6 addition accompanied with ultrasonic stirring and (3) control specimen (without ultrasonic stirring or K2TiF6 addition). Microstructure and phase identifications were done by SEM and X-ray diffraction techniques (XRD) respectively. Hardness, ultimate tensile strength, proof stress and strain to failure comparisons were made between the samples casted at different conditions. The properties achieved were explained in correlation to the microstructure. The ultrasonically treated specimens showed microstructural modification in terms of the grain structure and particulate dispersion which is the reason for the improved properties.

Evaluation of erosion characteristics of glass-epoxy composites filled with red mud particles using Taguchi technique

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Red mud emerges as the major waste material during production of alumina from bauxite and enormous efforts have been directed worldwide towards red mud management issues i.e. of utilization, storage and disposal. Its potential as a filler material in polymeric matrices has not yet been reported and in view of this an attempt is made to explore the possibility of making a class of erosion resistant polymer composites with the red mud particles as the filler. Composites of different compositions (epoxy resin – glass fiber – red mud) are prepared and solid particle erosion wear trials are conducted following a well planned experimental schedule based on Taguchi's design of experiments. This systematic experimentation has led to determination of significant process parameters and material variables that predominantly influence the wear rate of the particulate filled composites reinforced with glass fiber. Factors like filler content, impact velocity and impingement angle are the significant factors affecting the erosion wear rate. It is further seen that the use of Taguchi model with the prescribed design strategy is quite effective for the parametric appraisal of the wear process within a well defined experimental domain.

Studies on combined effect of contact and non contact type ultrasonic casting of Al-Al_2O_3nanocomposite

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In the past few decades, aerospace and automobile industries are looking for new age materials consisting of high strength to weight ratio property. This has made the materials R&D to focus on composite materials as these materials exhibit excellent mechanical properties and can also be tailored easily. Along with the reduced weight, the few other desired contents in structural applications include low cost, quality and high performance. Aluminum based composites are playing vital role in meeting this demand. Especially aluminum based nanocomposites are of great interest. The key benefits of nanocomposites include isotropic properties and significant strength and yet low weight. The biggest challenge in nanocomposite is to achieve uniform distribution of nanoparticles in the matrix and lot of research is going on in processing of nanocomposites. Amongst the various synthesis techniques ultrasonic casting is drawing more attention especially in the case of bulk nanocomposites. Cavitation based ultrasonic casting is found to be efficient in distribution of nanoparticles and the refining of microstructure. The present work involves synthesis of Al-Al_2O_3nanocomposite through combined method of contact and non contact type of ultrasonic casting. The samples exhibit significant increase in hardness with increase in volume fraction of Al_2O_3. Microstructure studies show nearly uniform distribution of nanoparticles throughout the casting.

Fabrication and characterization of high performance carbon nanotubes reinforced aluminum composites through spark plasma sintering

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Carbon nanotubes metal matrix composites are being extensively studied since they have potential applications in many industries such as aerospace and automobile. In this work, arc discharge synthesized and purified carbon nanotubes (CNTs) were characterized using X-Ray diffraction (XRD), Transmission electron microscopy (TEM) and Raman spectroscopy analysis. Purified CNTs are coated with copper by electroless method for deposition time of 60 min at 60 °C and characterized using TEM. Different amounts copper coated CNTs and Al powders were ball milled for 1 h to prepare the composites. Copper coated CNTs reinforced Al composites were prepared by spark plasma sintering (SPS) under different pressures at 550 °C for 5 min sintering time. The prepared composites are characterized using XRD, SEM, Raman spectroscopy, and TEM. TEM pf carbon nanotubes shows that the impurities less in case of purified CNTs compared to as synthesized CNTs. TEM of copper coated CNTs shows the uniform deposition of copper was achieved on carbon nanotubes. It is observed from XRD of composites that there was no aluminum carbide formation. TEM images of ball milled powders and composites revealed the uniform distribution of CNTs in the matrix.

Effect of PMMA on photoluminescent behavior of lanthanide doped hybrid strontium phosphate phosphor

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Recently, lanthanide doped luminescent hybrid phosphors have cropped up as promising material in comprehensive opto-electronic devices and solid state lighting applications. Taking this into consideration, strontium phosphate phosphor doped with rare earth lanthanide ions, Eu^{3+} and Dy^{3+}alongwith PMMA as capping agent have been fabricated using ingenious combustion method. In this method, urea is incorporated as reducer and fuel. The surface morphology and structure of the phosphor powder is characterized by employing X-ray diffraction and scanning electron microscopy whereas the characteristic luminescent properties are extrapolated using emission and excitation spectra. The average crystallite size derived from Debye Scherrer formula is found to be 59nm. In the proposed work, the effect of varying concentration of PMMA in lanthanide doped hybrid phosphor are focused since PMMA being a linear polymer has the capacity of forming quasi crossed linked structure through strong dipole-dipole interactions which strongly favors the photoluminescent activity in long lasting phosphors. The decay curves and lifetime calculations display a clear trend in the enhancement of brightness and afterglow property due to presence of PMMA in host phosphors which are subsequently ascribed to the presence of shallow and deep traps.

Hardness, wear and biocompatibility studies of UHMWPE based nanocomposites

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Ultrahigh molecular weight polyethylene (UHMWPE) is the most frequently used material for the manufacturing of bearing components of large joint replacements due to its excellent bio-compatibility and sufficient mechanical properties. However, the long-term service of UHMWPE and thus the lifespan of the whole joint replacement system are limited by two principal material-related reasons of UHMWPE failure, viz. wear and oxidative damage. In this research work, a preliminary effort has been made to improve the wear resistance of UHMWPE by reinforcing it with carbon nanotube and also with nano-crystalline metal chips. The biocompatibility of the composites was tested through cytotoxicity and genotoxicity studies. A remarkable improvement in the hardness and wear property was observed in the composites in both the cases.

Synthesis and characterization of graphene reinforced Cu base composites prepared by two step thermal processing methods

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Copper is one of the most suitable engineering materials for thermal and electrical application. The copper matrix has been successfully strengthened with graphite reinforcements - particles and discontinuous or continuous fibers. The system Cu/C shows good thermal, electrical and mechanical properties in comparison to other Cu-based metal matrix composites. In our present work, a two steps thermal processing technique has been used to synthesize Cu- Graphene composite. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques were employed to study the Microstructural architecture of the composites as a function of weight percentgraphene. Micro hardness measurements on the as prepared composites show significant increase in hardness with increase in weight percent of Graphene and also conductivity measurements shows variation in electrical conductivity with increase weight percent of graphene.

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Reaction sintering and mechanical properties of TiB_2 composites reinforced with carbon nanotubes and graphene nanoplatelets

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Transition metal borides such as TiB_2, ZrB_2, and carbides, nitrides having melting point of above 3000 ˚C are referred as ultrahigh temperature ceramics (UHTCs). They have low density, high elastic modulus (> 450GPa) and high hardness (> 25GPa). This group of materials generally show low electrical resistivity (10-30 $x10^{-6}$ Ω cm) and high thermal conductivity (60-120 $W.m^{-1}K^{-1}$). As a result, UHTCs are one of the candidates for applications such as thermal protection of re-entry vehicles, rocket nozzles, armour materials and crucibles for molten metals. However, full densification without cracks during sintering is difficult to achieve in case of UHTC powders due to the strong covalent bonding nature and presence of oxides on the surface. Oxidation and ablation resistance of these materials also needs to be improved for critical applications. This study reports the simultaneous synthesis and densification of TiB_2 with and without additions of CNTs and Graphene as reinforcements. Mechanically milled mixtures of Ti, B and CNT/Graphene are sintered using reaction spark plasma sintering (RSPS) at 1400 ˚C. Dense composites having ultrafine grain size with 1, 2, 4 and 6 vol. % CNTs and Graphene as reinforcements have been synthesized. XRD results confirmed the formation of TiB_2 with fewer amounts of TiB and TiC phase. Addition of CNTs reduced the sintering and reaction rate and acted as an effective grain growth inhibitor. Oxidation studies have been carried out at 1400 ˚C in air utilising thermo gravimetric analyser. The effect of CNTs and Graphene addition on the mechanical properties has been studied using microhardness and nanoindentation. Addition of CNTs to TiB_2 increased the indentation fracture toughness significantly.

Effect of in-situ developed ZrB_2 particles on microstructural and mechanical properties of AA5052 metal- matrix composites

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In the present work AA5052-x vol. % ZrB_2 [x = 0, 3, 6, 9 vol.%] in situ aluminium matrix composites(AMCs) have been prepared by in situ reaction of two inorganic salts K_2ZrF_6, KBF_4 and molten aluminium alloy AA5052 at 860°C. Phase evaluation, microstructural and mechanical properties of composites were characterized by XRD, OM, SEM, EDS TEM, density, hardness and tensile testing. XRD and EDS analysis confirm the existence of in situ formed ZrB_2 particles in alloy matrix. Microstructural examinations reveals grain refining tendency of ZrB_2 particles which were found mostly in hexagonal and few in rectangular morphology. Most of the ZrB_2 particles were of nanometres size up to 45 nm whereas very few of micron size upto 3.5 µm. Density of base alloy and composites was calculated by using the Archimedes principle and found to be increases with increased ZrB_2 content. Hardness (BHN) was observed to be increased with increased ZrB_2 content. Tensile properties of base alloy and composites were evaluated by tensile testing at room temperature. Mechanical properties were observed to be improved significantly in comparison to the base alloy. Hardness, ultimate tensile strength (UTS), 0.2% yield strength (YS), was improved by 41.17%, 83.14% and 100% respectively for composite having 9 vol. % ZrB_2 particles in comparison to base alloy. Ductility (% elongation) of composites was also improved in comparison to the base alloy. Fractured surface of the tensile tested specimens have been investigated under scanning electron microscope and correlated with the mechanical properties.

Effect of alumina spacers on the kinetics of TiC_x formation in situ and properties of reaction spark plasma sintered Ti-TiC_x-CNT composites

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Ti-TiC composites have potential applications due to their high wear resistance and high strength at high temperatures. In this study, Reactive Spark Plasma Sintering (SPS) has been used to synthesize Ti-TiC_x composites from 10hr ball milled Ti carbon nanotube (CNT) mixtures. Ball milling has been done to disperse the CNTs in Ti matrix and noticeable reaction of Ti with CNTs has been found. SPS was carried out at temperatures of 800 °C, 1000 °C and 1200 °C with and without alumina spacers between the punch and the powders. The alumina spacers (1.5 mm thick) act as an insulator and as a diffusion barrier and have significant effect on the diffusion of carbon from the punches. Densification behavior was observed by analyzing the displacement of the punches during SPS. The effect of alumina spacers on the phase evolution and properties of the composite was investigated. CHN analyser was used to measure the carbon content in the powders and the sintered compacts. Carbon measurements indicated an increase of 4 wt.% of carbon after 10 hours of milling due to the reaction of Ti with WC milling media and the milling jar as supported by thermodynamic predictions. Ti-5 wt.% CNT sintered without alumina spacer at 800 °C showed 100 % conversion of TiC_x whereas the same sintered with alumina spacers showed retention of Ti

in XRD pattern. Vicker's hardness values of the Ti-5 wt.% C compact sintered at 800 °C with and without alumina spacer was found to be 800 H_v and 1100 H_v respectively.

Mechanical modeling of ZnO interfacing with UHMWPE in ZnO-UHMWPE bicomposites

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Ultra high molecular weight polyethylene (UHMWPE) is well known material used for articular joint replacements but these implants generate wear debris during articulation against femoral head of hip prosthesis, and also due to infection caused by bacteria etc. So, in the current work, antibacterial Zinc oxide (ZnO) is reinforced in UHMWPE and their mechanical properties (tensile strength, yield strength, toughness, ductility and Young's modulus) are evaluated in two different ZnO morphologies viz. micro rods and nanoparticles in 5, 10, 15, 20 wt.%. ZnOmicrorods caused reduction in Young's modulus of ZnO-UHMWPE composite by 8.8% whereas ZnO nanoparticles caused an increase by 21.1%. This contrasting behavior of variation of Young's modulus is explained by introducing the effect of ZnO morphology in Halpin Tsai Equation and Rule of Mixture leading to modification in reinforcement geometrical factor ε of Halpin Tsai Equation for the two different ZnO morphologies.

Development and characterization of HAp dispersed Ti-based bulk metallic glass composite

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Among all the bulk metallic glass (BMG) systems, Ti-based BMG are regarded as good candidates for implant because of their high mechanical strength, low density and excellent corrosion resistance. As a candidate for implant, the biocompatibility of BMG should be considered. Hydroxyapatite (HAp), with similar composition and structure as those of human bone, has excellent biocompatibility. Moreover, HAp does not exhibit any cytotoxic effects and can directly bond to the bone. In this context, the Ti-based BMG dispersed with HAp can be considered as a potential candidate for implants. In the present investigation, $Ti_{60}B_{20}Si_{15}Zr_5$ was subjected tohigh-energy ball milling in a Fritsch Pulverisette P6 planetary mill at a rotational speed of 300 rpm with a ball-to-powder ratio of 10:1. Mechanical alloying has been done up to 30 hours. X- ray diffraction patterns of the 1 hour and 30 hours ball milled sample showed the intense sharp diffraction peak of exist elements and almost a hump corresponds to glassy matrix. The 30 hours ball

milled samples was further heat treated for 15 minutes at 900K to get crystalline phases. The XRD pattern of the ball milled powder after 30 hours was substantiating by transmission electron microscopic image. Ti-HAp and $Ti_{60}B_{20}Si_{15}Zr_5$-HAp powders with varying proportions of HAp *viz.,* 10, 20 and 30 wt. % were milled to obtained composite blends which were cold press at 500 MPa and sintered at 1100°C for 1 hour. XRD patterns of the compacted samples reveals presence of the dispersed phase (HAp).The microhardness measurement show increase in hardness values of the matrix with contents of HAp for $Ti_{60}B_{20}Si_{15}Zr_5$ as compared to Ti. The morphological features of the composite compacts were observed by scanning electron microscope. The compressive behaviour and nanoscratch measurements of the composites also show similar agreement with the microhardness values.

Development of new castable (Al-5Mg) alloy-TiB_2 metal-matrix composites

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There has been a constant drive for replacing monolithic aluminium alloys by aluminium based metal matrix composites (MMCs) in many industrial applications where high stiffness and strength to weight ratio are required. This work explains fabrication of a metal matrix composite where aluminium alloy is used as a matrix and ceramic particles as reinforcement. It also describes a process of Al-Alloy-10 wt.% TiB_2 MMCs on aspects relating to the formation of ceramic particles TiB_2 within the matrix by 'In-situ' technique. In 'In-situ' technique, the particles are formed by salt metal reaction i.e. Al-TiB_1 master alloy and KBF_4 salt. It involves the experiment of salt metal reaction, development of Al-Alloy-10wt.%TiB_2, mechanical characteristics, wear behavior of composite, microstructure, vibration damping behavior of composite, properties and applications. The present paper describes a cost effective route to produce Al alloy-10 wt % TiB_2 metal matrix composites by in-situ molten flux assisted reaction synthesisAluminium alloy is increasingly used as a matrix material in naval vessel manufacturing due to its unique combination of good corrosion resistance, low density, superior mechanical properties. With TiB_2 as particulate addition in Al – Alloy (Al-5Mg) matrix properties of alloy can greatly be improved. It increases the strength of aluminium. Also, such materials when machined with non-conventional methods will show better machinability. Al-Alloy-TiB_2 has been utilized in high tech structural and functional applications including aerospace, defense, automotive and thermal management areas as well as in sports and recreation. From industrial point of view, the (Al-5Mg) Alloy – 10wt.%TiB2 composite has an advantage of easy recycleability over conventional Al-alloy composites.

Structure and mechanical properties of discrete and functionally graded SS316-IN625 material prepared by Laser Engineered Net Shaping

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Functionally graded materials (FGMs) are a new class of advanced engineering materials in which composition, microstructure and hence properties are varied smoothly over macroscopic length scale for performance improvement. The family of laser freeform fabrication techniques are found to be promising

for the fabrication of FGMs. Laser Engineered Net Shaping (LENS) is one such technique wherein a high power laser beam is focused onto a suitable substrate creating a molten pool. In the present study, discrete and functionally graded materials of SS316 and IN625 alloys were prepared using the LENS process. The composition of individual layers was controlled by varying the feed rates of powders appropriately. In the case of discrete deposit, the composition is abruptly changed from SS316 to IN625 whereas in the graded deposit, the layer-wise composition is varied from 100% SS316 to 100% IN625 by increasing the concentration of IN625 by 12.5% in each successive layer. Interface of discrete and transition zone of graded deposits were characterized for porosity, microstructure, compositional variation and hardness.Fine cellular / dendritic microstructure was observed in both discrete and graded deposits. Elemental distribution in the transition zone of graded deposit matched very well with that of the computed values. The discretely deposited sample exhibited an interesting microstructural feature. Four distinct layers were seen (two each across the interface) when the composition of the layer is changed abruptly from 100% SS316 to 100% IN625. Each of these layers displayed a distinct composition with no variation in composition within each layer. Micro-hardness value across the transition zone was found to vary smoothly from SS316 to IN625 in the case of graded sample while discrete changes were observed in the hardness from one layer to another layer. An attempt has been made to correlate the difference in the microstructure, composition and microhardness between discrete and graded material and also explain their evolution based on diffusion of solutes in the molten condition of the alloy.

Dielectric behavior of precursor derived Si-Hf-C-N-O nanocomposites

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Hafnium (Hf) based materials are technologically important due to their attractive properties like high neutron absorbing capacity, high melting point and high chemical stability. Hence they find myriad applications in fields such as nuclear industries. Another striking property possessed by these materials is their high dielectric constant. High-k dielectric materials are considered to be a promising alternative for silicon oxide (SiO_2) in many devices and are becoming increasingly important in memory chips and gate dielectrics. These materials are effective in reducing the size of the capacitors in memory chips such that increased number of capacitors can be accommodated in a single chip to enhance its performance. Monolithic Hf based materials are tough to be processed due to limitations like relatively high processing temperature and pyrophoric nature. Also they are less abundant and relatively expensive. In this study ceramic nanocomposites containing hafnium based material has been processed using precursor derived ceramics route. Polysilazane was modified with varying percentage (0, 10, 20 and 30%) of hafnium alkoxide to produce Si-Hf-C-N-O. The modified polysilazane was cross-linked at 300 °C for 2 h followed by thermolysis at 1000 °C for 1 h. The as-thermolyzed Si-Hf-C-N-O was then crushed into powders and spark plasma sintered at 1400 °C for 30 minutes with a pressure of 30 MPa in vacuum. The sintered pellets were characterized using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) to determine the phases present and the microstructure was analyzed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Dielectric constant was determined using impedance analyzer to analyze the potential of the material to act as a capacitor.

Structure- property correlation of a novel 6351 Al-(Al_4SiC_4 + SiC) hybrid composite

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In the present research work 6351 Al-(Al_4SiC_4 + SiC) hybrid composite is developed through stir casting method where SiC (size: 25 ± 6 μm) and TiC (size: 430 ± 50 nm) particles are incorporated into the 6351 Al melt at 760°C with simultaneous stirring at 200 rpm for 30 min followed by casting in a standard cast iron mould. The addition of TiC results in the generation of in-situ Al_4SiC_4; while SiC remains as ex-situ reinforcement. Microstructure evolution in correlation to developed hardness and mechanical properties along with mode of tensile fracture has been studied in details. The experimental results envisage some interesting microstructural modifications (i.e. elimination of most of the dendrites, grain refinement etc) and nearly homogenous distribution of reinforcement particles without segregation so as to obtain excellent combination of properties. In general, both the reinforcements are found to act as the nucleation sites for primary α (causing extensive grain refinement) along with the evident engulfment effects promoting uniform particle distribution. Furthermore, the addition of TiC in 6351 Al melt results in the generation of Al_3Ti precipitates of nanometric size in the microstructure along with Al_4SiC_4 in-situ reinforcement particles. The effects of grain refinement, dominant presence of equiaxed α-grains (lower proportion of dendrites) and simultaneous reinforcement effects of hard SiC and Al_4SiC_4 particles along with the strengthening effect of Al_3Ti nano-precipitates eventually result in an improved combination of strength (UTS = 205 MPa) and ductility (%Elongation = 12) in Al-(Al_4SiC_4 + SiC) hybrid composite as compared to 6351 Al in the as cast condition (UTS = 119 MPa and %Elongation = 3).

Processing and characterization of SiC-B_4C-Al cermet prepared by pressureless sintering and spark plasma sintering method

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Substantial progress in research towards the upgradation of light weight, high temperature resistance and high performance materials. Presently, SiC-B_4C based cermets have drawn much interest in research and development towards their execution in several aero-engine components, high temperature cutting tools, heat exchangers, heat-engine parts, fusion reactors and armour plates. Boron carbide (B_4C) and silicon carbide (SiC) are considered as the best ceramic armour because of their low theoretical density, high temperature resistance and high hardness. Unfortunately, processing of B_4C and SiC is very difficult due to its high covalent bond, low plasticity and very low fracture toughness (2-4 MPa $m^{1/2}$), limiting its application as structural material. The aim of this study is the preparation of SiC-B_4C based cermets with proper densification and estimation of improved mechanical properties. The effect of Al (5, 10, 20 wt.%) addition on SiC and B_4C powder constituents have been studied. The powders were synthesized by high energy planetary milling followed by conventional sintering and spark plasma sintering (SPS) techniques separately. The as-milled powders were compacted into pellets in a hydraulic press followed by conventional sintering at 1950˚C for 0.5h in argon atmosphere. In another set of experiments, the as-

milled powders were loaded into a graphite die and SPS was carried out at 1100˚C at heating rate of 100°C /minute for 5 minutes under 50 MPa pressure. The structural and morphological changes and the phase evolution of the milled powder as well as sintered pellets were analyzed by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The physical properties and mechanical properties including density, hardness, flexural strength, compressive strength and fracture toughness of the cermets were studied and compared. SPS sintered SiC-B_4C–Al (10 wt. %) samples achieved around 97% theoretical density with a maximum micro-hardness of ~22 GPa; whereas, pressureless sintering technique yielded a micro-hardness of ~17 GPa with 93% of theoretical density for the same composition.

An overview on experimental characterization of abelmoschusesculentus oval chain structured fibre reinforced polyester composites

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Increase in awareness towards the sustainable development invites the attention of several researchers to manufacture green materials. Many authors had made several attempts in the characterization of various natural fibres under different conditions of extraction, processing. They were used to make composites and characterize at various conditions. N. Srinivasababu had introduced hybrid okra individual fibre and woven fibre (with slight manual modification of existing fibre) composites in 2009, 2010 itself. But a peculiar variety i.e. fibre in oval chain form in layers was first observed in this okra variety fibre in 2007. An attempt is made to outline the manufacturing and characterization aspects of okra oval chain structured fibre reinforced polyester composites as per ASTM procedures. Hand lay-up technique was adopted throughout the work for the manufacturing of the composites. Rolling process was adopted for better impregnation of the fibre in the matrix. Okra Oval FRP composites had shown maximum tensile strength and modulus of 35.82 MPa, 801.89 MPa respectively at maximum fibre volume fraction. The highest flexural strength of (94.5 MPa) and modulus (4.53 GPa) was achieved for the composites at 26.86 % okra oval fibre content. Charpy impact test on okra oval FRP composites revealed the impact resistance of 256.84 J/m at 23.19 % fibre volume fraction. The dielectric strength of the composites was decreased with increase in fibre content. Hence a designer will get an opportunity in selecting light weight high strength material. Hence this class of lightweight materials with good mechanical properties may be best suitable in manufacturing of the door panels of an automobile.

Synthesis and structural and optical characterization of variable ratio x bismuth ferrite-(1-x) nickel ferrite nanocomposite using chemical route

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The nanocomposite of x Bismuth ferrite- (1-x) Nickel ferrite is synthesized using chemical route where perovskite component comes from bismuth and ferric nitrate precursors, citric acid ethylene glycol while

spinel component using nickel and ferric nitrate by co-precipitation technique. Thermal analysis of mixed precursors helps in predicting crystallization temperature. Crystallization temperature obtained is about 426ºC for the nanocomposite system. The respective analyses of phases with respect to temperature, estimation of crystallite size, induced strain are determined from XRD studies. Micro-structural analyses, morphology of particles are estimated from FESEM studies and EDX gives the elemental compositions. FTIR analysis executes presence of vibration bonds required for the formation of the nanocomposite. Tauc plot is used to evaluate band gap of the synthesized materials to identified semi-conductive properties of synthesized materials. Band gap obtained are about 2.8eV, 2.5eV, 2.7eV and 2.6eV respectively for variable compositions and at different temperatures. PL spectra shows the photoluminescence characteristics of the synthesized sample after excitation at constant wavelength of 250nm having emissions at about 389.96nm, 385.9nm and 390.67nm respectively. This study revealed many interesting properties which can be used for optical and photocatalytic applications.

Synthesis and characterization of electroless copper coated carbon nanotube reinforced Cu-10%Ni alloy composites prepared by spark plasma sintering

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In this study, Carbon nanotubes (CNTs) are synthesized by arc discharge method and purified. Electroless copper (EC) coatings were performed on purified CNTs at pH of 7 at room temperature. Purified and coated CNTs were characterized using X-Ray diffraction (XRD), Transmission electron microscopy (TEM), and Raman spectroscopy. The matrix used was Cu-10%Ni alloy. The alloy was prepared by high energy ball milling (HEBM) of elemental powders (Cu, Ni, Fe, and Mn) in appropriate amounts for 2hr. Different weight fractions of copper coated CNTs were added as reinforcement in Cu-10% Ni alloy and ball milled for 1hr and characterized using XRD and TEM. Composites of ball milled powders were prepared by spark plasma sintering (SPS) at 950 °C for 10 min under 50 MPa sintering pressure. Sintered composites were characterized by XRD, scanning electron microscopy (SEM), and TEM. It is observed from Raman Spectroscopy that the graphitization is more in purified CNTs compared to as synthesized CNTs. XRD confirms the coatings of copper on CNTs and TEM revealed that the coating is uniform throughout the length of the CNTs. TEM of ball milled powders and composites shows that the distribution of CNTs are homogeneous in the matrix.

Wear behavior of Al-Al_3Ti in-situ metal matrix composites

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For structural application of moving components, the tribological properties are considered to be one of the major factors controlling the performance. In recent years, lightweight metal matrix composites (MMC) have received wider attention for their technological application, such as aerospace and automotive parts etc. In the present study, an attempt has been made to prepare and characterize Al-Al_3Ti

metal matrix composites with varying percentage of in-situ Al_3Ti (3, 5 and 7%). The composites were prepared by the reaction commercial purity aluminum 99.7% and K_2TiF_6 salt at a reaction temperature of 800 °C. The prepared samples were characterized by Scanning Electron Microscopy. The wear tests were conducted on all the prepared samples by varying parameters like wt. % of Al_3Ti particles, normal pressures, sliding speeds. Mechanical properties were assessed using computerized universal testing machine, Brinell hardness tester, Surface roughness tester and micro hardness tester. The worn surfaces were examined by optical microscopy after wear test. Al-3Ti, Al-5Ti and Al-7Ti alloys were prepared and effect of Ti content on hardness, tensile strength, volumetric wear rate and surface roughness were examined. Experimental alloys were fabricated by salt route method. Volumetric wear rate of the reinforced Al-3Ti, Al-5Ti and Al-7Ti alloys at room temperature were measured. The present results suggest that the wear resistance of Al-Al_3Ti composites increases with increase in percentage of Al_3Ti particles compared to pure aluminum. In addition, the improvement in mechanical properties of the composite was observed in Al-5Ti composite when compared to Al-3Ti and Al-7Ti and to the pure Al. Better tribological properties of these alloys can be achieved at Al-5Ti.

Effect of electric field on the synthesis of in-situ Al-$(TiB_2)_p$ composites

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The effect of applied electric field during liquid-solid transformation is long been a subject of research interest. The electron density is changed by the applied electric field during solidification of alloys. On the atomic scale, electron density plays an important role on the atomic structure; the changed electron density affects the kinetic energy of solute atoms and enhances the mobility in the liquid metal. It also leads to a change in the microstructure. However, little published information is available on the effect of electric field on the microstructural changes in composites specially in the in-situ particulate reinforced composites during the synthesis. The present study is aimed at to investigate the microstructural changes with electric field application in TiB_2 reinforced aluminum based composites and the resulting mechanical and dry sliding wear properties. The results show that with increase in current density the TiB_2 reinforcements are more refined and homogeneously distributed. The matrix is also refined. Significant improvement in hardness, wear resistance and tensile strengths are observed with increase in current densities.

Tribological behaviour of SiC based composites

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The composite approach is attractive in improving wear behaviour of ceramic materials. Dense SiC ceramics with 0, 10, 30 or 50 wt. % WC content are prepared by hot pressing method, and the effect of WC content on the unlubricated sliding wear behaviour of sintered SiC-WC composites at different loads: 5, 10 or 20 N against SiC balls is studied. The nano hardness of the sintered composites varied between 33 GPa

and 38 GPa with WC addition in SiC ceramics. A maximum fracture toughness of 6.7 MPa.m$^{1/2}$ is obtained when 50 wt% WC is added. The coefficient of friction varied between 0.4 and 0.5 when SiC-WC composites are slided. The wear volume of SiC ceramics increased with increase in load from 5 N to 20 N, whereas the specific wear rate decreased with increase in WC content. A maximum specific wear rate of 1 X 10^{-5} is observed for SiC ceramics whereas a minimum specific wear rate of 4 X 10^{-6} mm^3/ N-m obtained at 20 N load for SiC ceramics prepared with 50 wt% WC. Scanning electron microscope images of worn surfaces revealed SiC grain pull-out and fracture as major wear mechanism. The uniform dispersion of WC particles in SiC matrix is attributed to the increased fracture toughness, leading to an increase in fracture toughness. AFM results showed reduction in surface roughness of worn surfaces with increase in WC up to 30 wt% in SiC ceramics. The friction and wear results are discussed as function of grain size, WC inter-particle distance as well as hardness and fracture toughness

Session 3: Nov 13, 2014: [15.30 -17.30]

Study on polymer matrix micro- and nanocomposites for EMI shielding

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Polymer matrix composites containing micro and nano sized conductive particulates such as copper/nickel/stainless steel were studied for the electromagnetic interference (EMI) shielding applications. These composites were fabricated using hot pressing. It was found that the microhardness was increased significantly with increasing particulate content in the matrix. It was also investigated that the electrical conductivity of the composite depends upon the crystallinity of the polymer matrix and volume fraction of the particulate added to the matrix. For a given system, electrical conductivity of the hand mixed composites showed an increment of 13 orders of magnitude while those prepared by ball milling showed an increment of 2 orders of magnitude for the same volume %. This huge difference in the conductivity of composites prepared by different techniques is attributed to the dispersion of particulate in the matrix. Owing to better electrical properties, these micro- and nano-composites may be promising candidates for electronic applications like electrostatic dissipation (ESD) and electromagnetic interference (EMI) shielding.

Novel multistage spark plasma sintered HA-$CaTiO_3$ composites for biomedical application

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Hydroxyapatite (HA) is highly bioactive and biocompatibility. It is used as coating and bone filler materials due to their insufficient mechanical and electrical properties such as fracture toughness, electrical conductivity etc. In recent years, HA and calcium titanate ($CaTiO_3$) have gained much attention in the field of biomedical material researches. Calcium titanate (CT) is a good substrate for apatite growth and plays dominant role in enhancing osseointegration. To exploit the advantages of both materials, we used novel

multi-stage spark plasma sintering (MSSPS) technique to get HA-40wt% $CaTiO_3$ composite by varying sintering condition, in order to establish optimum processing parameters. In this study, an attempt was made to develop an understanding of the densification behavior, phase stability by X-ray Diffraction (XED) analysis, microstructure by Scanning Electron Microscopy (SEM) analysis, and mechanical property by Vickers Indentation and single edge V-notch beam (SEVNB) method. The results indicated that 99% of theoretical density could be achieved with uniform microstructure and without decomposition of HA into α and β- tricalciumphosphate ($Ca_3(PO_4)_2$), while fracture toughness was found to be 1.7 MPa m1/2. In conclusion, our findings suggest that HA-$CaTiO_3$ could be considered an important candidate as a biomaterial for medical applications.

A review on mechanical and wear behavior of Al-$(Mg_2Si)_p$ functionally graded in-situ composites synthesized by centrifugal casting method

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Functionally graded materials (FGMs) are a new class of advanced materials characterized by continuous, smooth variation in the microstructure and/or composition. As a result, sharp local stress concentration from one material to another can be avoided when two or more materials with distinct properties are required to be assembled together. Processing of FGMs through centrifugal casting route is one of the most effective methods. The centrifugal force causes to distribute the particles in liquid either towards the core or periphery of cylindrical components according to the relative densities of the particles. The FGMs are fabricated from in-situ particulate reinforced composites to achieve required properties either in the inner layer or outer layer of cylindrical components. Amongst the various particulate reinforcements Mg_2Si is highly attractive in an aluminum matrix as the Mg_2Si distribute in the inner layer due to its low density. Apart from these, desirable properties are high melting point, high hardness, low co-efficient of thermal expansion and a suitable Young's modulus. The present paper reviews the effect of compositions, process parameters, on the microstructural evolution, mechanical and wear properties of Al-$(Mg_2Si)_p$ FGMs.

Phase retention of YSZ in plasma sprayed YSZ-CNT reinforced Al_2O_3 matrix composites for thermal barrier coating

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Yttria stabilized zirconia (YSZ) is widely used as thermal barrier coating (TBCs) for gas turbine engines bladesdue to its low thermal conductivity (1.8-2.2W/mK) and comparable thermal expansion coefficient (~$7x10^{-6}K^{-1}$)with that of nickel substrate.But, the coating failure occurs mainly due to its poor fracture toughness and development of residual stresses. In the present research, $Al_2O_3$was blended with 0, 3 and 8 mol.% Y_2O_3doped zirconia and deposited on Inconel 718 alloy. Further, 4 vol.% multiwall-carbon nanotubes (CNTs)are reinforced to enhance the fracture toughness ofplasma sprayedcoatings.

Complimentary spark plasma sintering technique is also utilized to produce bulk YSZ-CNT composites. Phase retention has been analyzed using x-ray diffraction, transmission electron microscopy and Raman spectroscopy. The retentionof ~26% transformable tetragonal ZrO_2phase is believed to play a major role in imparting enhanced fracture toughness (by 28%, from ~4.3MPa.m$^{1/2}$ to ~5.4MPa.m$^{1/2}$), whereas, CNTs have shown to provide synergistic toughening (from ~5.2to ~5.9 MPa.m$^{1/2}$). Thus, synergistic toughening can render enhanced damage resistance for enhancing the life of thermal barrier coatings.

Wood plastic composite – challenges for processing and mechanical properties

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The science of wood plastic composites (WPC) is focused primarily on chemical interaction between the dissimilar materials. Now with the advent of the basic research into the processing and mechanical properties, wood waste and plastic (either virgin or recycled) has huge engineering potential for the forming of WPC. This is making our life safer by sequestering the carbon in it and is economical too.This research work investigates the relative ease in manufacturing technology of WPC produced by the use of low density polyethylene LDPE and wood waste. It also includes the investigation in mechanical properties i.e. tensile strength, compressive strength, modulus of elasticity, modulus of rupture of the derived product. Anhydrides were used as coupling agents to enhance the strength related properties. Glycerol was added to reduce the density and viscosity of low-density polyethylene for the better compounding and surface properties. Talc powder was added to absorb the moisture content that was left even after the pre-drying of wood dust. Different samples prepared on the basis of varying amount of wood dust percentage by keeping in mind about its encapsulation in the polymer matrix. Furthermore, it lists the detailed description about challenges in processing of polymer based wooden fibers reinforced composite andpossible steps to be taken to avert the unfavorable situations. At present there are very few manufacturers of WPC in India and range of WPC products is very small. Here the author proposes WPC products as a replacement on the basis of comparison of mechanical properties with wood, plastic and other composites and alloys.

A study on effect of $BaTiO_3$ on the electrical properties of polymeric nanocomposites

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Dielectric for embedded capacitors requires materials that possess high dielectric constant, low dielectric loss, low processing temperature, high breakdown voltage, and sufficient stability which are compatible with printed wiring board (PWB). To achieve these properties, polymer matrix nanocomposites based on polycarbonate (PC) as matrix and barium titanate ($BaTiO_3$ nanopowder as reinforcement were fabricated using the solution method followed by hot pressing. The content of $BaTiO_3$ was varied from 0 to 50 wt.%. The dielectric constant of the nanocomposites increased significantly with increasing $BaTiO_3$ content in the PC matrix. There was no dispersion in the dielectric constant of the nanocomposites with increasing frequency from 1 kHz to 15 MHz. SEM showed good dispersion of the $BaTiO_3$ in the PC matrix.

Preparation and properties of poly(phenylene sulfide)/clay nanocomposites

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In the present study, the modified clay (C-15A) was incorporated in high performance poly phenylene sulfide (PPS), using planetary ball mill followed by hot pressing technique. The clay content was varied from 0 to 10 wt%. Nanocomposites were characterized by scanning electron microscopy (SEM), Vickers hardness tester, and pin-on-disk wear tester. The experimental density was very close to that of theoretical density. SEM showed almost uniform dispersion of clay particles in the PPS matrix. Vickers microhardness was increased approximately 14 % at 2 wt% clay content. The specific wear rate of the PPS/clay nanocomposite with 1 wt% clay was found lowest. SEM images of the worn surface of pure PPS showed adhesive wear mechanism, whereas nanocomposites with 1 wt% and 5 wt% clay showed mild abrasive wear and sign of microploughing, respectively.

Polymer/cobalt composites for electronic applications

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Polymer matrix composites filled with metals are widely studied for the applications in electrostatic dissipation (ESD) and electromagnetic interference (EMI) shielding. In view of this, the electrical properties of the polymer matrix composites based on polymer as matrix and cobalt (Co) powder as reinforcement were studied. The composites were prepared using solution method followed by hot pressing. The Co content was varied from 0 to 70 wt% in the polymer matrix. Optical microscope showed good dispersion of the particles in the matrix. The electrical conductivity of the composites increased significantly. A percolation threshold was obtained at about 50 wt.%.

Age hardening behaviour of Al 2014-TiB_2 composite prepared by powder metallurgy route

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This work deals with a comparative study based on the age hardening behaviour of Al 2014 metal matrix composite reinforced with TiB_2 particulates in different volume percentage. Al 2014–$x$$TiB_2$ (x = 5, 10 and 15 vol%) ex situ composites have been successfully prepared by P/M route. All the samples were solutionised; quenched in air and aged in a Tubular furnace. Field emission scanning electron micrographs at low magnification show uniformly distributed Tib2 particles throughout the matrix at 5% volume. Field emission scanning electron micrographs at higher magnification shows the presence of uniformly distributed secondary phase in the alloy matrix indicating a strong continuous bond between TiB_2 particle and the alloy matrix. TiB_2 particles enhance the aging kinetics and the time to peak aging at the aging temperature of 160^0C comes down from 6 to 5 h when the TiB_2 amount is increased from 0 to 5%volume.

Preparation of Fe-aluminide reinforced in-situ aluminium matrix composites by two stem thermal processing

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Aluminium based metal matrix composites reinforced with in-situ Fi-aluminide and alumina particles were prepared by two step thermal processing a powder mix of aluminium and nanosized Fe_2O_3 powders. The reinforcements were formed in-situ by exothermal reaction between the Fe_2O_3 nano crystalline powder and aluminium. The thermal characteristics of the in-situ reaction were studied with the aid of differential scanning calorimetry (DSC). X-ray diffraction (XRD), energy dispersive spectroscopy (EDS) and scanning electron microscopy (SEM) techniques were employed to study the microstructural architecture of the composites as a function of final processing temperature and volume percent reinforcement. Micro hardness measurements on the as prepared in-situ aluminium matrix composites exhibit significant increase in hardness with increase in hot pressing temperature and volume fraction of reinforcement.

Synthesis and characterization of Ti-aluminide reinforced in situ metal matrix composites by spark plasma sintering

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Aluminium-based composite reinforced with in-situ Ti-Aluminide and alumina particles was prepared by spark plasma sintering between a powder mix of aluminium and nanosized TiO_2 particles. The reinforcements were formed in-situ by exothermal reaction between the TiO_2 nanoparticles and the host aluminium matrix. The thermal characteristics of the in situ reaction were studied with the aid of differential scanning calorimetry (DSC). Scanning Electron Microscopy (SEM) along with the Energy Dispersive Spectroscopy (EDS) and X-ray diffraction (XRD) techniques were employed to study the Microstructural architecture of the composites as a function of spark plasma sintering temperature and volume percent reinforcement. Micro hardness measurement on the as prepared in-situ aluminium matrix composites show significant increase in hardness with increase in hot pressing temperature and volume fraction of reinforcement.

Effect of cryogenic treatment on adhesion phenomenon at interface in wollastonite reinforced PBT composites

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In this paper investigations on the influence of cryogenic treatment especially on extent of adhesion between filler and the matrix interface of PBT/wollastonitecomposite with varied content of filler loadings 0(W0),5(W1),10(W2), 20(W3) & 30(W4) % by weight are presented. The selected material is treated at different temperatures (-80°C,-140°C &-185 °C) for stipulated time period (4, 8, 12,16,20,24 hrs) in the cryostat and then tested at ambient temperature. The properties of 'untreated' and 'cryo-treated' materials are evaluated in a comparative manner for their mechanical, structural, and thermal properties. Tensile strength exhibited a marginal increase on cryo-treatment whereas a dramatic increase was observed in modulus with an increase in filler loading. The experimental results for the moduli were also compared with theoretical predictions which revealed good level of interfacial interaction (by Pukanszky model for interaction parameter 'B'calculation) in the cryo-treated composites. It was indeed observed that lower loadings of fillers were effective in significantly decreasing friction coefficient and wear loss as compared to virgin PBT, but higher wt. % deteriorated the wear performance in case of untreated sample. Whereas for all studied composites, the relative improvement in wear performance on cryo-treatment is observed irrespective of wt. % loading as compared to untreated samples. SEM images of composite authenticate the weak and good adhesion for untreated and cryo-treated samples respectively. This change in morphology is correlated with covalent bond formation as evaluated through FTIR study. Increase in melting temperature confirms the material is becoming thermodynamically more stable after cryo-treatment. The optimization of the cryo-treatment parameters was also done.

Ultrasonic assisted fabrication of graphite reinforced aluminium matrix nanocomposites

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Metal matrix composites are hybrid material with rigid reinforcements in the ductile metal matrix. They exhibit the properties of both the matrix and reinforcement which results in enhanced mechanical properties of the material such as high specific strength, stiffness and increased wear resistance. However, the ductility and toughness of metal matrix composite is significantly decreased by the incorporation of reinforcement particles. These disadvantages can be overcome by metal matrix nanocomposites where the nano sized reinforcement offers significant improvement in the strength of the material without appreciable decrease in ductility. Though it has great potential in aerospace and automobile industries, there are difficulties in attaining a homogenous distribution of the nanosized particles due to their low wettability with the molten metal. In recent years, ultrasonic cavitation treatment was employed to fabricate metal matrix nanocomposites. The present work aims to develop graphite reinforced aluminium matrix nanocomposites using ultrasonic cavitation treatment. Ultrasonic cavitation treatment can improve the wettability between reinforced particles and metal matrix, and distribute particles uniformly in the metal matrix. The microstructural study carried out using Transmission electron microscopy (TEM) shows the effect of ultrasound in breaking the micron sized graphite particles resulting in Al-Gr_pnanocomposites. The effect of ultrasonic cavitation treatment in improving the mechanical properties of Al-Gr_pnanocomposites have been studied and compared with the mechanical properties of Al-Gr_p composite fabricated by conventional stir casting technique.

Ageing, tribological properties and biomineralization of alumina and zirconia based bioceramics

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14 mol% ceria stabilized zirconia (14CSZ), 8 mol% yttria stabilized zirconia (8YSZ), 15 wt% zirconia (stabilized with 14 mol% ceria) toughened alumina (15CSZ-TA) and 15 wt% zirconia (stabilized with 8 mol% yttria) toughened alumina (15YSZ-TA) powder were synthesized by co-precipitation techniques from their respective nitrate salts. The synthesized powders were calcined at different temperatures, compressed uni-axially and sintered in pressure less condition inside conventional electrical heating furnace. Compaction pressure, calcination and sintering schedule were optimized to achieved best mechanical properties in terms of hardness and density. Grain size distributions of sintered specimens were estimated through image analysis of sintered, polished and thermally etched specimens. Accelerated ageing response of sintered and polished specimens were carried out (up to 100 h, at 134 ° C/0.02 MPa) through hydrothermal treatment (ageing) in the presence of simulated body fluid (SBF). After hydrothermal treatment, samples were characterized by X-ray diffraction, scanning electron microscope and further fretting wear were carried out. Fretting wear was carried out on freshly polished as well as hydrothermally treated samples in ball on flat geometry using tungsten carbide ball as counter body. Wear depth and frictional force were measured continuously using two different sensors. Actual wear depth and wear volume were measured through 3D surface profillometric scan analysis of the worn surface. Cleaned and polished samples were soaked into SBF inside a water bath which maintains a constant temperature of 37.5 ° C for a total duration of 28 days. After every alternate day the SBF was replaced and after every 7 days samples were characterized. It was observed that during ageing, CSZ specimens (tetragonal/monoclinic) converts into orthorhombic phase (some fraction) which helps to improve the wear resistance of the material. All the four systems were found to be osteoconductive, however, growth of mineral layer on CSZ specimens were appreciable.

Synthesis and characterization of in-situ A356/TiB_2 nanocomposites

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Aluminum based metal matrix nanocomposite (AMNC) are promising for aerospace, automobile structural applications. Although a variety of processing routes have been explored and studied over the years for fabrication of nanocomposites, the major problem i.e. synthesis and dispersion of thermally stable nanoparticles still remains unsolved. Ultrasonic cavitation assisted dispersion of *ex-situ* particles is a new technique for fabrication of AMNC. The major disadvantages of this process are feeding of particle and its thermal stability. Synthesis of thermodynamically stable nano sized *in-situ* reinforcement particle can solve the problem. In the present work there is an attempt made to synthesis and disperse *in-situ* nano sized

reinforcement particles using ultrasonic cavitation treatment.Aluminum A356/2TiB_2 composites were synthesized by salt melt reaction technique viareaction between K_2TiF_6 and KBF_4 salts. In order to generate nano sized TiB_2 particles, the composite melt was subjected to high intensity ultrasonic treatment for different time intervals in its liquid state and their microstructures and mechanical properties were evaluated. XRD analysis of extracted particles from composites ensures the formation of TiB_2 particle and its stability on ultrasonic treatment.Microstructural analysis of composites shows conversion of dendrites in to equi-axed globular structure along with improved dispersion of particles on ultrasonic treatment. High resolution transition electron microscope analysis shows considerable decrease in the TiB_2 particle size from micron level to sub-micron level by 1 minute ultrasonic treatment and reduced to nano level by 5 minute treatment. The *in-situ* Al A356/TiB2 nanocomposite has shown two fold increase in mechanical properties with low fraction of nano sized reinforcement.

Experimental studies on mechanical properties and machinability of 6061Al reinforced with B_4C particulate MMCs produced via stir casting method

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Metal matrix composites (MMCs) offer high strength to weight ratio, high stiffness and good damage resistance over a wide range of operating conditions, making them an attractive option in replacing conventional materials for many engineering applications. Typically the metal matrix materials of MMCs are aluminum alloys, titanium alloys, copper alloys and magnesium alloys, while the reinforcement materials are silicon carbide, aluminum oxide, boron carbide, graphite etc. in the form of fibers, whiskers and particles. Presenceof harder reinforcement particles makes them difficult to machine.In the present study, the experimental results of mechanical and machinability properties of Boron Carbide particle reinforced aluminum metal matrix composites are presented. The influence of addition of 7 wt% of B_4C_p on mechanical properties and machinability were examined. Addition of B_4C_p resulted in improvement of hardness and tensile strengths to the extent of 71% and 38.4% respectively. Micro structural characterization was investigated by optical and scanning electron microscopy (SEM) to ensure the uniform distribution of reinforcement particles in the Al matrix.Fabricated samples were turned on medium duty lathe of 3 kW spindle power with Poly crystalline diamond tool (PCD) of 10µm particle size at various cutting conditions. The effect of machining parameters, e.g. cutting speed, feed rate and depth of cut on cutting forces and surface roughness was studied.

ADVANCED MATERIAL CHARACTERISATION/ MICROSTRUCTURE AND TEXTURE

Invited talks

Session 1: Nov 12, 2014: [13.30 -15.30]

Redefining the horizons of imaging and fabrication in materials research; Electrons, ions, and X-ray

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The advent of the internet has created a new age in Materials Research resulting in new industries and technologies that did not exist a decade ago. From tablet computing to wearable technologies, be it with touch sensitive screens or longer lasting batteries, materials are a key enabler that have helped drive this development. The Scanning Electron Microscope (SEM) has been the cornerstone for imaging and characterization of materials. Since its commercialization in the 1960s, SEM technology has steadily evolved over six decades keeping pace with the needs of materials research. Recent improvements to this technology include the development of correctors that allows high resolution imaging at lower kV (<100eV) for imaging beam sensitive materials, and the development of the Multi-Beam SEM which allows for high resolution imaging over large fields of view enabling applications such as mapping of brains and failure analysis of semiconductor materials. Charged particle beams also offer the greatest flexibility and the highest throughput for prototyping customized devices for research and development. Traditional approaches utilize a gallium focused ion beam or electron beam for fabrication. While the gallium ion beam is ideal for rapid material removal, the smallest features that can be machined by this technique are about 30 nm in size. Electron beam lithography is routinely used to expose resist and to fabricate structures. However, the technique is limited by proximity effect for dense patterns at the sub-10nm scale. Ion microscopy with helium or neon beams created from a gas field ion source (GFIS) shows great potential and flexibility for many imaging and nanofabrication applications. With a helium or neon ion beam, sub-10 nm structures can be routinely fabricated even in very sensitive materials such as graphene. Additionally, the beam-sample interaction dynamics of helium/neon ion beams offer unique contrast and stunning surface detail at sub 0.5nm lateral resolution. X-Ray Microscopy has brought synchrotron level performance to the laboratory scale. Using a high brightness X-Ray source (8keV) and X-Ray optics, it is now possible to achieve nondestructive imaging with 50nm spatial resolution in the lab. This has provided new insights into the 3D microstructure and defect quantification, and enabled 4D and in situ experiments into different materials. The future of microscopy in Materials Research involves combining the information generated from the disparate imaging modalities, and developing correlative work flows to produce comprehensive information from the same location within the sample.

Materials characterization using Time-Of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS)

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Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is a highly sensitive and versatile analysis technique that provides detailed information concerning sample composition and structure in the near surface region.TOF-SIMS typically employs an ion beam from a Liquid Metal Ion Gun (LMIG) which is used to analyze the material under vacuum conditions. The resulting secondary ions are detected and recorded according to mass in the TOF analyzer.The secondary ion mass spectrum obtained from the sample provides a "fingerprint" which enables the chemical composition to be determined by identification of characteristic mass peaks. Secondary ions with mass up to 12,000 AMU can be detected with a mass resolution of up to 16,000 which is necessary for the elimination of mass interferences.By rastering the ion beam over the sample surface in imaging mode chemical mapping is performed with a lateral resolution of down to 70 nm.Sub-surface composition is investigated by dual beam depth profiling through the use of a second ion gun which delivers a high current cesium/oxygen beam that sputters the material and chemically enhances the emission of secondary ions from it.The combination of imaging and depth profiling modes provides full 3D characterization of the analyzed material.Traditionally, porous materials have been difficult to measure using standard profiling due to the problem with depth calibration caused by the presence of voids. Recently a successful solution to this issue has been obtained through the use of FIB sectioning and imaging the vertical crater wall using TOF-SIMS. Multiple in-situ FIB sectioning enables sample tomography to be performed up to a depth of 100 microns into the material giving full 3D characterization. In this presentation an overview of the sample characterization using modern TOF-SIMS shall be given and examples of its application presented.

Session 2: Nov 12, 2014: [16.00 -18.00]

Redefining the horizons of nanoscale composition characterization –Laser-assisted Atom Probe Tomography

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In the 15 years or so since the introduction of the first commercially-available Atom Probe Tomography (APT) instruments the improvements in the capability of the technique have been quite remarkable. Whilst still maintaining the unique capability to detect, identify and position individual atoms with near atomic resolutions in three dimensions, analysis volumes have increased dramatically, improved detector performance and mass resolving power has produced real enhancements in detection sensitivities, compositional accuracy and precision, and the introduction of laser mode has enabled analysis of very many material systems beyond traditional bulk metals. In addition, the maturation of FIB-based specimen preparation methods has made site specific analyses truly routine. This presentation will provide a brief overview of the APT technique and its evolution and will show examples of state of the art applications ranging from metals, semiconductor device structures analyses to the characterization of ceramics and geological materials.

Novel insights into second phase formation afforded by 3d Atom Probe Microscopy

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Three-dimensional atom probe (3DAP) microscopy or atom probe tomography (APT) is a powerful technique for the characterization of the composition (or chemistry) and structure (or morphology) of multi-phase materials systems at sub-nanometer length scales. The basic underlying principal is determining the spatial coordinates and the identities of the individual atoms in the specimen and then reconstructing this information in the form of a 3D compositional map of the microstructure. The composition of the locally analyzed volume of the specimen is determined directly from the number of atoms of each element present within the collected dataset, thus giving a quantitative measure of the local concentrations at near atomic length scales. However, the full potential of this technique is recognized when it is coupled with other advanced characterization techniques such as transmission electron microcopy (TEM), including HAADF-STEM based techniques, as well as scanning electron microscopy (SEM) - electron back-scattered diffraction (EBSD). When employed in a complementary manner, these techniques can provide novel insights into various phase transformation mechanisms as well as other aspects of microstructural evolution. This talk will briefly discuss some examples which will be used as case studies both for highlighting the various aspects of 3DAP as well its complementary nature when coupled with TEM and other techniques. These case studies are listed below:Precipitation of multiple generations of gamma prime precipitates, distributed over multiple length scales, during the continuous cooling of nickel base superalloys.Nanometer scale omega phase precipitation in the beta matrix of titanium alloys.Role of Cu and Nb on the nucleation and growth of Fe(Si) nanocrystals within the amorphous matrix of Fe-Si-B-Cu-Nb (Finemet) based soft magnetic alloys.Multi-scale phase separation during the solidification of a Co-free $Al_2CrCuFeNi_2$ high entropy alloy.

Phase formation and thermal stability of phases in nanocrystalline high entropy alloys

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HEAs are a new class of multi-component equiatomic (or near equiatomic) alloys, which form simple solid solutions due to their high configurational entropy. The formation of nanocrystalline HEAs has made them more interesting due to their fundamental and technological importance. Recently nanocrystalline HEAs have been made for the first time by the present author's group employing mechanical alloying route. The present talk will highlight the work done in the group of the speaker on the synthesis, phase formation, stability and properties nanocrystalline HEAs. It is important to note that all multi-component equiatomic alloys do not lead to the formation of single phase solid solution or for that matter mixture of solid solutions. In a number of cases, these so called HEAs, have shown the formation of intermetallic phases depending on the enthalpy of mixing of the individual constituents. The present talk uses X-ray diffraction, transmission electron microscopy and atom probe tomography techniques effectively to understand phase formation and thermal stability of a few HEAs on a nanoscale. An attempt has been made to predict phase

formation a priori by evaluating various thermodynamic and topological parameters in the present study. The efficacy of these parameters in predicting the phase formation in HEAs is also presented.

Temporal evolution of nano-scale precipitates in metallic systems: Combined Atom Probe Tomography and Small Angle Scattering study

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APT microscope today is an essential tool for studying nano-scale phenomena. However, APT analysis is limited to a few hundred nanometres. Small Angle Scattering (SAS), on the other hand, has a high field of view and offers the advantage of large statistics at the same length-scale. These two techniques complement each other perfectly and a combination can provide a more complete description of the evolution of fine precipitates. The current study will combine these two techniques and present two examples from (i) an age-hardenable commercial Al-alloy W319 and (ii) Ni-rich NiTi. W319 shows two coherent precipitates on aging: θ′ and Q. Results of the 3DAP analysis of the compositional evolution of these precipitates will be combined with in-situ SAXS (Small Angle X-ray Scattering) results from this alloy. Similarly, in case of NiTi, combined SANS (Small Angle Neutron Scattering) and APT study will be presented as an illustration of the excellent capabilities of these complementary analytical techniques.

Session 6: Nov 15, 2014: [11.00 -13.00]

Tribology basics and advanced surface characterization

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Going green is the theme for designing new machines and components recently. There is a steadily increasing demand for better materials to be used in applications such as scratch-resistant or self-healing coatings, machining, bearings, gears and metal forming where friction, lubrication and wear play an important part. Compared with other engineering or physical science disciplines, tribology is one of the most interdisciplinary subjects known. The objective of this presentation is to provide engineers and researchers an overview of the important tribological considerations (such as surface roughness, material compatibility, contact stresses, etc.) required for designing or manufacturing products, or designing of tribology experiments. It discusses how tribological evaluation and testing under the proper conditions can guide researchers and engineers to choose correct materials, predict the lifetime of a component or system. In each tribological event surfaces are in contact and in relative motion. It is utmost important to characterize surfaces quantitatively in order to understand surfaces before and after the tribological testing. Recent development in 3D optical microscopy based on white light interferometry and AFM (atomic force microscopy) techniques will be discussed for surface characterization.

Contributory papers

Session 1: Nov 12, 2014:[13:30 - 15:30]

Study of austenite to martensite transformation in 9Cr-1.4W-0.06Ta-0.22V-0.12C steel by automated crystal orientation imaging microscopy

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Automated Crystal Orientation imaging Microscopy in TEM is an emerging technique to study the microtexture at higher spatial resolutions than SEM based EBSD. Microstructural analysis of the INRAFM 9Cr-1.4W-0.06Ta-0.22V-0.12C steel, the candidate material for the TBM of nuclear fusion reactors, by conventional electron microscopy showed a fully martensitic structure after normalizing from the austenite phase field. However, analysis of orientation maps in TEM revealed the presence of fine grains of retained austenite at prior austenite grain and lath boundaries in low amounts, which was also supported by Mossbauer spectroscopy studies. Such retention of austenite is not expected in view of the high Ms temperature of the steel and low temperature interval between Ms and Mf. The reason for the retention of austenite in normalised steel is due to the reduction in the transformation kinetics of austenite to martensite close to the finish temperature. Retained austenite was not observed in the steel tempered after normalizing. The observation of both retained austenite and martensite phases shown by orientation imaging facilitated the direct identification of the orientation relationship between the parent and product phases. Formation of multiple variants of martensite was also established from the analysis of the inverse pole figure maps. Details of the study will be presented.

Transmission kikuchi diffraction: innovative microstructural characterization technique for nanomaterial with enhanced resolution

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Electron Backscatter Diffraction (EBSD) coupled with Field Emission Gun Scanning Electron Microscopy is recognized as a proven characterization tool for microstructural features having spatial resolution of about 100 nm. Recently, with the advent of low energy Kikuchi Transmission Diffraction (TKD) (also known as transmission-EBSD) giving an improved spatial resolution of less than 10 nm, opening new horizons in

microstructural characterization of nano-materials. The present work illustrates an understanding of the TKD methodology and its different variables that affect the diffraction signal. Effect of specimen thickness, tilt, working distance, probe current and voltage are correlated with Kikuchi pattern. Examples of TKD on severe plastic deformed aluminum, aluminum alloy and aluminum composites performed under optimum condition will be presented. Effective resolution of microstructural features from 10 to 100 nm Nickel Aluminide, Al-TiB2 and Silicon thin film samples will also be discussed. The arrangement of TKD sample holder with EBSD detector and electron column is shown in Fig 1(a). Systematic investigations were carried out for understanding the optimum condition required for TKD. Severe plastic deformation pure aluminum [Fig. 1(b)], friction stir processed aluminum alloy (Al-30Si) [Fig. 1(c)] and nanocrystalline silicon thin film investigated by TKD technique shows resolution of less than 10 nm. Conventionally by TEM the characterization is relatively difficult to perform and the information about grain size and texture are challenging. With the advent of TKD technique, characterization of microstructural features such as grains, sub grain boundaries and severe plastic deformed (SPD) alloys at 10-100 nm level can also be effectively studied and quantified.

Challenges and solutions in mechanical testing of high strength steels

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The increasing demand for lightweight materials is challenging the steel producers. Steels are undergoing constant improvements and their properties are evolving continuously in order to meet the requirements of the automotive industry. Also advanced metal forming processes are increasingly contributing to improved materials properties. This leads to higher requirements for material testing equipment as modern alloys become harder and stronger. As a consequence classical mechanical testing tools are reaching their limits with respect to sample preparation, sample gripping technology, test speed control, load and strain measurement, etc. As a leading supplier of advanced material testing systems, the Zwick Roell Group is constantly innovating and developing testing solutions that meet the new challenges of the industry. By referring to some examples it will be shown how newly developed testing solutions help customers in research and development as well as in quality assurance to better characterize advanced metallic materials.

Session 3: Nov 13, 2014: [09.00- 11.00]

Evolution of texture of finished product cold rolled non- grain oriented electrical steel sheets

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Cold Rolled Non-Grain Oriented (CRNO) electrical steel sheets have been specifically used for making core of rotating machines in particular electrical motor, generators, and core of small size transformers. In the present investigation, the finished products of four different types of CRNO steel sheets (CN1, CN2, CN3

and CN4) are chosen. The details about magnetic properties, and evolution of micro-texture were analyzed. In CN1 grade steel sheets the main texture components are (112)[5 -9 2] , (001)[1-2 0] and (554)[-1 -3 5], moreover (111) [1 -2 1] and (554)[-2-2 5] are weak texture components. Texture components (111)[-1-1 2], (554)[-2 -2 5] and (332)[-1-1 3] and (001)[1-3 0] are found in CN2 type steel sheets. In CN3, the main texture component is (110)[001] and the other texture components are (111) [-1-1 2], (554)[-2-2 5] and (001)[1 -2 0]. (001)[1-3 0], (111)[-1-1 2], (554)[-2-2 5] and (110)[1-1 5] orientations are developed in CN4 type steel sheets. In all above mentioned four type of CRNO steel sheets, α*-fibers ($(h\ k\ l) < \frac{h}{l}+1, \frac{h}{l}+2, \frac{h}{l} >$) which is parallel to α-fiber (RD//<110>) are observed.

The microstructure and mechanical properties of thin wall austempered ductile iron

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In the present work, ductile iron plate castings with different section thicknesses of 2 and 3 mm were cast with an appropriate casting design to assure good mold filling, shrinkage free without chill. Short term austempering treatment process was used to obtain thin wall ADI castings. The samples of 2 and 3 mm thickness were austenitized at 910 °C for, 30 minutes followed by holding at 350°C and 400°C for 5 and 10 minutes for each temperature. The mechanical properties like hardness, yield strength and ultimate tensile strength decreases, if the austempering temperature is increased with austempering time. Hardness and ultimate tensile strength are higher as in case of ausferritic matrix than the ferritic matrix structure.

Deformation and magnetic properties in electrical steel

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This is a consolidated study on deformation, both elastic and plastic, and changes in magnetic properties in electrical steel. Controlled deformation was imposed on CRGO (Cold rolled grain oriented) and CRNO (Cold rolled non-grain oriented) with post-deformation monitoring of microstructures and magnetic properties.This study begins with anisotropic tensile deformation of CRGO. Then it describes the experimentally observed deformation behavior with microstructural observations, discrete dislocation dynamics simulations and textural stability. It also relates degradation in magnetic properties empirically with various aspects of deformed microstructures. Finally, the role of strain mode and elastic deformation on CRGO were also explored. In both cases, dominant role of residual strain, or changes in d-spacing, were established as a critical factor. In CRNO, on the other hand, clear evidences of mechanical stress relief were shown. Near yield point plastic deformation altered orientation gradients, removed prior deformation, in-grain misorientations, altered signatures of residual stresses and imposed clear improvements in magnetic properties. Such observations were also extended to CRGO, clearly highlighting

technological possibilities of mechanical stress relief as a means for improving performance of electrical steels.

Evaluation of austenite texture through variant selection from ferrite in hot deformed microalloyed steel

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Crystallographic texture is considered as one of the important characteristics on which various properties of the material like strength, ductility, toughness etc depend. Micro-alloyed steel consists of C (<0.1%), Nb, V, Mo (<0.05-0.15% total alloying additions) etc. These high strength structured materials should possess yield strength of 550 to 600 MPa along with good weldability. During the processing of these steels, there is a continuous change in its texture. That is why the study of evolution of texture is important. Development of undesirable textures can lead to weakness and failure of the material. During hot deformation of micro-alloyed steels, austenite phase gets deformed and later with cooling, this austenite transforms to ferrite. Austenite to ferrite phase transformation proceeds through a change of crystal structure from fcc to bcc and during this transformation, certain orientation relationships between the crystallographic planes and directions of the parent and product phase are followed. Thus, the ferrite phase inherited texture from the parent austenite phase.In this work, austenite texture has been evaluated from the ferrite texture measured at room temperature employing variant selection through K-S orientation relationship. An approach has been suggested to calculate the high temperature phase texture of micro-alloyed steel by using EBSD data of the ferrite phase. By monitoring the austenitic texture, the ferritic texture can be controlled and if this can be done, then the ambient temperature properties of micro-alloyed steels can be manipulated according to the requirements.

Effect of material size and microstructure on mechanical behavior of SS 304 thin sheet

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In this study, the effect of heat treatment and plastic deformation on microstructure and mechanical characteristic of Stainless Steel 304 (SS 304) thin foil subjected to room temperature uniaxial tension was investigated. SS 304 of three thickness in micro-meter range (30 µm, 50 µm and 90 µm) were selected as working material to study the miniaturization and grain size effect. Different grain size were produced by heat treatment process in different holding time interval. Electron backscattered diffraction (EBSD) was used to quantify the grain growth at different time interval. Tensile test was carried out to understand effects of thickness, grain size (both surface and cross section grain) on mechanical behavior and elongation of material. The effect of annealing twinning fraction and misorientation on mechanical behavior were investigated by EBSD imaging followed by TSL analysis. The deformation twinning fraction and misorientation development were quantified by the same EBSD and TSL analysis. The texturing effect with different thickness of material was investigated by annealed and deformed microstructure. A pure

thickness effect on yield stress (YS), ultimate tensile stress (UTS) and elongation was investigated at nearly same grain size. The grain size effect on YS, UTS and elongation for various thickness was studied. It was observed that the mechanical behavior varies with different thickness of material as well as different grain size.

Effect of processing path on mechanical behaviour of 316 stainless steel

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Specimens of 316 L stainless steel were subjected to different processing routes by rolling at room temperature and cryogenic temperature to obtain a distinct defect structure. In addition, the change in strain path was introduced during rolling by subjecting the samples to unidirectional rolling, two step rolling and multi-step cross rolling to strain of 2.3 in plane strain compression. This yielded stainless steel sheets with distinct crystallographic texture along with distinct microstructure and defect structure. These samples were subjected to short term annealing at different temperatures to obtain recovered and recrystallized microstructures. Tensile properties were performed on these samples to determine the processing route for optimum combination of strength and ductility. A detailed characterization of the processed and tested samples was carried out using Scanning Electron Microscopy based Electron Back Scatter Diffraction and X-ray diffraction. The exact micro-mechanisms responsible for optimum combination of strength and ductility will be discussed.

Microstructural characterization and mechanical behaviour of interstitial free steels during deformation

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As received Interstitial-free steel of composition 0.0029C – 0.15 Mn – 0.054 Ti – 0.011 Nb was heated to various temperatures ranging from 740°C to 930°C, held for 20 minutes and were quenched in water. The microstructural observations of the samples thus quenched were made and are taken as reference to map the changes in structure due to application of deformation at these temperatures. The samples from same steel are deformed at the above mentioned temperatures; the deformation percentages at each temperature are 30% and 50%. Optical and transmission electron microscopy were carried out for microstructural study. EBSD studies were done to understand the effect of deformation on the transformation behavior of austenite. In order to gather information about the elastoplastic behavior of the ferrite formed due to deformation, nanoindentation technique was used for a few selected samples. Also transformation kinetics was studied by DSC analysis.From the results of DSC experiments, it is found that the activation energy for transformation of austenite in steels heated to different temperatures, and followed by quenching, is higher for lower reheat temperature. Application of deformation has shown that higher percentage of deformation at a particular temperature reduces the activation energy for transformation. Also it is revealed that for a particular deformation percent increase in temperature of deformation increases the amount of DSIT ferrite. From the microstructural analyses, different types of

ferrite grains are identified. TEM observation has shown that deformation at higher temperature, 740°C by 30% gives rise to considerable amount of deformed ferrite. EBSD analyses have given evidence of the formation of regions, separated by low angle boundaries. More than sixty percent of the grains contain such small subgrains of less than a micron. These act as the nuclei of DSIT ferrite.

Study on microstructural characteristics in 21-4N engine valve steel material

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21-4N engine valve steels are demanded for exhaust valves of engines where service temperatures have been on the constant rise. The steel is usually supplied in the solution annealed and aged condition. The desired microstructure is having nitrogen pearlite in the matrix for engine erosion resistance against the exhaust gases of combustion. The steel is a high carbon high nitrogen austenitic stainless steel and it is of interest to examine the microstructure development and mechanical properties at various conditions of aging to understand the steel behavior. In the present study, samples were cut and solution treated at 1050°C/1 h. These samples were aged at three different temperatures [500°, 600° 760°C] for varying time durations. The C-curves were examined. Selected samples were tested for high temperature tensile behavior. The wear and erosion resistance of an automotive engine valve is enhanced by the cellular Cr_2N precipitation, although toughness and ductility of the steel deteriorate.

Initial microstructural evaluation of tamahagane steel

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Tamahagane steel produced from tatara furnace is a hyper-eutectoid steel with very few impurities, which makes it an ideal raw material for manufacturing of the samurai swords. Tatara furnace operates at temperatures below the melting temperature of Iron, resulting in an inhomogeneous mass distribution of steel. Swordsmith used this to their advantage by forge-welding different grades of tamahagane and laminating them into layers to develop soft low carbon core and hard high carbon case. Hot deformation caused ultra-fine grains in the microstructure, leading to high formability of steel due to superplasticity. In the present study, a raw steel product from tatara furnace forged at 700-750°C was used for mechanical and microstructural investigation. Electron Probe Micro Analysis (EPMA) results showed overall 1.25 % carbon composition with trace amount of impurities. Microstructural studies from optical and SEM showed pearlitic matrix and cementite plates precipitated at prior austenite grain boundaries and some having widmanstatten intra-granular morphology. Ferrite and cementite layer spacing in the pearlite banding is found to be in the order of 100-400 nm.EBSD results showed fragmentation of ferrite grains and very less recrystallization and recovery of the microstructure. From the grain orientation spread (GOS) maps, fracture of cementite plates as well as different orientation in the same cementite plate is observed due to heavy deformation. Our study confirmed the hyper-eutectoid composition of the tamahagane steel causing formation of pearlite in the matrix and cementite precipitating along the grain boundaries during cooling.

Effect of alloying addition in low Mn (8-12wt%) TWIP/TRIP steels and correlation with deformation and mechanical properties

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The technological importance of Mn steels increased in last 5-10 years because of their very high strength, ductility and ease of thermomechanical processing, which leads to huge market potential in automobile industries. However, processing of Mn steels is still a problem because of Mn segregation and needs to addressed. Present study deals with the minimization of Mn content for further processing of TWIP and TRIP/TWIP steels. Here we report the thermo-mechanical processing (Rolling) of low Mn-steels and consequent evolution of texture and microstructure. Low-Mn austenite steel has been deformed upto 90% by cold rolling to investigate the fractional martensitic transformation and deformation mechanisms. Electron Back-scattered Diffraction (EBSD) and X-Ray Texture measurements have been used to study deformation mechanisms (Twinning Induced Plasticity and Transformation Induced Plasticity) and evolution of bulk texture for the low-Mn steel. Vibrating Sample Magnetometer (VSM) has been used to study Transformation Induced Plasticity by saturation magnetic moment mesurement. Tensile test has been performed to get the strength and ductility, its revealed 1GPa ultimate tensile strength and 50-60 % elongation upto fracture for the low-Mn steels.

Effect of annealing on recrystallisation texture of cold worked twin induced plasticity (TWIP) steel.

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Twin Induced Plasticity (TWIP) steel is considered to be one of the attractive materials in automotive sector due to their good combination of high strength to weight ratio. The present work describes the phenomenon of recrystallisation kinetics and change in texture components in cold worked TWIP steel with respect to annealing. The microstructure and micro-texture evolution of 40% cold-rolled and isochronally annealed Fe-22.5Mn-1.5Al-0.35C TWIP Steel was investigated using Electron Back Scattering Diffraction (EBSD) technique. In Cold rolled condition bulk texture study showed retention of strong alpha-fiber, with higher intensities of Brass ({111} <112>) as compared to Goss ({112} <110>) components which is very similar to other low SFE materials. However fully recrystallised sample showed along with strong rotated Goss component similar intensity of A ({110} <111>) orientation which has been observed in this present TWIP steel. This is unique and has never been found in other TWIP steel of different composition. A comparison between bulk and micro texture study was also investigated. End texture also contained negligible intensity of E ({111} <112>) and F ({111} <110>). This is because E and F have less advantageous growth conditions.

A set of algorithms for generating uniform random microstructures

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Microstructural features play critical role in governing the material behavior under various loading conditions. Those features can be categorized in two sections: (i) morphological features such as grain size and shape and (ii) crystallographic features such as orientation distribution function. In order to develop novel materials theoretically, one needs to generate artificial microstructures based on the phase transformation laws as well as orientation assigning methodologies followed by testing the novelty of the artificial microstructure by FE analysis. This process helps in identifying the key features of tailored microstructures. To generate random microstructures, both morphological and crystallographic features need to be generated artificially. In reality, microstructure evolution is a complex phenomena in which both nucleation and growth rates vary with space and time based on the thermal gradients, nuclei orientations, density of nucleation sites etc. This paper introduces an algorithm for generating artificial grains in a finite 2D domain based on nucleation and growth mechanisms. Assigning unbiased random crystal orientations is not as trivial as it seems to be. So, algorithms for assigning truly uniform crystal orientations to the individual grains are discussed.

Session 4: Nov 13, 2014: [13.30- 15.30]

Evolution of {001}<100> texture in cold and warm-rolled Al-2.5wt.%Mg alloy

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The evolution of recrystallization cube texture ({001}<100>) was investigated in severely deformed Al-2.5wt.%Mg alloy. For this purpose the alloy was cold and warm-rolled to 97% reduction in thickness at room temperature and 473 K (200°C). The deformed materials were annealed at temperatures ranging between 473 K (200°C) and 673 K (400°C). The deformed materials showed fine lamellar structure and strong copper type texture. However, the warm-rolled material showed much stronger cube texture ({100}<001>) as compared to the cold-rolled material after different annealing treatments. Oriented nucleation of cube grains during recrystallization was observed in warm-rolled material as compared to the cold-rolled material. Strong oriented nucleation behavior of cube grains was due to more recovered structure of cube oriented grains in warm-rolled material. In contrast, dislocation pinning by solute Mg atoms during cold-rolling inhibited the recovery of cube grains and diminished the strength of cube texture after recrystallization.

Structure- property correlation of a novel 6351 Al-(Al_4SiC_4 + SiC) hybrid composite

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In the present research work 6351 Al-(Al_4SiC_4 + SiC) hybrid composite is developed through stir casting method where SiC (size: 25 ± 6 µm) and TiC (size: 430 ± 50 nm) particles are incorporated into the 6351 Al melt at 760°C with simultaneous stirring at 200 rpm for 30 min followed by casting in a standard cast iron mould. The addition of TiC results in the generation of in-situ Al_4SiC_4; while SiC remains as ex-situ reinforcement. Microstructure evolution in correlation to developed hardness and mechanical properties along with mode of tensile fracture has been studied in detail. The experimental results envisage some interesting microstructural modifications (i.e. elimination of most of the dendrites, grain refinement etc) and nearly homogeneous distribution of reinforcement particles without segregation so as to obtain excellent combination of properties. In general, both the reinforcements are found to act as the nucleation sites for primary α (causing extensive grain refinement) along with the evident engulfment effects promoting uniform particle distribution. Furthermore, the addition of TiC in 6351 Al melt results in the generation of Al_3Ti precipitates of nanometric size in the microstructure along with Al_4SiC_4 in-situ reinforcement particles. The effects of grain refinement, dominant presence of equiaxed α-grains (lower proportion of dendrites) and simultaneous reinforcement effects of hard SiC and Al_4SiC_4 particles along with the strengthening effect of Al_3Ti nano-precipitates eventually result in an improved combination of strength (UTS = 205 MPa) and ductility (%Elongation = 12) in Al-(Al_4SiC_4 + SiC) hybrid composite as compared to 6351 Al in the as cast condition (UTS = 119 MPa and %Elongation = 3).

EBSD study of in-situ tensile/heating of Al- 0.1Mg alloy

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In-situ experiments in the SEM combined with crystallographic data gathered using EBSD data can provide insight of mechanisms operating during deformation and recrystallization of materials. In order to conduct such experiments suitable scanning electron microscope chambers that can accommodate in-situ tensile-heating stages are required. In addition suitable EBSD systems that can handle infra-red radiation emitted from hot samplaes are important. EBSD combined with in-situ tensile and heating experiments on fully recrystallized Al 0.1Mg alloy with average grain size 310 um, will be described in this presentation to give insight into the generation of deformation and subsequent recovered and recrystallized microstructures within the same set of initial grains. SEM experiments were done in a high resolution FEG MIRA 3 XM TESCAN microscope, with a GATAN Microtest EH2000 tensile stage incorporatating heated grips.Results from preliminary tests on the Al 0.1Mg alloy showed that during tensile loading the in-situ accumulation of deformation occurs mainly by slip and is orientation dependent. Existing cube oriented grains give rise to new recrystallized cube grains during heating, at the expense of elimination of rotated Goss oriented grains.

Phase transformation of friable chromite ore during heating in air and argon

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Cr content in chromite ores varies in the range of 30-45% in India. Very few fundamental studies have been carried out to understand the phase transformation and thermo-chemical reactions occurring when Indian chromite ores interact with oxides under different environmental conditions. Such a study is vital to design appropriate agglomeration strategies as well as to improve the efficiency in the production of ferro-chromium in the submerged-arc furnace. This paper aims to bridge this major gap in the knowledge about the thermo-chemistry of these processes and their influence on the physical and chemical nature of the ore. These studies would help in designing appropriate process routes to treat raw ores and to produce suitable agglomerates from the finer ores for improving the efficiency of production of ferro-chromium.The ore was taken from Sukhinda area, crushed and sized at NIT Jamshedpur. Powder of 150 mesh was utilized to make polished section after various heat treatments at 900, 1000, 1100, 1200°C for 1h in presence of air and Ar gas before microscopic study. This study reveals the dissemination of Chromite particles in its parent phase, with the increase in temperature. Powder of 150 mesh after the series of heat treatments at 900, 1000, 1100, 1200oC for 1h in presence of Ar gas and was taken for XRD study. We observe that interaction between the phases takes place at different temperatures. Generation of new phases and integration of the few phases takes place. These integration and generation of the old and new phases may be the possible reason for the fusion of the pellets in Ferro-chrome industries during the process. Sample studied were very few in number, hence further study is required.

Friction stir processing of magnesium: defining the microstructural anisotropy

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A commercial magnesium alloy was processed through Friction Stir Processing (FSP). FSPs were done at different (same direction, reverse and transverse) pass schedule and holding temperatures (air, water and liquid nitrogen). Other than observations on location dependent microstructural refinement, clear trends in shear anisotropies were made. Use of a visco-plastic simulation with appropriate velocity gradients could capture the trends in texture anisotropies under different FSP pass schedules. Finally, finite element simulations enabled predictions of temperatures at different locations and for the respective holding temperatures. This was confirmed through direct experimental measurements of temperatures, and indirect observations on process zone dimensions. A combination of temperature and shear strain distributions defined the microstructural anisotropies of the magnesium FSP.

Effect of iterative thermo-mechanical processing on grain boundary character distribution of Ni based superalloy

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Inconel 600 is a standard nickel based superalloy for high temperature applications (typically greater than 1000 °C) and corrosion resistance. Several methods have been proposed to improve the usability of such materials in these harsh environments like applying thermal barrier coatings, designing microstructures etc. Designing microstructure involves modifying the grain boundary structure which plays an important role in functional properties such as intergranular corrosion and oxidation resistance as well as mechanical properties such as fatigue and creep resistance etc. Various thermo-mechanical processing (TMP) routes have been shown to have a better control over grain boundary structure.Aim of this is to study the effect of iterative thermo-mechanical processing on the grain boundary character distribution (GBCD) of Inconel 600 and in turn, on material properties. Specifically, we are trying to understand the role of processing cycles in the evolution of low ∑ coincident site lattice (CSL) boundaries, especially $\sum 3^n$ (n=1-3) based on grain boundary parameters and finally, its correlation with material properties. Total six TMP cycles were carried out to generate the set of microstructures where each processing cycle involved a low level of deformation (4%) followed by annealing at 1000 °C for 10 minutes. Characterization of the processed material was done using electron back-scattered diffraction (EBSD) technique to determine the CSL boundary fraction as well as grain size. EBSD maps were used in analyzing the effect of processing parameters on the microstructure of material. Mechanical properties like hardness and fatigue tests were also assessed using Rockwell hardness and high cycle fatigue,respectively.

Development of magnetic anisotropy in nano-composite Nd-Fe-B/α-Fe rare earth magnets

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The nano-composite Nd-Fe-B/α-Fe Rare Earth Magnets have great potential to compete with the permanent magnet demand with lower cost and less complicated processing.However, the magnetic anisotropy is one of the prime factor to improve the magnetization. The plastic deformation induced magnetic anisotropy in the nano-composite magnet has been demonstrated to generate high value of magnetic anisotropy in such system under critically controlled processing parameters. A systematic studyon hot deformation has been carried out by us for two different compositions ($Nd_{1.6}Fe_{14}B$+0.5Ga) and ($Nd_{1.8}Fe_{14}B$+0.5Ga). The alloy was induction melted at1673-1700K, then melt-spun and hot deformed in the 973K to 1123K and true strain rate of 10^{1} to $10^{-3}s^{-1}$.It has been observed in $Nd_{1.6}Fe_{14}B$+0.5Ga and $Nd_{1.8}Fe_{14}B$+0.5Ga nano-composite samples that with the lowering of Nd content, where α-Fe phase is higher, tendency for magnetic anisotropy is reduced. It shows very unique feature of isotropy of value within 5% in $Nd_{1.6}Fe_{14}B$+0.5Ga, whereas the value in $Nd_{1.8}Fe_{14}B$+0.5Ga system is not so. The degrees of the texture (D_T) have been calculated for all the deformed samples. $D_T(\%)= [Br_{(//)}]^2/ [\{Br_{(//)}\}^2 + \{Br_{(//)}\}^2]$ x100. The result shows D_T = 51 to 64%. The lowest texture was found in the sample deformed at 1023K at $1x10^{-2}$/sec, The reason for the drop in texture was confirmed by the presence of (410) and (331) planes which

are not the easy axis of magnetization. The optimum degree of texture (D_T) was obtained in the sample deformed at 1073K at the strain rate of $1x10^0$/sec. In this case it has the (006) and (105) planes along the axis of magnetization. The observations show that there is great potential to improve the degree of texture under controlled process parameters of temperature and strain rate.

The microstructural evolution of a Ti-6Al-4V alloy during annealing treatment

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The variation in microstructural behavior with respect to minute changes in the processing conditions such as temperature and time during heat treatment makes titanium and its alloys difficult to process in industry. Ti6Al4V is a promising alloy in the aviation industry, but the narrow processing window of time and temperature due to the phase transformation affects the heat treatment behavior. In this work the effect of temperature and time on the annealing behavior of Ti6Al4V alloy was analyzed. The hot rolled and mill annealed Ti6Al4V consisted of partially equiaxedand elongated deformed α grains with a large number of sub-grain boundaries. The β transition temperature of the alloy was found 995°C. The alloy was reheated to different temperatures in the α+β region and held for different time intervals followed by cooling at a rate of 8°C/min inside the furnace. Electron back scattered diffraction (EBSD) technique was used to study the microstructure evolution in each phase constituent. During annealing, the population of low angle boundaries gradually decreased with time, resulting in the transformation of elongated deformed α grains to equiaxed grained structure. In addition, the volume fraction of β phase increased on annealing, due to the decomposition of supersaturated initial α phase. The extent of these microstructural changes was significantly influenced by the annealing parameters (i.e. isothermal holding time and temperature).

Accurate determination of orientation relation in martensite transformation of 9Cr-1Mo-0.1C steel by microtexture analysis of product ferrite boundaries

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In low-carbon alloy steels, the governing orientation relationship (OR) during martensite transformation of austenite (γ) is known to be close to the Kurdjumov-Sachs (KS) relation. The OR have been determined conventionally through TEM studies by comparing the spot/Kikuchi line diffraction patterns of the product ferrite (α) variant and the remnant γ phase. Recently, methods have been suggested for arriving at the optimum OR of steel by analyzing the EBSD microtexture data of the product α crystallites. The procedure for deducing the average γ→α OR based on experimentally observed misorientation between product variants would be presented.The tempered martensite microstructure got by Normalizing & Tempering treatment of 9Cr-1Mo steel was characterized by SEM-EBSD technique to obtain the microtexture data from a large area scan. The crystallographic orientation of all neighboring pair of points in the array dataset was analyzed to find misorientation angle (ω)-rotation axis (***r***), and plotted in a neo-Eulerian space.

The (ω,***r***) peaks corresponding to the relation between product α variants was determined, and could be identified to be close to the product variant misorientations arising from KS OR. A set of 3 (ω,***r***) peaks denoting the misorientation of three variants with respect to a reference variant was plotted as tan(ω/2)***r*** in Rodrigues-Frank (RF) space. In RF space, the set of orientations equi-distant from the reference variant (at origin) and another variant at tan(ω/2)***r*** is an in-between plane with its normal as ***r*** and at a distance of tan(w/4) from origin. The parent γ orientation need to be equi-distant from all of its product variants, and could be determined as the intersecting point of the three equi-distant planes (constructed for the three observed misorientation relation of α variants). The misorientation between parent-γ and product-α could be deduced as (ω=44.15°,***r***=[0.1587 0.1411 0.9772]), which is 2.3° away from KS OR.

Hardness characteristic and shear band formation in metastable β-titanium alloys

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The use of metastable β-titanium alloys in the aerospace industry has been increasing day by day due to their attractive properties. These are good strength, hardenability, fracture toughness and reasonable ductility. These alloys do not form martensite but retain the β phase upon quenching from the β phase field. As a result, the microstructure can be engineered during heat treatment with the help of the retained β phase which in turn governs the mechanical properties. The mechanical properties of these alloys are largely affected by the volume fraction, morphology and distribution as well as size of the hexagonal α phase precipitates within the matrix. Shear bands around the edges of indenter have been observed mainly in bulk metallic glasses. However, observation of shear bands during indentation in crystalline titanium alloys is rather limited. These bands have been reported in different β-titanium alloys during different modes of deformation such as rolling, compression etc.Shear band formation is one of the important deformation and failure mechanism and frequently occurs in short time. Therefore, it is quite useful to examine the formation of shear bands during different modes of deformation. The present work is thus concerned with the hardness characteristic and shear band formation during indentation in different metastable β-titanium alloys. The alloy Ti-5553 and three other alloys which are derived from Ti-5553 by keeping Mo equivalent (8.15) constant have been used for this study.Shear bands have been observed during Vickers indentation with indentation loads in the range of 0.1 to 20 Kg in four metastable β titanium alloys in β water quench condition. Extent of solid solution has marked influence on the evolution of shear bands. All the four alloys exhibit the shear bands. The spacing between shear bands increases with increase indistance from the indenter edge.

Study on fatigue deformation behavior of Ti-6Al-4V titanium alloy using EBSD technique

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Electron backscatter diffraction (EBSD), also known as backscatter Kikuchi diffraction (BKD), is an SEM based microstructural-characterization technique to study/analyze the crystallographic orientation. EBSD can be used to discriminate crystallographically dissimilar phases by comparing the interplanar anglesmeasured from the diffraction pattern, with calculated angles from a set of candidate phases and selecting the best fit. To characterize the crystallographic orientation of different regions on local fracture surface, EBSD technique was employed in the present study for low cycle fatigue tested specimens of Ti-6Al-4V alloy. The orientation images captured at different locations on the specimen surface for two different specimens have been compared. Further, the Schmid factor has also been computed based on the defined loading conditions. Significant variation has been observed in the slip plane orientations and distributions at these locations. This correlates well with the fracture micromechanism observed in this alloy. It was found that near basal orientation of hcp phase along with very high slip activity is responsible for crack initiation as discussed in this paper. The EBSD data was also used for mapping the stiffness profile onto various specimens with the help of MATLAB software.

Effect of orientation on recovery, recrystallization and α - β phase transformation of Ti-5553 alloy for aircraft structural applications

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Recovery, recrystallization, and α to β phase transformation behaviour for Ti-5553 alloy were investigated using dilatometric experiment and orientation image microscopy. Continuous heating of deformed samples at constant heating rates were carried out in dilatometer set up, during which different phenomena like recovery, recrystallization and α to β phase transformation occur. Expansion or contraction observed in the dilatometry curves (Relative change in length vs. Temperature) were caused by combined effect of expansion due to α to β phase transformation and contraction due to recovery and recrystallization. Dilatometer samples of different orientations were prepared keeping its longitudinal direction parallel to rolling direction (RD), normal direction (ND), and directions at angles of 30° and 60° from RD direction in the RD – ND plane. With change in sample orientation dilatometry behaviour has been found to change significantly. When samples' longitudinal direction was parallel to RD, then no contraction was observed during heating; instead a continuous expansion was seen in the temperature range of 250°C - 810°C. However, for the sample whose longitudinal direction was parallel to ND, significant contraction was observed in the temperature range of 610-915 °C in addition to expansion behaviour observed due to phase transformation. In the present paper these interesting observations would be explained in terms of texture present in the initial deformed material and its effect on phase transformation and recovery and recrystallization behaviour would be illustrated.

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Microstructure evolution and pre-transformation microstructure reconstruction in Ti-6Al-4V alloy

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In the present investigation, Ti-6Al-4V alloy was subject to solution annealing treatments in β (1066°C) and α+β phase (930°C and 850°C) followed by quenching, air cooling and furnace cooling to room temperature, respectively. The effect of solution annealing and cooling on the microstructure was studied by using optical microscopy (OM), scanning electron microscopy (SEM), electron backscattered diffraction (EBSD) and x-ray diffraction (XRD). The chemical composition of the β phase for different conditions was determined with the help of energy dispersive spectrometer (EDS) attached to SEM. Furnace cooling resulted in the development of coarser structure (α+β), while air cooling resulted in much finer structure with widmanstatten morphology of α at the grain boundaries. Quenching from solution annealing temperature formed α' martensite, their proportion being dependent on the temperature in β phase field. It is well known that the transformation of β to α follows Burger orientation relationship (OR). In order to reconstruct the microstructure of parent β phase, a MATLAB code was written using neighbor-to-neighbor, triplet method and Tari's method. The code was tested on the annealed samples (1066°C solution annealing temperature followed by furnace cooling to room temperature). The parent phase data thus generated was then plotted using the TSL-OIM software. The reconstruction results of the above methods were compared and analyzed. The Tari's approach (clustering approach) gave better results compared to neighbor-to-neighbor and triplet method.

Mechanical and microstructural behaviour of IMI 834 titanium alloy severely deformed by high pressure torsion

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IMI 834 titanium alloy is a near alpha (HCP) titanium alloy used for high temperature applications with the operating temperature up to 600°C. This alloy is extensively used in gas turbine engines such as high pressure compressor and low compressor discs. Desired better dwell fatigue life and creep properties demand ultrafine grain microstructure with random crystallographic orientations for these applications. Alpha (HCP) titanium phase, being limited by lesser number of possible crystallographic slip systems, upon conventional deformation generates strong texture. On the other hand severe plastic deformation (SPD) imparts large plastic strain without change of sample size and can generate ultra-fine grained microstructure, in turn imparting higher material strength and may generate random texture.The present work deals with severe plastic deformation (high pressure torsion) of IMI 834 titanium alloy at various plastic strains (several rotations) and then study of its mechanical-microstructure evolution. The hardness profile of the SPD samples will demonstrate its mechanical properties and the orientation maps with crystallographic texture and grain boundary details will provide its microstructure evolution.

On the effect of tin content and texture on the twinning behaviour of Zr-based alloys

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Samples of binary alloys of Zirconium (Zr) and Tin (Sn) of varying Sn content (0.33, 1.2 and 2.9 wt.%) were made in two orientations: (I) compression direction parallel to rolling direction and (II) compression direction parallel to transverse direction. The evolution of microstructure, with emphasis on twinning

behaviour, was studied during low temperature (-80°C) compression of the samples in both the orientations. Samples of type I showed more $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ twinning as compared to samples of type II for a given extent of deformation for each alloy composition. Increasing Sn content in the alloys was also seen to increase twinning activity for a particular extent of deformation in each orientation. The twinning behaviour was quantified by EBSD as well as X-ray Diffraction technique.

Mathematical model correlating texture and slip activity in HCP titanium alloy

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Deformation behavior of Ti alloys is of great interest because of its applications in the aerospace industry as landing gear beams and engine parts. Since Ti has a hexagonal crystal structure, the available slip systems are limited and twinning also plays a significant role in deformation. Both slip and twinning are dependent on crystal structure and orientation, in addition to processing parameters such as the stress state, temperature, and strain rate. In the present work, we have calculated resolved stresses on various slip systems in Titanium for various types of loading. A comparison with CRSS values has been used to identify active slip systems and their contribution to slip. By considering various grain orientations and the active slip systems, we can predict textures that would yield desired plastic deformation. Results will show contributions of various slip systems to deformation in grains of different orientations.

Effect of deformation strain on microstructural evolution of Zircaloy-4

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The effect of deformation strains on the microstructural evolution of Zircaloy-4 at liquid nitrogen temperature has been investigated in the present work. The electron backscattered diffraction (EBSD) and transmission electron microscopy have been used to study the evolved microstructure. The deformed microstructure has been studied in three orthogonal directions, which were marked with numerous mechanical twinning and shear bands. To sustain uniformplane-strain deformation, both twinning and shear bands were formed. The extension twins of type $\{10\bar{1}2\}\langle\bar{1}011\rangle$ and $\{11\bar{2}1\}\langle\bar{1}\bar{1}26\rangle$and, contraction twins of type$\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$ have been observed. The dominance of $\{10\bar{1}2\}\langle\bar{1}011\rangle$ extension twins has been observed as reported in the literature, which is attributed to a lower activation stress compared to the other operable twinning and slip systems. The commencement of mechanical twinning and macroscopic shear bands led to grain refinement due to intersection amongst these twins and twins' subdivision as a result of formation of cross twins, which are evident from the EBSD and TEM results in the present work.

Materials science perspective of metal fatigue resistance

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An interdisciplinary view of metal fatigue in polycrystalline metals is presented. Fatigue resistance is defined in terms of difficulty of crack growth in one of two possible directions, the first being related to the texture of a materialand the second to the orientation of applied loading system. The fatigue initiation phase is considered to be negligible for polycrystalline metals, and fatigue limits are equal to the one of two threshold conditions, one quantified in terms of microstructural fracture mechanics, and the other determined by continuum mechanics. The importance of the intensity and distribution of microstructural barriers to fatigue crack growth is underlined. Especially in relation to mechanical conditions such as stress-strain state and to material conditionsuch as grain size and the shape and orientation of inclusions and their size relative to microstructural barriers.

Studies on hardenability of AISI 9260H and its influence on microstructure

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AISI9260H is potential material for several applications such as springs, wheel, shells etc due to their high wear resistance and resilience. The material is used in spheroidized anneal followed by cold drawing in small section or in the hardened and tempered condition for applications such as automotive leaf spring. The steel has high C [0.65%] and Si [2%] as major alloying and is devoid of other alloying elements. Hence, the response of the steel to heat treatment is of importance. In this study, the effect of hardenability is measured by the Jominy end quench test based on which the hardness distribution in the as quenched bar could be established. The microstructure obtained in jominy sample could be correlated to the microstructure obtained in literature. It was found that as the bar diameter 140mm RCS hardened samples [equivalent dia 150mm] there is a drop in surface hardness that could be correlated with actual hardness obtained using hardenability penetration diagram (U curve). The mechanical properties in various heat treated conditions are also mentioned. The YS/UTS in quench and temper condition was observed to 0.87 compared to annealed and normalized which was 0.53 and 0.63 respectively. The impact toughness of quench temper condition was observed to be approximately twice compared to annealed structure and four times compared to normalized structure.

Effect of thermal and thermo-mechanical processing parameters on the microstructure and properties of the API steel

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The properties of API steels can be remarkably improved through proper selection of a suitable alloy composition and an appropriate thermo-mechanical processing route. The experiments presented in this

work consists of heating the specimens of X60 grade steel to austenizing temperature, isothermal holding at these high temperatures and then cooling at various rates. Final microstructures and hardness values are observede to be influenced by the cooling rate. Specimens have been hot deformed in austenizing temperature with strains of 0.5 with a constant strain rate of 1 s^{-1}. After hot deformation, one specimen has been allowed to cool freely and another has been cooled linearly at a rate of 1˚C/s. It has been observed that faster cooling rate in free cooled deformed specimen has resulted in finer grain ferrite in comparison to the specimen cooled linearly at a rate of 1˚C/s. Experiments were also carried out to see the effect of annealing temperature and time on the microstructure and properties of the heavily deformed specimen. In these experiments, 70% warm rolled specimens have been annealed at two different temperatures of 750 and 850˚C with varying annealing time. The specimens have been allowed to free cool after annealing for microstructure and properties evaluation. It has been observed that the volume fraction of recrystallisation increases with increase in annealing hold time for warm rolled specimens annealed at 750˚C and 850˚C is high enough for the occurrence of recrystallisation and ferrite grain size increases with increase in annealing hold time. It is also observed that hardness values are also influenced by annealing temperature and time.

Microstructural dependence of the thermo-mechanical properties of CRGO steels

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The properties of a material are highly dependent on its microstructural features. This paper aims to study the effect of deviation from the exact GOSS orientation ((1 1 0) [0 0 1]) on the properties of Cold Rolled Grain Orientedsteel. The microstructures were deformed in five different loading directions and further studied using the EBSD (Electron Backscatter Diffraction) technique. The experimental micrographs were imported in the Object Oriented Finite element (OOF2) program for analysis. The finite element mesh was created in the OOF2 software followed by the calculation of thermal flux. The analysis showed a significant dependence of the loading direction and orientation on the property. A mathematical model was developed to introduce systematic orientation variation within the grains. The microstructures obtained by the model were further analyzed using the Object Oriented Finite element (OOF2) program.

Anisotropy in mechanical properties of SS 316L rolled sheet

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Tensile and low cycle fatigue (LCF) behaviour of SS 316L rolled sheet was carried out along specimens extracted along the rolling direction (RD), transverse direction (TD) and 45° to the RD to study the effect of planar anisotropy. The anisotropy in mechanical properties viz. yield strength (Y.S.) and ultimate tensile strength (UTS), strain hardening exponent (n) is studied along different directions. Stress response at constant strain (in LCF) was investigated along different direction. Fractography and width vs. thickness variation of fracture specimen was investigated through scanning electron microscopy (SEM) along RD, TD and at 45˚ to RD. Macro and micro texture was investigated through x-ray diffraction (XRD) and electron back scattered diffraction (EBSD). A detailed analysis of miro-texture and microstructure evolution along different directions is discussed in the present work.

Variant selection and pre-transformation phase reconstruction during deformation-induced transformation in AISI 304 austenitic stainless steel

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Austenitic stainless steels are widely used and give a good combination of properties. When this steel is plastically deformed, a phase transformation of the metastable Face Centred Cubic Austenite to the stable Body Centred Cubic (α′) or to the Hexagonal close packed (Ɛ) martensite may occur, leading to the enhancement in the mechanical properties like strength. The current work is based on variant selection and corresponding texture analysis for the strain induced martensitic transformation during deformation of the parent austenite FCC phase to formthe product martensite HCP phase and the BCC phases separately, obeying their respective orientation relationships. The automated method for reconstruction of the parent phase orientation using the EBSD data of the product phase orientation is done using the MATLAB and various theoretical aspects related to variant selection and transformation. The method of triplets is being used here which involves the formation of a triplet of neighboring product grains having a common variant and linking them using a misorientation-based criterion. This leads us to the proper reconstruction of the pre-transformation phase orientation data and thus to its microstructure and texture. The computational speed of this method is better compared to the previously used methods of reconstruction. The program is checked for EBSD data sets of five samples and the program is giving a similar type of results for all of them. The reconstruction of austenite from ε and α′ martensite was carried out and their texture plots i.e. ODF and the inverse pole figures were compared. The comparison gives us the idea for estimating the correct sequence of the transformation i.e.γ → ε → α′ or γ → α′, during deformation of AISI 304 austenitic stainless steel.

Phase analysis of twip steel with 3%, 6%, 8% and 10% aluminium with strain

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We investigated Fe-26Mn-xAl-1C steels with different aluminium concentrations (3%, 6%, 8%, and 10%) in stacking fault energy (SFE) of 37-60 mJ/m^2. The steels (3Al, 6Al, 8Al, and 10Al) were prepared by induction melting, cast to rectangular ingots and then hot rolled. Interrupted tensile test was done on the samples up to fracture stepping at 0, 5, 10, 30% strains in between and X- Ray diffraction test was done on the strained samples using diffractometer. The results were obtained in the form of relative intensity of diffracted beam vs. 2Theta (40° to 120°). The results were then analysed using Material Analysis Using Diffraction (MAUD) Software. Various parameters linked with the software were refined. After complete refinement we obtained proper fit of the curves. We found that 3Al, 6Al, 8Al TWIP steels are austenitic whereas 10Al TWIP steel contains traces of carbide as well, at room temperature. For the refined samples, values of Lattice Parameters, crystallite size and micro strain of every strained sample for each reflecting plane of FCC system (i.e. (111), (200), (220) and (311)) were plotted against percentage strain and the variations in the plots were observed and the results were concluded.

Evolution of microstructure and texture for AA 7075 alloy during homogenization

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Al–Zn–Mg-Cu age-hardening alloys with high strength and low density are widely used in aerospace applications. Usually, the residual coarse intermetallic particles deteriorate the strength and toughness performance. The chemical composition, casting and homogenization process also influence the volume fraction, morphology, and intrinsic character of coarse particles. The evolution of intermetallic particles was studied by SEM, and XRD in detail. In commercial Al–Zn–Mg–Cu alloys, several eutectic structures between 400°C and the solidus can occur during solidification, which are often $MgZn_2$. A phase transformation of primary intermetallic phase from $MgZn_2$to Al_2CuMg is observed during homogenization and $MgZn_2$phase gradually dissolved in the matrix. In the present investigation, AA 7075 alloy has been subjected to homogenization for different time, the change of crystallographic texture along with the effect of particles from casting to different homogenization time have been investigated. It has been observed that change of texture during homogenization is nominal.

Effect of strain path change and annealing temperature on microstucture and texture evolution of deformed 5083 aluminium alloy

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AA 5083 aluminium-magnesium alloy samples were deformed upto 90% at cryogenic temperature by unidirectional rolling, two-step cross rolling and multi-step cross rolling followed by annealing in the temperature range of 150-400 degree Celsius for an hour. The effect of strain path change and temperature variation on the evolution of microstructure and texture is investigated using optical microscopy, X-Ray Diffraction and Electron Back Scatter Diffraction. Annealing treatment at different temperatures yielded different microstructure representing, recovered microstructure as well as recrystallized microstructure with uni-modal and bi-modal grain size distribution. Tensile test and hardness test have been carried out to study the effect of cryo rolling and annealing on the mechanical behaviour of aluminium alloy sample. Strategies of optimizing strength and ductility by tailoring the crystallographic texture of the samples will be discussed.

Strontium as a modifier for Al-Si Alloy

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This paper presents a study of the influence of strontium on the structure of Al.Si10Mg alloy. Changes are observed in the morphology of eutectic silicon and intermetallic phases. Effect of strontium on structure defects, mainly cavities formation is also observed.Mechanical properties of Al-Si casting alloys, especially

elongation, depend on alloy structure and on the eutectic silicon which may have an acicular or lamellar form. Elongation is improved when Sodium is added to Al-Si alloys to form a fibrous eutectic structure (the major drawback of this practice is that the effect of sodium disappears rapidly by evaporation or oxidation during the holding of the molten metal).Strontium is also used as modifier of Al-Si-Mg alloys. In this paper we will present the advantages of strontium as modifier over sodium. We will observe and compare the effect of sodium and strontium on the microstructure and mechanical properties as modifiers of Al-Si alloys and the modification effect of various modification agents containing strontium in various amounts. This paper confirms the influences of strontium on intermetallic phases containing iron and on structure defects of Al-Si alloys.

Effect of friction stir process parameter on the texture of Mg-9Al-1Zn alloy

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AZ91 magnesium alloy has been subjected to Friction Stir Process (FSP) under different conditions. Different conditions are achieved by changing one of the two process parameters. First one is the tool rotation speed and the second is tool traverse speed. By maintaining the constant tool rotation speed at 1025rpm, the traverse speed has been varied. Four different traverse speeds under which the AZ91 alloy was subjected to are 50 mm/min, 75 mm/min, 100 mm/min and 125 mm/min. By decreasing the ratio of tool rotation rate/traverse speed it is possible to get reduced recrystallised grains. But its effect on texture of the material will have to be studied. By using Electron Back Scattered Diffraction technique, the texture of four differently FSPed specimen will be analyzed by comparing with each other and also with as cast AZ91 alloy

2D and 3D EBSD characterisation of recrystallisation texture in bulk nanocrystalline Ti-Ta-Nb alloy

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Over the last several years we are involved in the development of α+β Ti-5Ta-2Nb alloy for reprocessing applications and demonstrated that, it is possible to increase the strength of this alloy without sacrificing the alloy's outstanding corrosion resistance. This was achieved by imparting strains as high as 2.3 by cryo-rolling resulting in a bulk nanocrystalline material with favorable grain boundary character. Such a nanocrytalline material was subjected to annealing at 300 ˚C, 400 ˚C, 500 ˚C and 600 ˚C for 5 min, 2 hours and 3 hours. The hardness of the samples was measured for the different samples using the microhardness tester Leitz Miniload 2. 2D and 3D EBSD analysis has been carried out on these treated alloy samples. The measurements and analysis are carried out by FE-SEM and commercially available software. Initial studies reveal an activated behaviour for the hardness from which the activation energy was calculated as 27 KJ/mol. For samples subjected to different annealing temperatures, the hardness values were plotted as a function of annealing durations. A sudden drop in hardness was observed at 5 min duration for the samples annealed at 500 ˚C and 600 ˚C. The combination of prismatic slip $\{10\bar{1}0\}<11\bar{2}0>$ and basal slip $\{0001\}<11\bar{2}0>$ in Ti result in the textures with basal poles tilted away from

the normal direction. The similar texture is observed for the 90% cryo-rolled material before and after annealing. The 3D EBSD analysis is in progress.

Microstructure-texture evolution during thermo-mechanical processing of Ti-5Al-5Mo-5V-3Cr alloy

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Ti-5Al-5Mo-5V-3Cr (Ti-5553) is a high strength β titanium alloy based on the Russian alloy VT22 (Ti-5.7Al-5.1V-4.8Mo-1Cr-1Fe). Due to its ameliorated properties and easier processability, Ti-5553 has become favourablefor aircraft structural applications, such as landing gear components.However, like other β titanium alloys, obtaining uniform recrystallization is not easy for this alloy. Therefore it is very important to find out the thermo-mechanical processing window to obtain uniform recrystallization for this alloy.Hence, the main objective of the present investigation was to develop this processing window by studying microstructural and textural evolution during hot deformation and post-deformation heat treatment in the β phase field. Aspects of dynamic recrystallization, static recrystallization and grain growth have been investigated as a function of thermo-mechanical treatment parameters. Effects of % reduction at the deformation step and temperature of post-deformation heat treatment on recrystallization behaviour have been studied using dilatometer and orientation image microscopy. Dilatometric behaviour of the samples that underwent different % reductions has been related to initial texture present in the deformed samples.EBSD technique has been employed in order to study the evolution of texture (transition from deformation texture to recrystallization texture) and its intensityat different deformation and recrystallized conditions.Vickers micro-hardness and Ultrasonic tests were carried out to correlate mechanical properties with amount of recrystallization. Through this work, a thermomechanical processing window (in terms of % reduction and post deformation heat treatment temperature) has been developed to obtain uniform recrystallization of β grains.

Session 6:Nov 15, 2014:[11:00 - 13:00]

Erosion induced deformed features and microstructural derivatives of AISI 52100 steel

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Solid particle erosion process has been found to be constituted by breaking tendency and shearing of martensite lath, tempering to low carbon martensite, precipitation of nano carbides, restoration of dislocation by extensive dynamic recovery and normal growth of lath martensite. Martensite lath boundaries have been found to be dissociated with the precipitation of nano carbides. The erosion has been observed to be ductile erosion. The ductility and toughness enhancement by the extensive dynamic recovery of martensite have been found to increase the erosion resistance. The characteristic microstructural derivatives like dynamic recovery and normal lath growth of martensite against pinning by nano particles have been p. The dynamic recovery of stressed and curved dislocation network is defined by extrinsic properties like temperature, strain rate and intrinsic properties like orientation based Taylor factor and stacking fault energy or equilibrium distance of separation of partial dislocations. Deformed features like low angle boundary and shear band developed during erosion have been investigated. Larger strain rate and greater degree of plastic deformation of normal erosion compared to oblique erosion resulted in greater extent of dynamic recovery and shear band development though the abundant shearing is totally restricted. Oblique erosion has revealed higher fraction of low angle boundaries.

Influence of cryogenic treatment on wear and hardness behavior of SAE 8620

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Gear materials like SAE 8620, 20MnCr5, AISI 4340, En-353 are widely used typically in hardened and tempered state and case carburized condition. Sub-zero treatment for enhancing the properties of the material is not yet fully explored and a limited level of standardization is realized by the user of this particular process. Sub-zero treatment has shown encouraging response to dimensional stability, elimination of retained austenite, fatigue life, low coefficient of linear thermal expansion and wear resistance. In the present research work, hardening of SAE8620 was done in a laboratory muffle furnace at 920°C for 25min followed by tempering at 300°C for two hours in a separate muffle furnace. Specimens were then subjected to post-subzero treatments in a cryoprocessor maintained at -185°C for varying soaking times from 8 to 24hrs. Post- subzero treatment, all specimens were subjected to soft tempering in the muffle furnace at 100°C for 1hr.Pin-on-disk wear testing machine was used to measure the loss of material. Wear test was performed by taking constant load of 6kg and constant sliding velocity 2.5m/s. Wear loss was recorded as an average of three readings. Hardness measurement was done on Rockwell hardness testing machine, C-scale. An optimum cryosoaking time was established at 16hrs based on wear and hardness results.

Controlled reductionof Cu Coated SiO_2particles in Albased composite

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Particle reinforced Metal Matrix Composites (PRMMCs) with metal coated reinforcements have excellent wettability with matrix resulting in enhanced mechanical properties. The intention of present study is to carry out the quantative analysis of wear debris, wear rate and coefficient of friction of Al based MMC reinforced with Cu coated SiO_2 particles extracted from rice husk ash (RHA). The reactivity and amorphosity of the RHA SiO_2 is highest as compared to commercially available SiO_2. The worn out particles from the composite as debris were characterized quantitatively using a scanning electron microscopy (SEM) coupled energy dispersive spectroscopy (EDS). Comparative X-ray diffraction (XRD) analysis of composites was carried out to investigate element and phase analysis of wear debris. It was observed that matrix–particle interaction coupled with lack of (?) wettability in the particles/matrix interface is responsible for particle debonding and surface damage of MMC. Results show that composite with Cu coated SiO_2 particles as reinforcement have homogeneous distribution of SiO_2 particles and better interface with matrix alloy. When Mg reaches 1.5 wt%, wettability of matrix with reinforcement is maximum, leading to formation of hard compounds such as CuO, $CuAlO_2$ and Al_2CuMg at interface. This enhances interfacial bonding strength as well as load transfer from matrix to reinforcement, which leads to higher tensile strength along with ductility.Thus by making thebestuse of RHA SiO_2 in synthesizing the composite ,the wear behavior of composite reinforced with Cu coated SiO_2 particle was observed to be superior in comparison to uncoated SiO_2 particles.

Effect of boron and carbon addition and heat treatment on wear behaviour of Ti-10V-2Fe-3Al

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Titanium alloys have been of great interest in recent years because of their very attractive combination of high strength, low density and corrosion resistance. Application of these alloys in areas where wear resistance is also of importance calls for thorough investigation of their tribological properties. In the present study, the effect of addition of boron and carbon and heat treatment (solution treatment and solution treated plus aged conditions) on dry sliding wear behaviour of Ti-10V-2Fe-3Al alloy was carried out in order to highlight their wear behaviour under different loads (20-50N) and sliding speeds (0.5-1.5 m/s). Sliding wear tests were carried out using pin-on-disc wear and friction monitor for a total sliding distance of 1000 m. The weight loss, coefficient of friction and maximum temperature attained at the disc-pin contact surface were measured. The composition and morphology of wear debris collected was analyzed using XRD and SEM-EDAX. The worn surface of the selected pins was observed by optical microscopy and SEM. The addition of boron and carbon and heat treatment has affected the weight loss, coefficient of friction and maximum temperature attained at the disc-pin contact area.

DC and pulsed electro-deposition of Cu-Y_2O_3 composite coating - a study of process and surface mechanical properties

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Cu-Y2O3 composite coatings were electrodeposited from acidic baths containing copper sulfate and sulphuric acid with different concentrations (0, 10 and 30g/l) of Y2O3 nanoparticles. The whole process was carried out by standard current density 8A/dm2 at room temperature. The objective of this study was to improve the surface mechanical properties of Cu without much decrease in electrical conductivity by DC and pulse electroplated Cu-Y2O3 composite coatings. By Zeta potential measurement the iso-electric point of the procured ultrafine powder (Y2O3) was carried out prior to deposition. The microhardness and wear properties of deposited samples were studied systematically. The microstructure and phase analysis of the composite coatings were characterized using Field-emission Scanning Electron Microscopy (FESEM) and X-ray diffraction (XRD). Energy dispersive X-ray spectroscopy (EDXS) technique was also used to investigate the chemical composition of coating samples. Electrical conductivity measurement was done for all the deposited samples to check whether the value decreases rapidly in comparison to pure copper with concentration of second phase particle. From the results it was observed that the microstructure and performances of the coatings were greatly affected by Y2O3 content on the deposits prepared by DC and PED methods. Higher hardness was found in the optimum percentage of Y2O3 composite coatings. The PED composite coatings exhibited compact surface, higher microhardness compared with that of the DC composite coating. The proposed synthesis method is novel and cost effective.

Characterization of plasma sprayed AlCoCrFeNi high entropy alloy coatings

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High Entropy Alloys (HEAs) are a new class of alloys with multi-principle elements in equi-atomic ratio, which exhibit novel phases and outstanding properties. Due to their high temperature microstructural stability, enhanced oxidation and wear resistance, these HEAs are also considered as surface coatings for niche applications. One such potential application could be as a bond coat material in thermal barrier coatings of turbine blades in aero engines that are exposed to temperatures greater than 0.7 Tm. In the present study, mechanically alloyed equiatomic AlCoCrFeNi HEA powders were plasma sprayed in ambient atmosphere onto a mild steel substrate. Milled powders and coatings were characterized by XRD, SEM, and Vicker's hardness. The XRD patterns of the mechanically alloyed powders showed predominantly a BCC phase with minor FCC phase, while the coating revealed a major FCC phase with minor BCC phase besides oxides of Al and Cr. A detailed chemical analysis of the phases by EDS and EPMA analysis, and the

BSE-SEM phase contrasts together were correlated with the phases observed in the XRD pattern. The coatings have a porosity of 9.5 ± 2.3% and Vicker's hardness (HV0.3kgf) of 4.13 ± 0.43 GPa. Knoop hardness measurements on coatings suggest an anisotropic mechanical behavior in the coating due to its lamellar, composite type microstructure.

Mechanical property evaluation of neutron irradiated stainless steel components by remote tensile testing

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Cold worked austenitic stainless steel type AISI 316 is used as the material for fuel cladding and wrapper of the Fast Breeder Test Reactor (FBTR), India. Fuel pin consist of fuel encased in a clad tube which forms the first barrier and hexagonal wrapper tube housing a bundle of fuel pins forms the basic handling unit of fuel in the reactor. The evaluation of mechanical properties of these core structures is very essential to assess its integrity and ensure safe and productive operation of FBTR to very high burn-ups. The changes in the mechanical properties of these core structures are associated with microstructural changes caused by high neutron fluence and temperatures of 400–550 °C. Since the core components are highly radioactive, they are remotely handled and tested in shielded enclosures called hot cell using Master Slave Manipulators (MSM). The stress-strain curves of the irradiated AISI 316 steel is a function of the atom displacement damage, irradiation temperature and test temperature. For irradiation temperatures of less than 500°C, there is generally a hardening behavior with loss in work hardening and ductility, while for irradiation temperatures above 500°C, the extent of hardening decreases with minor recovery of ductility. However, with higher levels of swelling of AISI 316 beyond 60 dpa, there is softening and drastic loss in ductility. The methods of remote tensile testing of reactor core components along with trends of strength and ductility of AISI 316 steel subjected to displacement damage ranging from 2-80 dpa will be presented.

Characterization of high phosphorous containing hot rolled weather resistant structural steels

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Three steels containing higher amounts of phosphorous (G22: Fe-0.072C-0.028Si-0.20Mn-0.19P-0.38Cr-0.24Cu-0.17Ni, G32: Fe-0.072C-0.061Si-0.23Mn-0.19P-0.64Cr-0.14Cu and SCOR: Fe-0.09C-0.37Si-0.42Mn-0.15P-0.37Cr-0.35Cu-0.31Ni) were prepared and characterized for their mechanical, textural and corrosion properties. It was revealed that the YS, UTS and %EL of all the three steels were more than minimum values specified in IS2062 for E300BR grade and EN10025-2:2004 specification for S300JR grade. Charpy V notch impact energy of G32 steel was lower by 20 Joules at room temperature, which could be attributed to higher content of Cr. CVN impact energy of G22 steel at room temperature, in spite of having P content similar to that of G32 steel, was observed as 65±12 Joules and that of SCOR 118±8 Joules, against a requirement of 27 Joules as specified for both the grades i.e. E300BR and S300JR at room temperature. The reduction in area values obtained through hot tensile test performed in the Gleeble between 850-1100°C temperature range were found to be greater than 60% for G22 steel, while for the G32 steel the

minimum value was observed as 36%. Improved ductility in G22 steel is attributed to the beneficial effect of Ni. Electrochemical impedance spectroscopy conducted for measuring resistance of the rust layer formed on the samples exposed for a period of four months to the atmosphere revealed that the impedance of G22 and SCOR steels were similar to that of SAILCOR. In these steels, although Cu and Ni contents are low, compact rust layer formed due to higher P content seems to provide improved corrosion resistance. EBSD studies revealed that γ({111}||ND) fibre content was higher in G22 steel as compared to other grades. The other fibres such as α(<110>||RD), ε({110}||TD), θ({001}||ND) and ξ({110}||η) were also observed in these steels, which are expected in hot rolled grades.

Session 7: Nov 15, 2014:[14:00 - 16:00]

Observation of de-cohesion damage in aluminium metal matrix composite at INDUS-2 imaging beamline using micro-tomography

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The imaging of de-cohesion damage in Al metal matrix composites using micro-tomography has been carried out at India's first imaging beam line at INDUS-2, a 2.5 GeV Synchrotron source. An aluminum metal matrix composite consisting of 20%vol SiC(P) in the extruded form was tested in compression to a true strain of ~ 0.9 – 1.0 over a range of temperatures and strain rates. Small coupons of about 500μm thick were extracted from multiple locations of a set of tested samples and subjected to micro-tomography scans. The tomography scans were carried out using a mono-chromatic beam of energy 25 keV, at an incident flux of the order of 108 photons / sec on the sample. The illuminated volume of the sample was obtained in the form of reconstructed slices dataset which were subsequently visualized in 3D to reveal the nature of de-cohesion damage in terms of its morphology, relative content and spatial distribution. The relationship of the development of the damage, its possible relation with the SiC particles is explored. Statistical and correlative relationships between peak stress and internal damage in the samples are also investigated.

Role and effectiveness of plastic deformation and stress-relief in zirconium: origin of residual stress

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Zircaloy-4 clads subjected to burst test have shown different ductility determined by microstructure and crystallographic texture; but was shown to have a clear scaling with hydrostatic component (Ph) of the measured tri-axial residual stress matrix. The study then tried to bring out the origin & effects of residual stress in zirconium. In rolled Zircaloy-4 a clear gradient of elastic and plastic strains were predicted through finite element simulation. Such predictions were verified against experimental data on microtexture and

bulk crystallographic texture. Micro-focused X-ray also provided clear distinction in residual stress developments between different features of the deformed microstructures. For example, deformed Zirconium grains were classified as fragmenting and non-fragmenting. The latter, mostly basal, had strongest signatures of residual stresses. During recovery, microstructure dependent stress-relief was established. Initial recovery or stress relief was primarily through reductions in orientation gradients in non-fragmenting grains. Only during the latter stages, recrystallization and stronger orientation sensitive stress-relief was noted for the fragmenting grains.

X-ray diffraction study on cooling rate induced $\gamma \rightarrow \varepsilon$ and $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ martensitic transformation in homogenized and forged high manganese steels

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Thermal stability of austenite in high manganese steels has great importance form microstructure aspects that can offer two different mechanisms such as Twinning (TWIP) or martensite (TRIP). In present work thermal stability of some homogenized and forged high manganese steels with carbon content (0.1-0.35 wt %) have been studied through X-ray diffraction technique. The decomposition of austenite was observed to be very sensitive to the cooling rate. A very high proportion (30- to 49 %) of ε-martensites was found in the differently cooled specimens as well in the hot rolled specimens. Formation of (9-20 %) α′ martensite is also found in high manganese TRIP steel after quenching in different media. Significant variation in the extent of γ_{fcc}-ε_{hcp} and γ_{fcc}-ε_{hcp}-α'_{bcc} martensitic transformation was interpreted in terms of athermal and isothermal martensites formed during cooling and grain size of austenite. The austenite grains were found to be comprised of a high to moderate density of stacking faults (~10^{-3} to 10^{-2}) and negligible twin fault probability (~10^{-5}). On the other hand, in the ε-martensites, the dominant planar fault was twins (~10^{-3}). Approximate values of the dislocation density within the retained austenite were also evaluated using the crystallite size and root-mean-square (rms) strain values obtained from the ''size-strain-shape'' analyses.

TEM characterization of gallium nitride grown on c-plane sapphire by MOCVD

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Gallium Nitride based materials have tremendous potential as a futuristic material for optoelectronic and semiconductor applications. Devices based on these materials have the capability of operation in the high power, high frequency domains. Therefore these materials are the ideal materials of choice for the High Electron Mobility Transistors and the high power blue LEDs and lasers. Due to the unavailability of matched substrates, these device structures have been grown on foreign substrate materials such as

sapphire, silicon, SiC etc. Growth on these lattice and thermally mismatched substrates lead to formation of high dislocation densities (~10^9-10^{10}/cm^2) and various other defects.Present study is aimed at understanding the microstructural evolution of Gallium Nitride during the various stages of growth on c-plane sapphire. This study may further enhance our understanding on the genesis of dislocations in these nitride materials. For the current work, GaN growth runs have been carried out in a MOCVD reactor (AIXTRON make) using the standard two step growth recipe under different nitridation temperatures (530, 800 and 1100°C). This study aims to investigate the different stages of GaN growth and understand the nature of the initial low temperature GaN (LT-GaN), its morphology and defect structures. Then the subsequent high temperature GaN (HT-GaN) layers have been investigated for correlation with the underlying LT-GaN, which acts as the nucleation site for the HT-GaN layers grown above it. Microstructural characterization of the LT-GaN and HT-GaN layer under different nitridation temperatures has been carried out. Conventional TEM and High-Resolution TEM and advanced microscopy techniques such as Weak Beam Microscopy, Convergent Beam Electron Diffraction have been used to investigate the microstructure, various defect structures, morphology, polarity etc. of the formed of GaN and its evolution during the different stages of its growth on c-plane sapphire. These will be discussed in detail in the presentation.

Determination of no-recrystallization temperature (T_{nr}) of a pipeline grade steel

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The microalloying elements present in pipe line grade steels form several precipitates in ferrite matrix and lead to strengthening through grain size refinement as well as precipitation strengthening. However, during hot rolling, these precipitates have to be dissolved in order to get a homogenous deformation state of the austenite. During hot rolling, depending on the temperature of rolling, there are possibilities of recrystallization of austenite leading to finer austenite grains. Later with continuing deformation at lower temperatures, there will be formation of elongated grain structure and fragmentation of austenite grains. For an effective ferrite grain refinement, it is therefore very much required to know the rolling schedule at proper temperature region.Hence, critical temperatures of micro alloyed steel such as non-recrystallization temperature (Tnr) to properly plan the rolling schedule. The value of Tnr is also dependent on alloying elements like Nb and deformation parameters like interpass time, deformation strain per pass and strain rate. Several empirical equations have been developed and utilized in the literature and industry. But these equations have severe limitations with respect to its applicability. The present study has been carried out to determine Tnr experimentally by double hit method using a Gleeble® 3800 Thermal mechanical simulator. The samples were deformed at different temperatures by keeping other processing parameters constant. The flow curves were analyzed by different analytical methods to determine the fraction of softening between deformation passes. The Tnr corresponds to fractional softening value of 20% between the two deformation passes in the double hit method. The microstructure of deformed samples was characterized by prior austenite grain size (PAG) etching and Scanning electron microscopy. The Tnr temperature of the pipeline steel is found to be ~900°C as determined by the double hit method and confirmed through the microstructural studies.

Fabrication of sputter deposited single-bi-layered and double-bi-layered Ni/Ti thin film and comparison of their mechanical property

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Nickel/Titanium thin film plays a major role in Nano-electro-mechanical-devices (NEMS) used in a wide range from aerospace industry to many home appliances. In this investigation, Ni/Ti thin film was fabricated with the help of DC/RF magnetron sputtering technique. Layer-wise deposition of Ni and Ti on Si(100) substrate was carried out with Ni as the bottom layer and Ti as the top layer, to ensure its biocompatibility. As-deposited thin film is amorphous in nature. To develop the shape memory effect in the film, annealing was carried out to get a crystalline film. Annealing treatment was carried out by taking four annealing temperatures (300 ºC, 400 ºC, 500 ºC and 600 ºC) with 1 hour soaking time. This presentation explains the fabrication processes and the mechanical characterization details of the as-deposited as well as the annealed thin films. The novel mechanical properties in this material system is described and compared between the single-bi-layered and double-bi-layered Ni/Ti thin film, with the help of nano-indentation and nano-scratch testing. Nano-indentation was carried out by taking a constant load of 6 mN to find out the hardness, percentage of recovery, modulus of elasticity and wear resistance. From nano-scratch testing, we find out the co-efficient of friction of the film under constant load as well as in ramping mode. It is found that the mechanical properties mainly depend on the formation of intermetallics in between layers and some metastable precipitated of phases.

Structural and optical properties of Cr doping ZnO crystalline thin films fabricated by PLD technique

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High-quality Cr-doped ZnO thin films of single phase with preferred c-axis growth orientation were formed on Si (100) substrate by pulsed-laser deposition. The structural analyses demonstrate that all samples have wurtzite structure and are preferentially oriented along the c-axis perpendicular to the substrate surface. The intensity of defect-induced mode frequency for Cr-doped ZnO film increased showing the formation of such high-quality crystal. CrZnO thin films consist of nanorod-shaped grains with different sizes. Each of rods has a width in the range of 85-93 nm and a length up to several hundred nm. Photoluminence spectra of the films confirmed PL characteristics with the peaks in UV and defect-related deep level emission in visible region (blue, violet, green) between 419 to 398 nm and 450 to 546 nm. In the visible region of the PL spectra, the green light emission peak of samples at about 567 nm was associated with defects in ZnO. It is observed that RMS roughness increases with Cr doping which means the surface becomes rough and grain size increases. Thus it reveals that our fabricated thin films can be used in different applications like dye sensitized solar cells (DSSC), catalysis etc.

Small punch tests to evaluate creep properties of P92 steel

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Small punch creep (SPC) testing technique has been employed to measure the creep properties of grade P92 steel at 600°C at various load levels. The results of SPC creep tests have been compared with conventional uniaxial creep tests to determine the correlations between the parameters obtained from the two techniques. The creep curves obtained by SPC tests were similar to that of conventional uniaxial creep curves characterized by primary, secondary and tertiary stages of creep. The minimum creep rate obeyed Norton's power law and exhibited a two slope behavior. The values of the load exponents in the low and high load regimes were in accordance with the conventional creep data. This two slope behavior has been explained on the basis of the change in creep deformation mechanism with a decrease in load. The fracture mode in SPC specimens changed from ductile transgranular to brittle intergranular with a decrease in load level. Simple empirical correlations were established between SPC load and minimum deflection rate with uniaxial stress and minimum creep rate. This study demonstrates SPC as a reliable technique for evaluating creep behavior of materials.

Ductile tearing resistance of steel sheets from tensile tests

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A multiple gage length tensile testing procedure has been proposed in the literature for evaluating the ductile tearing resistance of sheet materials with fixed initial thickness. An alternative proposal is to use the energy absorbed from onset of necking to fracture in a tension test as a measure of resistance to ductile fracture. However, in tension tests with sheet specimens, while the onset of necking coincides with the load maximum, it is initially extremely diffused, and the overall neck development shows significant statistical scatter. The authors therefore argue that the energy absorbed in the specimen from the onset of significant necking to fracture should yield a more realistic measure of the resistance to ductile tearing. They also proposed two different criteria to identify the point of significant necking from test data, and identify one of these as a very simple yet practical alternative that can be easily adopted. In this paper, viability of the new proposed parameter is further established by a new testing protocol, specifically, by testing a series of specimens with the same gage length but with different widths. The results confirm that the new parameter can in effect be considered to be a material parameter (for a given sheet thickness). This new parameter also proves considerably superior to the result from multiple gage length tensile tests. The reasons thereof are discussed. The test material is a DP 590 grade automotive steel sheet with thickness 1.53 mm. The tests have been carried out with specimens of both longitudinal and transverse orientations.

Characterization of solid-state bonded high strength steel

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Joining of high-carbon steel is challenging because of inherent lack of weldability through conventional fusion welding. Diffusion bonding (DB) is a potential alternative. A diffusion bonded high strength steel joint was characterized, in this study, to determine the distribution of alloying elements along the joint and intermetallic formation. A Cu based alloy of composition Cu-40Mn-10Ni (wt.%) in the form of foil was introduced as an interlayer in diffusion bonding of high strength steels at 900°C for 35 min. under uniaxial load of 40 MPa. Optical microscope and FEG-SEM were used for microstructural characterization of the joint along with the effect of diffusion of chemical species in reaction zone using point analysis in EDAX. Line profile analysis was obtained by EPMA. Microstructure of substrate was changed from tempered martensite to austenite at bonding temperature and subsequently to ferrite–pearlite during cooling to ambient temperature. The thickness of the diffusion bonded zone was observed around 25 µm in optical micrograph. There is a thin diffused layer close to each of the steel substrates with approximately 5 µm thickness which mainly comprises of Fe3Mn7 phase. Mechanical properties such as yield strength and elongation were evaluated by performing tensile test. Ultimate tensile strength of ~244 MPa was obtained along with 1% ductility. Fractography was carried out using FEG-SEM equipped with EDS; while phase analysis was done using XRD. Diffusion of alloying elements from the filler and the substrate promotes intermetallics formation in the diffused zone lowering the bond strength. Diffusion zone exhibited formation of intermetallic compounds (mainly Fe3Mn7) along with some carbides (mainly Cr7C3). Vickers microhardness profile revealed the brittleness of the intermetallic. These and several other microstructural features and mechanical properties are discussed in light of experimental evidence and relevant analyses of similar work.

Optimisation of charge composition for the production of silicon carbide via carbothermal route

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Silicon carbide (SiC) finds applications in various fields such as cutting tools, refractories, composite armour, heating elements, automobile parts, steel making. It is a well-known fact that more than 95% of the world's SiC production comes via carbo-thermal reduction process. In carbo-thermal process, the raw materials used are carbon and silica and a mix of this is placed around a graphite rod and a massive current is passed through the same.Heat is generated at the surface of the electrode which facilitates the reaction. The temperature at the electrode reaches more than 2000°C during the production and CO gas is evolved during the reaction which is burnt at the top of the charge and is open to atmosphere.This process is also known as the Acheson process. However, the process efficiency is low which can be improved. Production of SiC using the Acheson process depends upon the various parameters like reaction

temperature, reaction time, charge composition, reaction kinetics and mode of power supply. In the current study, the charge composition of the raw materials has been optimised using the Acheson process. To estimate and characterise the SiC content at the end of the experiment, two techniques have been employed which are Chemical Analysis and X-Ray Diffraction. Quantitatively, SiC percentage has been determined using chemical analysis. The phase information and morphology of the product has been obtained using X-Ray Diffraction.

Development of niobium silicide based alloys for high temperature applications

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Niobium silicide based alloys have been considered as potential candidate materials for next generation gas turbine applications where the operating temperature is much higher than the stability range of currently used nickel base superalloys. This is due to their higher temperature stability and low density in comparison to the conventional nickel base superalloys. In the present study, the effect of Ta, Ni, Zr additions on the microstructure and mechanical properties of Nb-16 at% Si has been investigated. The alloys were prepared by non-consumable arc melting furnace under argon atmosphere. The chemical compositions of the alloys were determined by SEM combined with EDS technique. The volume fraction of the constituent phases was determined by using image analyzer on the backscattered electron images. It has been observed that addition of Ta does not show any significant influence on the microstructure, where as Ni and Zr show a drastic change in the microstructure and morphology of the phases. The XRD pattern of Nb-16Si-3Ta and Nb-16Si-3Zr alloys composed of NbSS and Nb3Si phases similar to that of Nb-16Si, while an additional new phase of Nb4NiSi was observed in the Nb-16Si-3Ni alloy. The additions of alloying elements led to an increase in the microhardness values of the constituent phases as compared to that of hardness of phases in Nb-16Si alloy.

Effect of ultrasonic shot peening on microstructure and low cycle fatigue behaviour of Ti-6Al-4V alloy

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Ti-6Al-4V is known as work-horse titanium alloy because of its wide applications in aerospace industry, chemical processing plants and medical devices. It has unique combination of corrosion resistance and mechanical properties along with its light weight. It is a two phase alloy consisting of the primary α (HCP) and the metastable β (BCC) phase. A wide range of microstructures can be produced by various combinations of solutionising temperature and the cooling rate. In the present investigation the effect of ultrasonic shot peening (USSP) with hard steel balls of 3 mm diameter has been studied on microstructural modification, surface roughness, hardness profile and low cycle fatigue (LCF) behaviour, following peening for different durations; 5, 15, 30 and 45 minutes. The microstructural modification was characterised using scanning electron microscope (SEM) and transmission electron microscope (TEM). The shot peened surface was examined by XRD to check any phase transformation resulting from shot peening. While there was grain refinement to a large extent from USSP, cracking of the surface was observed due to shot

peening for long durations. LCF behaviour is being studied following ultrasonic shot peening for 5 minutes at various total strain amplitudes ($\Delta\varepsilon t/2$) of ±1.0%, ±0.8% ±0.6%, ±0.5% and ±0.45%. The results will be discussed in terms of increase in resistance against crack initiation and propagation due to proper ultrasonic shot peening and the associated compressive stresses. The effect of longer durations of shot peening on LCF behaviour was also studied.

Effect of aluminum on oxidation & hydrogenation of vanadium

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Vanadium and most of its alloys are generally produced via aluminothermy reduction of appropriate mixture of oxides. The process is highly cost effective. However, the product retains several atom percent of aluminum even after purification by evaporating aluminum using repeated electron beam melting. The data showing the effect of aluminum on the hydrogenation and oxidation of vanadium will help to implement aluminothermy process to produce vanadium and vanadium based alloys for various functional and structural applications. In present paper, V-Al alloys were synthesized by aluminothermy process and refined by electron beam melting. The refined V-Al alloys were characterized by various physico-chemical analytical techniques. Isothermal and non-isothermal oxidation and hydrogenation of these alloys were studied using an electrical furnace and CI microbalance, respectively. Based on the mass gain data, reacted fractions were calculated. The reacted fraction data were plotted against time using various mechanism functions to obtain the thermodynamic and kinetic parameters. The results thus obtained were compared with the pure vanadium metal.

To study the pileup phenomenon in ball indentation test using finite element analysis

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Ball indentation (BI) technique has been effectively used to evaluate the mechanical properties with minimal volume of material. In the present work, ball indentation test carried out using 0.76 mm diameter silicon nitride ball indenter was modeled using finite element (FE) method and analyzed for P92 steel (9Cr, 0.5Mo, 1.8W). The effect of test temperature (300 K and 923 K), tempering temperature (1013 K and 1053 K) and coefficient of friction (0.0-0.5) on the strength and pile up has been studied. The decrease in strength with increase in pile up height, test and tempering temperature has been observed. The decrease in pile up height and increase in strength was observed with increase in coefficient of friction from 0 to 0.3. Further increase in coefficient of friction from 0.3 to 0.5, resulted in no significant change in pile up height and strength. The results obtained from FE analysis have been validated with experiments from BI test.

Thermodynamic modeling of Mg-Zn-Ca-X (X=Fe,Mn,Ag) based glass forming compositions for biomedical applications

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Magnesium (Mg) based metallic glasses are gaining considerable attention as biodegradable implant material owing to their superior strength, elasticity and higher corrosion resistance than their crystalline counterparts. Though various Mg based metallic glasses have been developed, based on the physiological biocompatibility of the alloying elements, it is the Mg-Zn-Ca metallic glass system which is concurred as a favorite choice for bioimplant applications. These bulk metallic glasses (BMGs) of few µm thickness are not sufficient enough to make possible orthopedic implants. To achieve maximum diameter the alloy composition should embrace a high glass forming ability (GFA). Addition of alloying elements has a vital effect in improving the glass forming ability of Mg-Zn-Ca system provided the alloying elements are biocompatible. The present work involves exploring unidentified biocompatible amorphous forming compositions in Mg-Zn-Ca-X (X=Fe,Mn,Ag) quaternary systems using a newly modeled thermodynamic parameter P_{HHS}. P_{HHS} which incorporates all the three pivotal effects (i.e. electron transfer effects, effect of atomic size mismatch and effect of randomness) can be visualized as the energy barrier that exists between the transformations of random atomic structure of glass to ordered crystalline structure. Thus, when the P_{HHS} value is more negative, the energy barrier will be high, supporting easy glass formation. Validation of results is done through synthesis of the high glass forming compositions under controlled melting and characterization using X-ray diffraction (XRD), differential scanning calorimetry (DSC) and transmission electron microscopy (TEM). This thermodynamic model promises to serve as an effective tool in exploring ideal Mg-Zn-Ca-X compositions which show high glass forming ability paving the way for easier experimental validation.

Corrosion behavior of Mg-AE42 alloy

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Rare-earth containing Magnesium alloy AE42 (4wt% Al, 2wt% RE) exhibits enhanced creep resistance during exposure to temperatures found in automotive powertrain applications. Due to its low density, relatively high room temperature strength arising from precipitation strengthening, and its creep resistance between 150 – 175 ºC, this alloy is of great interest to automotive component/materials designers. The present work reports on the corrosion behavior of the as-cast AE42alloy in freely aerated 3.5% NaCl. Immersion test, salt spray test (fog test) and dynamic polarization test were carried out. Polarization test was done in two solutions (3.5%Nacl (pH-7-8) and 0.5N NaOH (pH- 12.5)) at room temperature. Electrochemical impedance spectroscopy (EIS) and polarization tests revealed active behavior of the alloy. Optical microscopy (OM) and scanning electron microscopy (SEM) were carried out

to investigate microstructural evaluation and corroded (oxide) layer. After immersion test and salt spray test, the oxide layer was studied by X-ray diffraction (XRD) and Fourier transformed infrared spectroscopy (FTIR). It was observed that the corrosion behavior was governed by alloy microstructure, and it varied based on the casting location from which a sample was extracted. The oxide film characterization indicated the corrosion mechanism involved microgalvanic cell formation, where the rare-earth containing precipitates acted as cathodic sites. The corrosion resistance of the AE42 alloy was found to be superior compared to some of the commercial Magnesium alloys.

ADVANCED NIOBIUM STEELS FOR MODERN AUTOMOTIVE APPLICATIONS

Session 1: Nov 13, 2014:[13:30 - 15:30]

Nb-bearing steels for automotive forgings

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The forgings used for automotive components are traditionally made from medium carbon high manganese steels that are quenched and tempered after the forging operation. This heat-treatment entails appreciable expenditure, reduction in productivity and even more important, causes distortion that is difficult to deal with. Over the past years, good efforts have been made for developing non-heat treated grades. Further, the billets go through a complete change in the as-received microstructure during the forging operation as the temperature involved is upwards of 11800 C and is associated with considerable grain growth. This presentation would initially deal with the current status of micro-alloyed and in particular Nb-bearing grades in order to improve strength-toughness and a host of other properties. An insight would be provided initially into the role of Nb in the forgings containing varying amounts of carbon and at various temperatures. The role of Nb in solution and its effect in retarding the austenite grain growth through solute-drag effect is still to be utilized in a quantified manner. Another effect of Nb is the austenite-conditioning that is worth re-examining since the subsequent ferrite formation temperature and its size would be influenced, apart from the nature and quantum of Nb-bearing precipitation. The synergy between some of the alloying elements (in particular, Mo & Si) and Nb in bringing about relative precipitations in austenite and ferrite would be looked into. Some of the newly acquired data would be presented on Nb-bearing forging grades based both on the medium carbon as well as low carbon steels. Lowering the carbon to low values (< 0.1 %) for auto tempering and very low (< 0.01 %) values for introducing cooling rate insensitivity (useful for large forgings) would be examined.

Advanced niobium steels for modern automotive applications

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High strength Low alloyed steel (HSLA) is the most promising option for light weighting in automotive industry and achieving the target weight owing to their low cost impact. Though HSLA steels offer advantages for weight reduction and increase in crashworthiness and safety, there are several issues to be addressed in forming HSLA. During the manufacturing process, materials undergo various forming operations such as, stretching, bending, deep drawing and flanging. Many times, there will be combination of above effects, while processing a component with intricate shapes.The choice of micro-alloying element to use in a steel is strongly influenced by the solubility of the micro-alloy carbide or nitride. Vanadium, Titanium and Niobium are the most familiar micro alloying elements which imparts the increased mechanical properties on the steel. Though fatigue strength of various micro alloyed steel with similar strength possess closer values they differ significantly in their forming and welding characteristics. In the

present investigation forming and welding characteristics of niobium added HSLA was assessed and compared with other micro alloying added steels and dual phase steel. The results showed that niobium added HSLA possess higher hole expansion ratio compared to other micro alloying added steels. This resulted in excellent stretch flangeability of the material with insignificant forming cracks. Additionally niobium addition yields good improvement in the resistance welding of the steel. The results will be discussed with few case studies in detail.

Metallurgical aspects of production ofhigh strength steel strips for automotive application

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Proportion of Advanced High Strength Steel and Ultra High Strength Steel in automotive industry is on the increase, driven by the requirements of better fuel efficiency, stringent emission norms and increasing safety standards. To be successfully used in the body parts of the automobiles, the AHSS sheets should also possess good formability in addition to high strength. The combination of strength and formability can be controlled by the controlling the fraction of various phase components and their individual composition. Solid solution and dispersed precipitated strengthened steel possess high strength but often lacks good stretch formability. Dual phase steels provide good combination of formability and higher strength, with relatively lower yield point. In dual phase steels, the strength of the martensite phase itself depends on the carbon content among other chemical compositions. The carbon content of the both ferrite and martensite phases can be controlled by suitable partitioning of the phases during processing. Most modern hot strip mills are provided with advanced run-out table cooling controls capable of high cooling rates and divided quenching. In case of cold rolled sheet processing, continuous annealing provides high cooling rates and over-aging capabilities to produce high strength dual phase and complex phase steels. At JSW steel limited, Vijayangar works, hot rolled high strength steel strips with tensile strength more than 780MPa were developed Cold rolled strips with properties more than 980Mpa have been developed and produced in the recently commissioned continuous annealing line. For each of the tensile strength levels, steel with different yield strength were developed to suit the various automotive applications. To obtain steel with properties beyond 600MPa, multiple strengthening mechanisms are adopted depending on the type of steel. To achieve higher strength levels, the strength of the ferrite matrix was increased by solid solution strengthening and carbide precipitate dispersion, along with higher fraction of martensite. The metallurgical aspects considered and adopted during production of these high strength steels, considering the various forming processes the steel sheet is subjected to, shall be discussed.

Close control of chemistry, process parameters and mechanical properties of Nb micro-alloyed steels at JSW steel, Dolvi

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Hot rolled strip/sheets are generally used for direct applications in automobile, structural and engineering industries. There is strong demand for steel with high tensile strength coupled with high formability and good impact toughness for this application. Micro alloying with Nb, V & Ti singly or various combinations

have made possible to develop many grades of steel for automotive, engineering and construction use. JSW Steel, Dolvi has also developed a wide range of HSLA steel grades for wide ranging application with properties much superior than similar steel grades without micro alloying. This paper discuss about the various Nb - bearing grade which has been developed at Dolvi, how close chemistry (especially Nb) and process parameters (especially FT/CT) is maintained to achieve a close range of mechanical properties and microstructure.

Increasing transportation efficiency by usinghigh strength niobium microalloyed steel for truck dumpers

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Companhia Brasileira de Metalurgia e Mineração, CBMM, has a strong, sustainable program from the mine to final niobium products and their respective applications. As a company that has fully integrated the sustainability concept, one of the main tasks CBMM continuously pursues is the reduction of carbon dioxide emissions at its Araxá works in Brazil. Transportation is always considered and CBMM's fleet is constantly upgraded to optimize fuel consumption and carbon dioxide emissions.This paper presents an example of a project that reduces the weight of CBMM mining dump trucks up to 25% by shifting from carbon steel to high strength niobium microalloyed steel. This weight decrease resulted in reductions of 3.7% in fuel consumption, 3% in carbon dioxide emissions and 2.2% in total transport costs during the first four months of operation. Due to these improvements, the investment in new trucks with light design achieved payback in less than three months.

Niobium microalloying for press hardening steels

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Press-hardened (PH) steels with ultra-high strength up to 1500MPa have provided carmakers with new opportunities to achieve the lightweight design. From the present state of the art usage of 26% PH steels in the car body, it is predicted to reach 40% in the future. Although the alloying concept of the standard press hardening steel 22MnB5 was not originally designed for automotive application, they are increasingly being used for crash-sensitive parts such that it is highly dependent on its toughness, bendability and resistance to delayed hydrogen induced cracking. Based on these considerations the potential problems of the conventional alloying concept will be discussed and some findings related to a better crash performance by niobium-microalloying will be presented.

CASTING AND SOLIDIFICATION

Invited talks

Session 1: Nov 13, 2014:[09:00-11:00]

Neutron diffraction: a method revolutionizing light alloy development

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Light weighting is a key aspect of component design for automotive and aerospace applications to improve vehicle performance and reduce greenhouse gas emissions. For lightweight alloys such as Al and Mg, challenges arise in casting and processing. For instance, many Al alloys are prone to hot tearing. Understanding of the mechanism of hot tear formation is important in ensuring the elimination of this defect. Neutron diffraction has provided a method to quantify and determine the factors that trigger hot tear formation. Furthermore, neutron diffraction has provided critical information assisting with the optimization of heat treatment parameters for elimination of cylinder bore distortion in Al engine blocks with cast iron cylinder liners. In addition, neutron diffraction has been applied to examine the solidification characteristics of Mg alloys. This novel approach has elucidated the changes in solidification with inoculants and solute additions to enabling improved castability and understanding of defect formation in Mg alloys.

Session 2: Nov 13, 2014:[13:30-15:30]

Glass forming ability and structural stability of rapidly solidified Fe-Zr-B-Cu alloys used for energy savings applications

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Recent trend in developing soft magnetic alloys for energy savings applications is to incorporate nanocrystalline phase in an amorphous matrix. These alloys are generally produced by controlled heat treatment of precursor amorphous alloys in the form of continuous ribbons with good quality surface finish, which are prerequisite for the final applications. Examples of such alloys are Fe–Si–B–Nb–Cu, Fe–Zr–B–Cu and Fe–Co–Zr–B–Cu. Out of these materials, combination of saturation, coercivity and permeability properties in Fe-Zr-B-Cu alloys containing α-Fe nanograins in the amorphous is better than other alloy systems. The present investigation deals with the rapid solidification of Fe99-x-yZrxByCu1 alloys with x+y = 11 and x+y = 13 in the form of continuous ribbons and evaluation of their structure and magnetic properties using X-Ray Diffraction (XRD), Differential Scanning Calorimetry (DSC), Transmission Electron Microscopy (TEM), Vibrating Sample Magnetometry (VSM) and Mossbauer spectroscopy methods. The substitution of Zr by B changes the structure of as spun ribbons from completely amorphous to cellular bcc solid solution coexisting with the amorphous phase at intercellular regions and then to completely dendritic solid solution. The glass forming ability, evaluated from thermodynamic properties is found to be

in agreement well with the experimental observations. On annealing, all amorphous ribbons except the Fe88Zr9B4Cu1, crystallize in two stages; first partially to nanocrystalline α-Fe and then to Fe3Zr from residual amorphous phase. The amorphous Fe88Zr9B4Cu1 ribbon shows three stage crystallization process; first partially to bcc solid solution which is transformed to nanocrystalline α-Fe and Fe2Zr phases exhibiting bimodal distribution, and then to Fe3Zr from residual amorphous phase. The stability of amorphous, α-Fe and Fe2Zr phases in different alloys can be explained from the molar Gibbs free energies (G), calculated as a function of temperature. A detailed phase analysis using Mossbauer spectroscopy through hyperfine field distribution of phases has been carried out to understand the crystallization behaviour.

Session 3: Nov 13, 2014:[15:30-17:30]

Global bench marking in productivity & quality achievement of CSP at JSW Steel, Dolvi

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The process of thin slab casting and subsequent rolling started in 1989 with world's first CSP plant at Nucor-Crawfordsville, Indiana, USA. With its success of operation thin slab casting and direct rolling concepts, technology developed later on. There are 29 CSP plants worldwide.CSP process has typical property features in product coil such as homogenous structural and mechanical properties.JSW Steel (earlier ISPAT Industries) commissioned India's first CSP in 1998 at Dolvi. Second CSP was commissioned in 2003. It has created global benchmark in both single caster & double caster operation with record of highest casting speed. This paper describes the journey of JSW in achieving highest productivity, quality & rich product mix to service wide applications of flat steel products.

Contributory papers

Session 1:Nov 13, 2014:[09:00-11:00]

Microstructure of the Al-6Si and Al-6Si-20Pb alloys prepared by spray forming technique

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In the present work, the microstructural studies of Al-6Si and Al-6Si-20Pb alloys were conducted from top to bottom and from centre to periphery of the spray formed preform. SEM and Optical micrographs were taken at three different locations of the preform viz. (a) top (b) bottom and (c) peripheral regions. The size of the aluminum grains was lower at the peripheral region as compared to the top and bottom regions. Particles/droplets of two different types were found to deposit on the substrate. First type particles were aluminum rich and the second type were lead rich. Aluminum rich particles were coarser than that of lead rich. Lead particles were observed in inter-dendritic regions of the particle microstructure and it was

uniformly dispersed in the spray deposit. The size of lead particles was higher at the centre as compared to that of periphery of the deposit. Some silicon particles were observed to be surrounded by lead at the grain boundary of aluminum phase.

Metal-chill interfacial heat transfer: effect of surface roughness and chill material

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Zn-Al alloys have emerged to be a viable replacement for CI, brass & aluminium alloys in several industrial applications, with Die casting and Permanent mold casting methods being the major manufacturing techniques. An extensive study of various factors governing the metal-mold interfacial heat transfer gradients would be beneficial. Here, the effect of roughness at the metal-mold (chill) interface & the thermo physical properties of chill over heat transfer during upward solidification of ZA8 were investigated. Chills were made of copper, aluminium, bronze and SS304 which offered a wide range of heat diffusivities. Chill surfaces were grounded to roughness varying from 0.1µm to 1.0µm. Heat flux at the interface was determined by Inverse Heat Conduction Method using TmmFe inverse solver software. This experimental setup offered a unique technique to analyze the effect of chill material and its surface asperities over the heat transfer gradients simultaneously. Smoother chill surfaces offered better heat transfer rates as the formation of air gap at the chill-metal interface decreases the peak heat flux across rougher surfaces. Heat diffusivity of chill too was a significant factor as the peak heat flux across the interface increased with the increase in heat diffusivity of chill material. Though aluminium had higher heat diffusivity, heat flux across aluminium chill surface was lower compared to that at brass surface. It was attributed to the insulating oxide layer formed at the aluminium chill surface. Chill surface roughness had significant influence on peak interfacial heat flux for low heat diffusivity chills. On the other hand, mean heat flux over the initial 40s showed that roughness had no effect over heat transfer for low heat diffusivity chills. Mean heat flux increased over 27% with decrease in surface roughness of the copper chill from 1.0 µm to 0.1 µm.

Ultrasonic assisted in-situ grain refinement in aluminium 2124 alloy

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Aluminium, the light weight engineering material with a unique combination of specific properties like strength, formability, durability, corrosion resistance and scrap recycling underpins them in automobile and aerospace, utensils and packaging, construction and equipment and electrical industries. Among them, automobile and aerospace industries share the biggest aluminium markets and recognise its scope economically as well as environmentally. Casting is an industrially versatile and foremost metallurgical process for manufacturing most of the cast and wrought aluminium components. Grain refinement by inoculation is a well-established pre-solidification technique practised in industries to achieve equi-axed grains during casting. Industrial importance of the process has motivated lot of researchers to carry out an

extensive investigation in the field of grain refinement. Of all the refiners, Al-5Ti-1B master alloy with Al3Ti and TiB2 particles is established as the most successful grain refiner for Al alloys. In spite of its grain refining efficiency, problems still exist in the form of toxic fluoride emissions during synthesis of Al-5Ti-1B master alloys by salt flux method. Owing to extensive Al consumption, research and development on eco-friendly grain refiners for aluminium alloys is desirable. Oxide particles are generally considered detrimental during casting. Recent studies on the grain refining performance of Al_2O_3 and $MgAl_2O_3$ particles showed that oxide particles can act as potent nucleating sites in aluminium. In this study, $MgAl_2O_4$ oxide particles were synthesized using SiO2 precursor and their nucleation potency in Al 2124 alloy was investigated with and without ultrasonic treatment. Compared with conventionally synthesized $MgAl_2O_4$ particles, the size distribution and the interface of ultrasonic assisted $MgAl_2O_4$ particle was found to be narrower and clean, thereby enhancing their grain refining performance in Al 2124 alloy.

Thermodynamic prediction of semi solid processing range forMg-Al-Sr-Ca alloys

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Semi solid processing is a growing interest in automobile industries due to its ability to produce near net shaped parts at a high production rate, improved mechanical properties with tight dimensional control. Recently, research is directed towards developing high performance magnesium alloys which are amenable to semi solid processing. Among various magnesium alloys, Mg-Al-Sr-Ca alloy possess better mechanical properties both at room and elevated temperatures. However, very little is understood in terms of semi-solid processing of Mg-Al-Sr-Ca alloy. In this study, an attempt has been made to theoretically predict the semi-solid processing capability of Mg-Al-Sr-Ca alloy under equilibrium and non-equilibrium solidification condition. Semi solid processing criteria such as solidification range (ΔTS-L), temperature process window (ΔT0.3-0.5) and fraction liquid sensitivity (dfl/dT) of Mg-Al-Sr-Ca alloy has been thermodynamically predicted based on CALPHAD approach. Effect of chemical composition on semi solid processing parameters is analyzed using Thermo Calc software with assessed magnesium alloy database. The non-equilibrium solidification condition is estimated using the Scheil-Gulliver simulation. Thermodynamic prediction reveals that with increasing strontium content, the fraction liquid sensitivity decreases and the solidification range and temperature process window increases. Therefore, higher strontium content facilitates the semisolid processing capability of Mg-Al-Sr-Ca alloy. The solidification range (ΔTS-L), temperature process window (ΔT0.3-0.5) and fraction liquid sensitivity (dfl/dT) of Mg-4Al-2Sr-1Ca alloy are found to be 115.1oC, 28.9oC and 0.0069/oC, respectively. Based on the simulation results, Mg-4Al-2Sr-1Ca alloy can be considered as one of the most promising Mg alloy for semi solid processing.

Modeling and experimental validation of deformation of Al-33Cu droplet during impingement on a substrate

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Impingement of a molten Al-33Cu droplet over a substrate causes formation of a splat after spreading and solidification. Modeling of heat transfer and fluid flow during impingement and solidification of metal droplets on a substrate is helpful for better understanding and control of related spray casting and thermal spray coating processes. Authors have thus developed a comprehensive mathematical model of droplet impingement using FLUENT 6.3.16 platform. The model takes into account all the inter-related phenomena, viz., (a) heat transfer through the droplet, solid substrate, solid-droplet interface and adjacent gaseous atmosphere, (b) fluid flow in the droplet and adjacent gas and (c) solidification. The droplet undergoes very large and rapid deformation during the impingement. The model predicts the deformation as well as thermal and flow field within the droplet. Present work reports the experimental validation of model predictions on the droplet deformation, which was carried out using a high speed camera.

Thermal stress modelling in high speed twin roll strip casting of Al-Cu alloy

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Twin-roll strip casting is a near net shape technology for the production of thin strips directly from the liquid metal by combining casting and rolling into a single step. The process provides better control over the microstructure and mechanical properties of the cast strip. It offers advantages of low capital investment and low operational cost and the strips produced have a refined solidification microstructure, which has attracted interest of global metal producers. Twin roll casting technology can produce thin sheets of materials and thus one can surpass the need of rolling etc. However one needs to control the final structure of the twinned rolled products. This can be done by controlling the cooling during solidification. In the present work, a three dimensional steady state thermal model was developed by using commercial package ANSYS 15. From the simulation results, the thermal stress inside the molten pool as well as on the roll surface, which arises from the thermal gradient and solidification profile of the liquid metal inside the molten pool are studied.

Effect of inoculation and mould materials on the structure-properties of thin wall ductile iron

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The use of Ductile Iron for light weight automotive components has limited in the past by the capability of the foundries to produce as cast carbide free thin-wall (1.5-3mm) parts. For almost a decade, the Ductile Iron industry has invested significant amount of money and time to develop a technology that would allow the manufacture of thin walled ductile iron castings for automobile parts. A key parameter for the production of thin-wall casting is to control the nodule count in a range of 500-700 per square mm for which the mechanical properties and the microstructure are optimum. This is obtained by controlling the cooling rate of the parts and optimizing the inoculation process. This paper presents some of the key information on the effect of various design and process parameters to obtain carbide free thin wall ductile iron castings. A solidification simulation technique was used to design the thin wall plates of ductile iron

casting. The effect of inoculants and moulding materials on structure-properties of thin walled ductile iron casting was studied.

Casting simulation for prediction and removal of shrinkage and optimization of gating system

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Solidification modeling, or computer simulation of the casting process, is being used by more and more foundries around the world to design the process for production of castings before castings are made or before equipment is built or altered. The primary objective of using casting modeling is to improve the quality of the casting produced; both in the initial castings made as well as over the production life of the part and also to minimize cost overheads. Casting simulation can be used effectively to reduce the shop floor trials and assure a defect free casting. Casting simulation approaches are based on either finite element method (FEM), finite difference method (FDM) or finite volume method (FVM). In this study, an attempt has been made to use FDM for casting solidification simulation and optimization of casting. The casting of impeller consisting of thin section blades connected to a thicker hub section usually is more prone to shrinkage defects during solidification, generating scrap and increased development lead times. Optimization is a technique, wherein a problem is structured such that the input variables and constraints are identified and an objective is specified. An optimum solution to the problem is then arrived using an optimization algorithm. The technique which we have settled on for performing optimization with casting solidification modeling is known as multi-variable response surface optimization, which basically considers the amount of change which results from modifying one or more design variables. The optimization of gating system is done for defect free layout with maximum yield.

Shrinkage and property prediction in ductile iron castings by solidification simulation

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Today there is an increasing trend in the industry towards alloys that provide increased strength over traditional alloys. In order to determine whether the casting process produces a part with proper as-cast mechanical properties, microstructure prediction is required. Additionally, casting geometry, molding process and cooling rates can be optimized to produce desired mechanical properties in as-cast parts, thus allowing the foundry engineer to produce castings that may not require a heat treatment operation. Casting solidification simulation assists in identifying hot spots, the potential locations of shrinkage porosity defects. It can be extended for optimizing the feeding system design considering quality and yield. Solidification simulation can be performed using Finite Element Method (FEM) or Vector Element Method (VEM). Simulation using FEM primarily depend upon Interfacial Heat Transfer Coefficient (IHTC) and is also influenced by the type of mesh, size of mesh, and the type of solver.A benchmark casting for sand casting process was designed to experimentally verify and validate the simulation results. The benchmark casting geometry was modelled in such a way that it satisfies general method guidelines and standard test specimens can be made from its solidified parts to evaluate the properties of the solidified cast alloy. The results of simulation are compared with experimental castings made in ductile iron. It was found that

simulation predicted the location and extent of shrinkage defect. The castings were further tested for mechanical properties such as tensile strength, ductility and hardness. The simulation analysis was also done to predict cooling curves in different locations in the castings. The experimental and simulated mechanical properties results were found to be a close match. These features make solidification based programs more suitable for implementation in the industry.

Heat transfer, fluid flow and solidification modelling of twin roll casting process

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Twin Roll Casting (TRC) is an energy efficient process designed to get thin strips of metal. The transport phenomenon is applied to TRC, in order to understand fluid flow, heat transfer and solidification behaviour of the metal. The governing equations of continuity, momentum and energy are solved with the help of ANSYS FLUENT software package. The focus of this study is to predict temperature of solidified strip at outlet of domain by changing various input/process parameters like inlet temperature, roll velocity, setback distance etc. Modification of the input parameters have an effect on changes in mushy zone length, outlet temperature etc. The phase change during solidification is handled by enthalpy-porosity technique and latent heat of fusion is taken care by specific heat capacity of metal.

Session 2:Nov 13, 2014:[13:30-15:30]

Phase selection in heusler alloys

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Heusler alloys are an important category of ferromagnetic shape memory alloys. Phase evolution and microstructure in these alloys play an important role in determining the magnetic properties of these alloys. In this talk, studies on alloys of Ni-Mn-Ga and Fe-Mn-Ga systems will be presented. Undercooling experiments were performed using electromagnetic levitation and flux undercooling. Rapid solidification experiments were performed using melt spinning. Microstructure and phase evolution are studied as a function of processing parameters. Microstructure characterization showed that Ni-base alloys could be made as phase-pure at high undercooling while it was not so in Fe-base alloys. A complete thermodynamic assessment of these systems is not currently available. We propose possible solidification paths in these alloys that could contribute to the determination of the metastable phase evolution in these systems. Magnetic property characterization studies will also be presented to demonstrate the correlation between the structure-property correlations in this system.

Effect of hot top geometries on solidification behavior of low alloy steel ingot

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Good quality of cast steel ingot is the key for achieving superior end product. As an attempt to improve ingot quality and yield, the effect of various hot top geometries on the solidification behavior of a typical 4MT ingot of AISI 4140 steel grade was studied through finite element based TherCAST simulation software. Various types of hot top geometries that included square, rectangle, round, wide end up square and wide end down square, with a typical volume of about 20% of body were studied. With the progress of solidification, the model predicted the phenomenon associated with fluid flow, temperature, solid front movement, mushy zone, thermal gradient ahead of the solidification front, local solidification time, porosity and piping. The wide end up and round hot tops were found to loose heat faster than other hot tops. The fluid flow patterns of molten metal were similar for all the hot tops. The wide end down and rectangular hot top showed deeper mushy zone and conical solidification front while other hot tops did not show distinct differences. Micro segregation measured in terms of local solidification time were found to decrease in the order; wide end down square > wide end up square > rectangular > square > round hot top. The columnar to equiaxed transition region was not affected by hot top geometry. Based on Niyama criteria which is a measure of liquid feeding to mushy zone in the final stages of solidification, it was observed that round hot top had least piping, low central porosity and highest yield for this grade, while the ingots with wide end down hot top gave highest porosity. Thus FEM based simulations helps in optimizing the hot tops used for a given mould and grade of steel chosen. A typical experimental validation carried out would be demonstrated.

Microstructure evolution during solidification of Ti-based multi-component in-situ ultrafine alloy composites

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Ti-based multi-component alloys find extensive applications in the aerospace and the defense sector. Traditional Ti-alloys (Ti-6Al-4V) have their limitation as far as maximum achievable strength with optimum ductility are concerned. The requirement of light weight Ti-alloys with high strength (>1.5 GPa) and optimum ductility (>10%) has triggered research activities on Ti-based multi-component in-situ composites with ultrafine microstructure via novel solidification routes. In the paper, we shall elucidate two such cases, Ti-Fe-Sn and Ti-Fe-Co alloys. It will be shown that the microstructures can be tailored by varying Sn and Co contents in the eutectic Ti-Fe alloy. The solidification of these alloys needs to be properly understood to develop the required microstructure. Therefore, we have adopted vacuum arc melting-cum-suction casting route to process rods of different diameter. In Ti-Fe-Sn alloys, the Sn content was varied in the suction cast hypereutectic Ti71Fe29-xSnx alloys with x = 0, 2, 2.5, 3, 3.85, 4.5, 6 and 10 atom%, followed by suction casting of these alloys, carried out in water-cooled split Cu molds under argon atmosphere. This study conclusively shows that these alloys undergo either of the two ternary reactions: (i) ternary quasi-peritectic reaction, L + Ti3Sn ⇌ β-Ti + FeTi and (ii) peritectic reaction, L + β-Ti + FeTi ⇌ Ti3Sn. The later reaction was predominantly observed for alloys x ≥ 4.5. In Ti-Fe-Co alloys, Co content was varied

by replacing Fe in Ti70Fe30-xCox, (x = 2.5, 5, 10, 15, 17, 20 atom%) and samples were suction cast. Microstructural observation indicated, that for alloys with x 2.5, the microstructure consisted of eutectic between FeTi and Co-rich bcc solid solution with primary (Co)-rich solid solution. For alloys with 2.5 x 15, a similar microstructure was observed. However, the eutectic content decreased as Co content in the alloy increased. In all these alloys, the first phase to form was -Ti, which underwent a peritectic reaction, L + -Ti (Co). For the alloywith x= 17, the formation of eutectic between FeTi and (Co) with (Co) dendrites was found. In case of alloys with 17 x 25, eutectic formation was not observed. Rather, formation of dendrites of (Co) solid solution and FeTi were observed.

Numerics and experimentation behind development of rheo-pressure die casting technology of al alloys for automotive, aerospace applications

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Specially prepared semi-solid slurry having spheroidal primary phase, uniformly distributed in the liquid matrix, is the key factor in semisolid metal (SSM) processing. Rheocasting is the focus of semi-solid metal forming in recent years. Several works focused towards development of slurry generation techniques for Rheo and Thixocasting such as stir casting, electromagnetic stirring, stress-induced and melt-activated (SIMA) process, Magneto Hydrodynamic Stirring (MHD) stirring, vibration, gas bubbling etc. But these techniques are yet to get commercial viability. Whereas, use of cooling slope technique for slurry generation is known for a while now in terms of its simplicity, ease of operation and possible industrial implementation. The present work discusses semi-solid slurry preparation of Al-7Si-0.3Mg (A356) and Al-Si-Cu-Fe (A380) alloy using cooling slope beside a High pressure die casting machine to provide slurry on demand. Cooling slope facilitates heterogeneous nucleation, partial crystallization of primary Al phase and shearing of the solidifying liquid alloy to produce nearly spherical, non-dendritic microstructure of the primary phase. Heat transfer, mass transfer and momentum of the solidifying melt governs the microstructure formation during cooling slope semi-solid slurry generation process and to investigate that, a 3D non-isothermal, multiphase volume averaging CFD model has been developed to simulate the semi-solid slurry generation process considering three different phases such as; parent melt (primary phase), evolving solid primary Al grains (secondary phase) and air (another secondary phase). Melt pouring at the top of cooling slope has been specified by velocity inlet boundary condition, and allowed to flow subsequently along the slope under gravity. Key findings obtained from the CFD model are volume fractions of three different phases considered, grain evolution, grain growth, size and distribution of solid grains, temperature field, velocity field, macro segregation, micro segregation etc. The results obtained from the simulations are validated by performing quantitative experimentation. To investigate microstructure formation mechanism during semi-solid slurry generation, phase field method has been employed. The results obtained from mesoscopic phase field simulations include grain size, degree of sphericity, grain density, grain orientations etc. Quantative image analysis of experimental micrographs confirms the accuracy of simulation results. Finally based on the numerical and experimental research findings, Rheo pressure die casting system has been developed at CSIR-CMERI and automobile steering knuckle has been successfully developed as a prototype component.

Multi-scale modelling of casting solidification process: Existing strategies and future directions

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Solidification of molten metal during casting processes involves many physical phenomena including changes in chemistry, fluid flow, phase transformation, heat transfer, microstructure evolution, and mechanical stresses. The length scales of these processes range from nano (1-100 nm: crystal structure, precipitates) to micro (50-500 μm: eutectic phases, dendrites), and further to meso (1-10 mm: grains, macro porosity). In principle, transport phenomena during solidification can be fully described by invoking pertinent equations of mass, momentum, energy, and species conservation at a microscopic level. However, a direct application of the same set of equations for solidification modeling over multiple length scales is computationally involved and challenging. This paper provides a detailed review of advances in multi-scale modelling of casting solidification processes, including mathematical models as reported in the literature. This is followed by a description of research work carried out on macro and micro scale modelling of casting solidification at CSIR-NIIST over the past decade. Finally, the existing coupling strategies for micro-macro scale simulation as well as future directions for effective coupling in terms of numerical modelling are presented. This is expected to be of interest to all those interested in computer modelling and simulation of metal casting processes.

Design of gating and risering system for pump casing investment casting

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Turbo-pumps used in liquid/cryo rocket engines require many investment cast components due to complex volute and vane profile. In present paper, pump casing cast component is discussed which houses two stage pump and is used in cryo engine. It is made in duplex stainless steel and is realized through investment casting route and poured by gravity filling method. During initial phases, the yield was very poor due to consistent defects at multiple locations in the most of the components poured at different foundries. In order to improve the yield a systematic study was mounted. The mould filling and solidification behaviour were studied for various gating systems used by different foundries using a FEM based simulation software ProCAST. Effect of geometry on the solidification pattern was analysed through these simulations. Based on the existing design rules available in literature combined with an understanding of the solidification pattern by simulation, an optimized gating system was proposed. Fins and casting taper were used to achieve desired solidification pattern and to improve casting yield. The flow analysis showed homogeneous mold filling with minimal turbulence. The particle trajectories showed low particle velocities in the casting cavity (less than 0.5 m/s). The optimized design was successfully implemented at the work centre for realization of castings.

Effect of chill and sleeve sizes on temperature gradients in sand casting for LM6 alloy

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Directional solidification is important for producing sound castings free from shrinkage porosity defect. Directional solidification is achieved by sequence of solidification of different regions of the casting which in turn depends on thermal gradients in the casting. To enhance the efficacy of feeder for directional solidification, feed-aids such as chills and sleeves are employed. Design of feeds aids is done based on modulus extension factor (MEF). Chills decrease the MEF of the part by its cooling effect. Traditionally, the chill size is taken based on experience and the choice of chill material is based on the heat diffusivity of the chill material. Similarly, the use of insulating and exothermic sleeves can increase the modulus of feeder, so that a smaller feeder is more effective resulting in higher yield. In this study, effects of chill size and material on thermal gradients are analyzed using computer simulation. These computer simulations are performed for a typical sand casting process for LM6 material. The nodal temperatures obtained are used to compute the thermal gradients inside the part. Different materials of chill include cast iron, aluminum and copper. The thermal gradients are computed based on FEM based casting simulation software. Strong thermal gradients shift the hot spot of the part towards the feeder; these changes in thermal gradients are tracked for their sensitivity to chill size and chill material. Sleeves are yet another means of achieving directional solidification. Sleeves keep the feeder hot for a longer time, this promotes thermal gradients towards the feeder. The influence of varying insulating sleeves thickness on thermal gradients is also presented.

An establishment of IHTC value during solidification of Al-Si alloy casting

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Solidification phenomenon governs the casting quality. Solidification phenomena are affected by various parameters, like Interfacial Heat Transfer Co-efficient (IHTC), cast metal, mold material, casting shape, casting volume, pouring temperature etc. In the present study, IHTC values were evaluated based on thermal histories throughout the mold and inside the casting and the results were compared using simulation. Time - Temperature data was recorded by five K-type thermocouples to evaluate the IHTC values. Three IHTC values were taken (300, 500 and 600 W/m2-K) for analysis using Finite Element methodology based software. IHTC values and curves at five different points (casting mould face and inside casting) are displayed and compared. Results showed that at all points, 500 W/m2-K give better solidification curves as compared to other IHTC values.

Effect of varying content of Sr addition on dendrite coherency of near eutectic Al-13% Si alloy

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Computer-aided cooling curve analysis was used to determine the dendrite coherency point (DCP). DCP is the point at which the growing dendrites in metal/alloys start to touch each other and form interconnected networks throughout the sample. It is a point at which the mass feeding of the liquid metal shifts to inter-dendrite feeding and the casting defects, such as segregation, hot-tearing and shrinkage porosity form after this point. DCP is of great interest in solidification simulation as it influences the ability of the metal in filling complex shapes and in the formation of inter-metallic compounds. The DCP is determined using two thermocouples, one at center (Tc) and another near to the wall (Tw), to record temperature differences with respect to the solidification time. The first minimum point in the temperature difference (ΔT= Tw-Tc) curve corresponds to the DCP and the corresponding temperature is the dendrite coherency temperature (DCT). In the present work, the influence of strontium modification of Al-Si on DCP was investigated. The fraction solid at DCP was determined using by fitting a baseline to the first derivative curve (Newtonian analysis). The results indicate that the DCT and fraction solid at DCP decreased with the addition of Sr. This implies that the dendrite growth is suppressed by the addition of Sr to the alloy.

Session 3:Nov 13, 2014:[15:30-17:30]

High speed casting of peritectic steel grades for enhancing caster productivity

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With increased tube grades (Peritectic Steel), caster productivity is a bottleneck at casters of SMS#1. Metal casting at higher speed can address productivity bottleneck of continuous caster. However, the casting speed is limited by unstable operating conditions (mold level variation, caster breakout, and superheat) as well as slab quality (oscillation marks, transverse/longitudinal cracks). The metal casting speed can only be increased in such situations by shrewd changes in operating conditions that avoid the specific defects concerned. Casting at higher speed is constrained by metallurgical length, primary cooling, secondary cooling and chemical composition of steel. The present work describes steps taken to increase the casting speed of peritectic steel grades (%C = 0.07- 0.09 and %Mn<0.6) produced in JSW to enhance shop productivity with changed product mix. To enhance the casting speed, mould cooling water was increased by 8%, secondary cooling specific water was increased from 0.68 to 0.72 l/Kg and standard casting speed chart changed w.r.t superheat of the steel. The increased cooling water resulted in heat extraction with the heat flux of normal casting speed. The spray pattern was changed accordingly with increase in casting speed. The speed chart was modified considering the superheat of steel. The slab exit temperature was monitored at center, drive and non-drive side and was found to be around 850-920 0C which is similar to the normal casting speed. The judicious changes in operating parameters at higher casting speeds resulted

in slab quality (surface & internal) at par with the normal casting speed slabs. It was possible to obtain an additional production of 18% in these peritectic steel grades keeping the same quality standard.

Influence of sen structures on surface flow velocity and vortex formation flow patterns in continuous slab casting mold

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The quality of continuous cast steel is greatly influenced by the fluid flow in the mold, particularly at the meniscus. Slag entrainment in continuous casting mold is one of the major causes of macro non-metallic inclusions in continuous casting steel products. It leads to quality problems like sliver formation, surface cracks on the surface of final rolled products. The structure of submerged entry nozzle (SEN) is regarded as crucial factor to determine the flow related phenomena in the mold. In order to understand liquid steel flow at the meniscus it was decided to look at the existing as well as new design of submerged entry nozzles using water model study.Design variables include the port angle, port height, while the effect of submergence depth is investigated. Extensive water model study was carried out with the existing and few new SENs. All experiments were carried out on a 0.4 scaled water model of the slab casting at Tata Steel R&D centre. Experiments were carried out for different casting speeds (1.2-1.8 m/min), submergence depths of SEN (120-240mm) and SEN with different port angles (0o, 15o and 25o). Time averaged fluid velocity distribution along the mold width direction just under the meniscus was measured by impeller-velocity probes. Camera was used to record the vortexing flow patterns, which were visualized by injecting the black sesames into water. Vortex phenomenon was characterized by two parameters such as vortexing frequency and vortex depth. Submergence depth had a strong effect, with deeper submergence depths leading to a reduction in meniscus turbulence and a reduction in the risk of entrainment. Insights gained by this modeling efforts aid greatly in the development of processing conditions to avoid the formation of defects in the final products.

Internal quality improvement of slabs cast from caster 3, RSP with soft reduction technology

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Rourkela steel plant is one of the five integrated steel plants under SAIL. Slab caster 3, with the most advanced technological controls has been added under the capacity enhancement project of the plant to produce 4.2MT annually. Dynamic soft reduction with use of HSA segments has helped in improving the internal quality of the slabs for plate/structural grades to a large extent and further improvement is in process. Due to the volume contraction associated with the solidification process of liquid steel, shrinkage cavity is produced at the crater of the final solidified region. Another aspect is the centerline segregation, which is present in slab castings due to solute redistribution during solidification and fluid flow induced by thermal convection. For these reasons, soft reduction technology is used to compensate solidification shrinkage of slab castings through the reduction of roll gaps in the region of centerline mushy zone. The online soft reduction minimizes the formation of internal voids in the slab caused by volume shrinkage and prevents interdendritic fluid flow of the liquid steel especially in the transverse direction. On the other hand, the compression produced by the soft reduction can also promote the liquid steel to flow in the

reverse direction of casting, which may lead to the redistribution of solute elements in the liquid steel. Therefore, the solidified structure of slab castings is usually more uniform and compact under soft reduction with reduced centerline segregation and porosity. The key technology of online soft reduction is used in tandem with the dynamic spray water control (or dynamic solidification control, DSC) model to accurately predict the location of the centerline mushy zone and to determine the position of soft reduction and a total soft reduction amount with the consideration of the different steel characteristics. The use of soft reduction technology has improved the internal quality of the slabs cast from caster 3 from Mannesmann standard 3 to standard 1 in respect to centerline segregation and transverse cracks.

Analysis of black line defect on the hot- rolled coil surface

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A good Cu-plate surface condition, proper mould powder operation and therefore a good meniscus without any effects in Cu plate like cracks, grooves, or “dent marks”, is very crucial for the formation of a good initial strand shell without any surface defects like longitudinal depressions /cracks on the cast product like thin slab produced from CSP caster. During the beginning of the current calendar year, there was a regular problem of the formation of lamination marks, termed as a Black line, seen on the hot rolled coil surface through the online parsytech inspection system. A strong co-relation was found between the location of the black lines and the dent mark found in the Cu plate mould after various data collection, online inspection and follow up of the coils during rolling and investigation of the Cu plate condition after finish casting. The present study analyzed the desired parameters like primary cooling in the mold, charge mix at EAF, mould powder improvement, water quality, standing wave measurement etc, which affect the copper plate surface damage and eventually led to minimization of black line defect.

Numerical simulation of solidification in continuous casting of steel blooms

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Continuous casting of steel essentially consists of pouring liquid steel in to a tundish, a ceramic container that acts as a buffer between the ladle and the mold. Water cooled copper mold is positioned below the tundish. Solidification begins at the mold walls while the cast steel is withdrawn from the mold. As the casting leaves the water cooled mold, it still contains liquid metal inside a thick solidified shell. The mold is subjected to oscillations in vertical direction in order to separate the solidified steel from the copper mold. In addition mold powder is also introduced into the mold to prevent adhesion of the solidifying steel with copper mold walls. Numerical simulation of continuous casting of steel blooms has been carried out with an objective to investigate the effect of process parameters on microstructure and metallurgical quality. Coupled fluid flow, heat transfer, phase change, and solid mechanics of the process have been investigated. Careful definition of boundary conditions and validation of the model for various grades of steel (low, medium and high carbon) and casting parameters resulted in the development of a reliable model. Evolution of shell thickness, solid fraction, surface and interior temperature profiles were analyzed.

Several boundary conditions were assigned such as heat boundary conditions for side surfaces, casting faces, bottom surfaces of the mold, temperature boundary condition for top of the casting. In addition, velocity boundary condition consistent with the pouring rate was assigned. The pressure boundary condition was assigned to ensure mass conservation. Although the results include both transient and steady state, the steady state results are relevant for continuous casting process. Steady state approach involving Eulerian method was implemented. Calculated shell growth and corresponding metallurgical length for a given casting speed were correlated.

Optimization of mould friction stress for different casting powder and steel grades in continuous casting

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The mould friction stress caused by interaction between the strand shell and mould copper plates is the most effective parameter in judging the situation of casting in mould equipped with hydraulic oscillator. Proper control of heat transfer and lubrication at this stage of the process plays a vital role in avoiding the formation of defects in the solidified skin. Mould heat transfer is usually determined by inserting thermocouples in the mould wall. In this study, the influence of mould friction stress is evaluated for 2000 heats for different casting powders and steel grades and correlated to surface defects in slab to standardized operating practice. The mould-strand friction forces depend on the properties of casting powder and therefore, in normal casting practice, casting powder is used depending on the steel grade to be casted. Different casting powders were evaluated with respect to surface defects in the product slab on the basis of friction stress and norms for threshold friction band (upper and lower friction stresses) were set. Effect of steel composition on the friction stress was analyzed on the basis of carbon content in steel and the threshold friction band was established. The finding was further extended to steel grades micro-alloyed with Ti and Nb. Increased surface defects were observed in micro-alloyed (Ti and Nb) steel, as higher fluctuations in friction stress were exhibited in casting theses grades. The mould slag properties may have modified in these micro-alloyed steel during interaction with the liquid steel. The heats cast outside the friction band resulted in higher surface defects in the product slabs. The surface quality under such circumstances were controlled through operating parameters viz., decreased casting speed, changing casting powder with higher viscosity and increasing primary cooling.

Optimization of EMS parameters to maximize equiaxed zone in continuously cast billets

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In several applications of casting, dendritic microstructure is not desirable as it results in poor mechanical properties. Enhancing the fluid flow in the mushy zone by stirring is one of the means to suppress this dendritic growth. The strong fluid flow detaches the dendrites from the solid-liquid interface and carries them into the mold to form slurry. This slurry has globular solid phase consisting of fragmented dendrites, instead of the conventional dendritic structure, immersed in liquid. When this slurry solidifies, the microstructure is characterized by globular, non-dendritic primary phase particles, separated and enclosed

by a near-eutectic lower-melting second phase. The globular microstructures, when partially remelted, offer less resistance to flow even at high solid fraction. This property represents a great potential for further processing in a semi-solid state by various forming techniques such as pressure die casting and forging.Electromagnetic stirring is a well-established technique for improving quality of continuously cast billets. EMS current and frequency are the major parameters which govern the stirring intensity in the liquid pool inside solidified steel shell. A proper stirring yields a refined cast structure with more preferred equiaxed grain structure over columnar one. It also helps in reducing segregation in cast billets. Stirring intensity through EMS depends upon casting speed and superheat. SMS-II shop at JSW Steel Ltd. is equipped with an 8-strand billet caster. A major problem associated with such big billet casters is differential superheat distribution at different strands through tundish urging the operator to maintain different casting speeds at individual strands. Hence, it becomes imperative to optimise the EMS parameters at different casting speeds so as to maximize the equiaxed zone in cast product.

A review of rhomboidity in billet caster at JSW, Salem works

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JSW Steel Limited, Salem Works (JSWSL), is an integrated steel plant, having a production capacity of 1.0 Mtpa (Million tons per annum) of high grade automotive special steels. The steel making route comprises of Blast furnace - Energy Optimising furnace - Ladle Refining furnace - Vacuum Degassing - Continuous Casting. At JSWSL, rhomboidity in billet caster is a major contributor to billet rejections. Rhomboidity defects increases the inspection and any missed out causes problem in mills due to twist. Rhomboidity was experienced mainly due to asymmetric cooling of solidifying strand in mould as well as in the upper zones of the secondary cooling. Rhomboidity rejection has been brought down by optimizing the mould life for different grades, proper secondary spray alignment, increasing the intensity and back pressure of the secondary cooling water at spray zones just below the mould and preventing clogging of nozzles by periodic cleaning of filters and nozzles. This paper discusses the various measures taken for effective controlling of rhomboidity defect in billets.

Hydrogen induced sticker breakout in slab casting: Mechanism and control

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Breakout in continuous casting of slab is associated with heavy production and equipment loss. The cause of breakout can be many but breakout which is caused by abnormal sticky behavior in the mould due to poor heat transfer is a phenomena which is prominent during the monsoon seasons. Abnormal sticky behavior in continuous casting of steel refers to an event when in an instrumented mould, temperatures of all four layers of thermocouple drop to a single temperature level (that of the lowest thermocouple layer in the mould) and thus doesn't help in identifying any phenomena happening in the mould. No signal is generated for any sticker or any other event and sometimes this leads to breakout in the mould. In this type of situation the operator has to manually slow down the casting speed to minimise the risk of sticker breakouts. The knocks on effect are casting delays and slab downgrades. The frequency of this kind of phenomena occurs more during monsoon seasons. This study was carried out by collecting top slag

samples and mould slag films from the mould for both normal and abnormal heats. Optical microscopy revealed presence of large size pores in the slag film of abnormal heats. The formation of porous structure in the slag film creates high thermal resistance to the heat flow from the slab to the mould, which retards the solidification of the slab. SEM analysis of the slag films in the abnormal heats showed presence of large numbers of bigger sized crystals well distributed throughout the film. These crystals in the slag film hindered heat transfer between solidifying shell and water cooled mould and led to abnormal sticky behavior. The reason of abnormal sticky behavior was further investigated through hydrogen measurement in tundish for both abnormal as well as normal heats. There is an increase of approx. 40-50 percent of hydrogen content in liquid steel where abnormal sticky behavior had happened compare to normal heats. Hydrogen causes faster crystallization of mould slag film which hinders the heat transfer to a great extent. The primary and secondary steelmaking parameters were looked into and it was found out that the choice of material added and addition pattern along with treatment time has major contribution towards this kind of phenomenon.

X-Ray diffraction analysis of mould fluxes used in continuous casting of steel

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Over 90% steel production in the world is carried out by continuous casting. Hence, it is of great concern to look at various aspects of it, one of which is the heat transfer through mould fluxes. Mould fluxes are basically powdered synthetic slags used to cover molten steel in a continuous casting mould. The fluxes used in continuous casting belong to the system CaO-SiO2 -Al2O3-Na2O-CaF2. The success of continuous casting depends on horizontal heat transfer through mould fluxes. Mould fluxes control horizontal heat transfer between steel shell and the mould; but improper conditions of casting cause a serious problem called "Longitudinal Cracking (LC)". Thus, it is important to control heat flux for better results. This decrease in heat flux is achieved by the crystallization of mould flux and its thickness. During crystallization, Cuspidine (3CaO - SiO2 - CaF2) is the primary phase formed in the flux film. To simulate conditions prevailing in actual steel plants, proprietary compositions of these fluxes were decarburized, rapidly melted and then subjected to crystallization at constant temperature for various time durations. To characterize crystallinity, X-ray diffraction (XRD) analysis of these samples was carried out. Results, thus obtained will be discussed in the light of percent crystallinity and its probable effect on heat flux.

Operability analysis of steel manufacturing processes- continuous casting slab process

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Continuous casting is one of the critical processes in the production of variety of steel products. The performance of the process is sensitive to the choice of operating condition and can be measured and monitored in terms of factors such as, productivity, yield and quality parameters like centre line

segregation, inclusions, etc. In order to better understand and improve the continuous casting process rigorous mathematical models were developed. These models are not amenable to optimization as they are computationally intensive. As an alternative, Response Surface Models (RSM) were developed from the observations of the rigorous model and used in locating robust operating conditions through compromised Decision Support Problem (cDSP) framework. In this study, we use RSM models to systematically explore and understand the process behaviour. To this end, we employ operability analysis to confirm the flexibility and sensitivity of the continuous casting process.In the input-output mapping of casting speed and mold level frequency (MLF) to productivity and oscillation mark depth (OMD), we observed input multiplicities. It meant that same OMD can be achieved at two different MLF values. The region where this phenomenon occurs is delineated. Further mapping study established that continuous casting process is sensitive and provides only a narrow feasible operating region. The bounds of the feasible were established by mapping the constraints on temperature limits on different segments and at unbending to input space in super heat and casting speed. As the approach is generic, similar analysis can be performed for other processes in the steel manufacturing.

Influence of density variation during sequence casting of different grades of steel

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Currently about 90% of the steel produced in the world is through continuous casting. Molten steel with desired temperature and composition is transferred from steel melting shop to the caster where liquid steel gets transformed to a solidified product. During liquid-solid conversion, typically a ladle is placed over a tundish and the latter feeds molten steel to one or many molds through submerged entry nozzle. During ladle change over operation, extensive mixing between different grades of liquid steel having different density and temperature can occur in the tundish. A differential density and temperature field in the molten metal has the potential to influence flow in tundish significantly, which in turn can influence the metallurgical performance of continuous casting system.This present paper discusses the behavior of grade intermixing time in steel making tundish systems by considering the combined effect of density (as in mixing of two dissimilar grades) as well as temperature variations (due to heat loss from ladle and tundish combined). This is important in steel making industry because grade intermixing is practiced frequently to meet customer demand. To understand the influence of composition and temperature, of incoming new grade of steel on mixing, on fluid flow, heat and mass transfer phenomena, a coupled turbulent fluid flow and heat and mass transfer model has been developed. Model equations were solved embodying the ANSYS™ CFD software platform Fluent. Predicted results indicate that with an increase in the density gap between two successive grades of steel, grade intermixing time tends to increase. Temperature differential to the tune observed in the industry, however, was found to have negligible influence on grade intermixing time. It is concluded that in order to minimize the grade intermixing time, change in concentration should be kept as minimum as possible.

Influence of superheat on porosity in continuous casting of microalloyed steel bloom

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The quality of continuous cast billets depends on the level of the as-cast bloom porosity and centerline loose structure. Casting parameters are usually optimized to improve the internal soundness in the as-cast bloom. In the present study, the influence of superheat on solidification behavior and centerline porosity formation in a large size continuous caster [cross section 320x400 mm2] was examined with a microalloyed steel grade, 38MnVS6. The solidification behavior is assessed using finite element model based software, TherCAST. The solidification simulations at two different superheat heat 350C and 500C were carried out. The simulation results predicted temperature distribution, solid front movement, thermal gradient, rate of solidification front movement, local solidification time and the center line porosity formation. The temperature distribution did not show significant difference with super heat. The solid front movement could be related to the extent of mushy zone, which increased with increase in superheat. The air gap between mould wall and solidified steel shell increased with increase in superheat. This is attributed to the resistance of the shell formed. At higher superheat shell thickness is less so the resistance is less. Increasing the superheat was found to increase the extent of centerline porosity as measured by Niyama criteria which measures the difficulty to feed the mushy zone bridged by the formation of dendrite from opposing faces. The solidification pool profile showed a deeper V-shaped profile at higher super heat condition which is conducive for porosity formation. The porosity development was experimentally validated in a typical trial bloom casting.

Investigation of causes of crack formation in CC billets and optimization of process parameters to minimize cracks

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In light of quality and productivity constraints, study of defects is likely to be a moving target. The present study is undertaken to examine the casting defects as part of an overall program to examine the customer complaints RINL received mostly from forging industries. An effort is made to account for defects in continuously cast steels as a function of steel making and continuous casting process parameters. In an as cast 250x250 mm bloom, few sub surface cracks and central porosity were observed. In some billets, the cracks open during subsequent forging operations. Analysis of operational, macrostructural and other related data is done to identify the source(s) of the problem. Scrutinizing and analysis of the operational data of refining at secondary steel making stage, continuous casting for the cracked billets as well as for necessary number of sound heats was done. Structural and compositional mapping of defective and sound billets samples were done. An attempt was made to correlate these properties to the soundness of bloom and billets.

COMPUTATIONAL MATERIALS SCIENCE

Invited talks

Session 1: Nov 13, 2014:[09:00-11:00]

Molecular dynamics simulation of thermal transport in carbon nanotubes

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Molecular dynamics (MD) simulation has become an established tool for understanding physical processes in various disciplines including chemistry, materials and biology at small scales of space and time, and to extract macroscopic properties of interest from these simulations. This talk will start with describing the dynamics and statistics of an isolatedsystem of many interacting particles. Since all physical processes take place in finite time and often in contact with a heat reservoir at constant temperature and/or under the influence of an external agent, MD simulation schemes for isolated systems are therefore modified to model such effects. The second part of this talk will focus on temperature control in MD with emphasis on a recently developed deterministic scheme that preserve microscopic reversibility. Finally, the simulation of systems under a thermal gradient will be taken up. The subject system in this talk will be single walled carbon nanotubes.

Atomistic modelling of materials using density functional theory

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Density functional theory is a widely accepted and used theoretical formalism to understand the physical and chemical properties of materials at the atomic scale from first principles. The basic formalism of density functional theory will be introduced. Our recent work in the area of nanomaterials using density functional theory based electronic structure calculations will be presented. I shall also present results on the structure and stability of nickel-aluminum superalloys and a ternary additive in the alloy. The utility and limitations of DFT calculations will be discussed.

Session 2: Nov 13, 2014:[13:30-15:30]

Computational thermodynamics of alloys using cluster variation method

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The accurate and easy computation of the free energy of a materials system plays an important role in determining the transformation pathway and thus the resulting microstructure. The cluster expansion method (for configurational enthalpy of mixing) and cluster variation method (for configurational entropy of mixing) (CE-CVM) together offer an accurate analytical procedure for such computations by suitably accounting for short range order unlike conventional CALPHAD methods. However, this increase in accuracy leads to an exponential increase in the computational effort for industrially relevant multicomponent systems. Owing to this as well as its algebraic complexity, the use of CE-CVM has been limited to binary and a few ternary systems. A procedure for simplifying the CE-CVM expressions to obtain analytical results for the behaviour of solutions at infinite dilution has been developed by us earlier. A procedure has also been developed earlier for increasing the accuracy of the method at the same level of approximation by treating the multiplicity of the clusters as a parameter. An elegant combination of these two procedures leads to an approximation of CE-CVM with CALPHAD like computational efficiency coupled with CE-CVM like accuracy.

Good practices in Gibbs energy modelling and optimisation

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It is widely recognised that knowledge of phase diagram and thermochemical data are very much essential to answer many practical questions that arise in the development, processing and application of engineering materials. The CALPHAD method offers a cost-effective and efficient alternative to conventional methods to obtain such information. However, its success very much depends on the reliability of the required Gibbs energy functions. This in turn depends on the underlying Gibbs energy models and optimisation strategies employed in the generation of these functions. This talk will concentrate on good practices in Gibbs energy modelling and optimisation of constitutional and thermochemical information that will help in obtaining reliable thermodynamic functions.

Session 3: Nov 13, 2014:[15:30-17:30]

Phase field modelling of microstructural evolution in systems with interfacial energy and mobility anisotropies

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Anisotropies play a key role in microstructural evolution. Phase field models are ideal for the study of microstructural evolution. Hence, incorporating anisotropies in phase field models is important and useful. In this presentation, I will present our results from a recent study on a class of phase field models based on the extension of the classical Cahn-Hilliard free energy to include higher order tensor terms. Such higher order tensor terms help us model interfacial energy anisotropies, with specific reference to cubic and hexagonal crystalline systems. The identification of the higher order tensor terms with the corresponding polynomials (either using invariant theory or by deducing it from first principles) has helped us incorporate anisotropies in atomic mobilities into phase field models of precipitate growth kinetics. I will also present

some of our preliminary results on the combined effects of these two anisotropies on the growth morphologies of precipitates during solid-solid transformations.

A crystal plasticity based multi-scale model for simulation of creep deformation behavior of nickel-based superalloys

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Nickel-based super alloys have been widely used in nuclear industry as steam generator tubes, through-wall nozzles and weld material etc. due to their superior strength and corrosion resistance properties. In these alloys, the shape and size of the phases control the mechanical and creep properties. It is very important to take into account of the underlying micro-structure in order to develop a reliable constitutive model for predicting the strength and creep deformations of these alloys. The aim of this work is to develop a physically motivated multi-scale approach for simulation of response of these types of alloys. At the lower scale, a dislocation density based crystal plasticity model is developed which simulates the response of various types of micro-structures within a single crystal. A new model for simulation of the mechanism of anti-phase boundary shearing of the precipitates with matrix dislocations is also developed in this work. The lower scale model is homogenized as a function of various micro-structural parameters and the homogenized model along with a new micro-twin initiation criterion is used at the next level (i.e., grain level) to simulate the polycrystalline response as well as the tension-compression asymmetry as observed in single crystal experiments of these nickel based super alloys.

Contributory papers

Session 1: Nov 13, 2014:[09:00 - 11:00]

Formation of long-period superstructures (LPS) in Al-rich TiAl alloys: experimental results and ab-initio simulations

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Al rich titanium aluminides offer an attractive combination of low density and good oxidation and ignition resistance with unique mechanical properties for aerospace and high-temperature structural applications whenever specific strength and stiffness are of major concern. Due to extensive non-stoichiometry of upto 72% at higher temperatures in L10 -structured TiAl, several LPS have been observed to form which adversely affect the mechanical properties of these alloys. TEM results indicated formation of ordered long-period superstructures (LPSs): h-Al2Ti, Al5Ti3 along with Al5Ti3', Al11Ti7 and Al3Ti2 LPSs, the latter are observed only as short-range ordered clusters at nanoscale level in Al-rich TiAl based alloys. Al5Ti3-type anti-phase domains with various sizes in diameter were also obtained in Ti-62.5at.%Al crystal by heat treatment at 600 C and 750 C after the floating zone melting and subsequent quenching from 1200 C. Further, the anti-phase boundary (APB) of type-A appeared with a quite high frequency than type-C and all these APBs laid on {110} plane. The space filling tiling arrangement and a symmetry analysis program were used to determine the unit cell and the crystallographic information of well-known h-Al2Ti, Al5Ti3 and

unknown Al5Ti3', Al11Ti7 and Al3Ti2 LPSs in terms of L10 fcc unit cell. First principles calculations have been performed to determine phase stability and mechanical stability of these LPS. Moreover, the family of five LPSs are having subgroup-supergroup relationships with γ-TiAl (Sp. gr. P4/mmm) and among themselves. The calculated formation energies showed that r-Al2Ti has the highest structural stability followed by h-Al2Ti, Al5Ti3, Al3Ti2, Al11Ti7, Al5Ti3' and γ-TiAl in that order. Further, we have also calculated antiphase boundary (APB) formation energies for two types of APBs, viz., type-A and type-C in ordered Al5Ti3 LPS using the supercell approach validating the experimental observations.

Role of clusters in bulk metallic glass formation

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Bulk metallic glasses (BMGs) are often described as configurationally frozen supercooled liquids. Similar to atomic arrangements in liquid structures, there exists certain short and medium range clusters in BMGs. These clusters play decisive role in glass formation and also on the nature of their crystallization. In the present investigation, an attempt has been made to understand dominant clusters that control glass formation in multicomponent Zr-Cu-Al-Ni alloy. Ascalaph Designer Molecular Modeling Suite is used to generate different models of clusters and the corresponding molecular potential energy is evaluated. Thermodynamic calculations have been carried out to validate modeling results. Such studies provide insights to decipher the role of clusters in glass forming liquids.

First-principles calculations of carbides in steels

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Metal carbides impart strength as well as toughness to steels. The mechanical properties of many special purpose steels, such as tool steels and secondary hardening ultra high strength steels are strongly influenced by the type and chemistry of carbides, their volume fraction, size and spatial distribution, as well as their morphology. Specific heat treatment schedules (such as hardening and ageing/tempering at different temperatures) are adopted to tailor the microstructure in order to achieve required properties for the intended application. Knowledge of the structural and elastic properties of the carbides forms the protodata in multiscale modeling and simulation. It is also of interest to know the nature and the strength of the bonding of the carbides to the steel (predominantly iron) matrix.In this work, first-principles based density functional theory calculations have been performed using Abinit, an open source software. We will present our results on the structural and mechanical properties and electronic structure of transition metal carbides (MC and M2C) that are commonly encountered in steels. For example, the calculated bulk modulus values of the monocarbides are in the range of 215 to 330 GPa. The carbides can be arranged in increasing order of the bulk modulus value as ZrC, HfC, TiC, VC, NbC, CrC, TaC and MoC. In the M2C carbides with Cr and Mo as metallic constituents, the effect changing the Cr/Mo ratio on the equilibrium lattice constants and the electronic structure has been investigated. Finally, results from preliminary efforts on the electronic structure and energetic of model matrix-carbide interfaces will also be presented.

DFT Study on Lauryl amine and N-dodecylethylene-diamine in Gaussian 09 along with their interactions with Fe atoms/ions

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Molecular modeling of N-dodecyl-amine (also known as Lauryl amine) and N-dodecylethylene-diamine (ND) are done using Gauss View (GUI), the structures were geometrically optimized on Gaussian09 by using DFT as calculation method with B3LYP as function and 6-31++G as basis set. MOs of the molecules are visualised & thereafter total energies & MO energies of the collectors are calculated from the mentioned DFT analysis. Fe atom, Fe^{2+} and Fe^{3+} ions are introduced with both of the collectors and their interaction with the atom/ions were investigated by using DFT calculations. Finally, the numerical results for the interaction energies of N-dodecyl-amine (lauryl amine) as well as N-dodecylethylene-diamine (ND) with Fe atom, Fe^{2+} and Fe^{3+} ions are presented. The optimised geometrical structures of lauryl amine and N-dodecylethylene-diamine can be used further for studying their behavior as cationic collectors with haematite in reverse froth floatation process.

A computational approach for designing new alloys

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Alloys have played an important role in the field of science and technology, as they offer much needed tunability to the properties of the parent materials. Alloys of newly discovered two dimensional (2D) materials, like graphene, boron nitride, metal dichalcogenides etc., are of great interest. This is because, although the numbers of parent materials are not too many, there lies a huge opportunity to design new 2D materials via alloying route. In this work, a study on alloy designing via a computational approach, namely the cluster expansion, will be presented. Using this method, alloying among different 2D materials have been studied. Utility of 2D alloy materials, like tunable electronic properties for device applications, will also be discussed.

Computational design of Ti alloys for improved performance in prosthetic applications

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Design of new β titanium alloys suitable for orthopedic and dental implants, with lower modulus of elasticity and greater strength with adequate biocompatibility and low cost, is done using several

computational intelligence based methods in tandem. Ti-6Al-4V, the most used bio-metal for this purpose nowadays, have the risk of toxicity of vanadium and higher elastic modulus compared to β Ti alloys. Low elastic modulus, close to that of bone to avoid stress shielding, along with adequate strength may be achieved through suitable modification of the composition and processing schedule. To achieve the above conflicting objective in the mechanical properties, a multi-objective evolutionary algorithm is performed to find the optimum combination of the composition and processing parameters and to get optimum solution, i.e. low elastic modulus and high yield strength. Artificial neural network (ANN) and Fuzzy Inference System (FIS), which are effective tools for developing data driven models for representing highly complex and nonlinear correlations in a materials system, are used as the objective functions, in absence of any physical model describing the relations between the above mechanical properties with the compositional and process parameters. In case of FIS modeling microstructural features are incorporated using prior imprecise knowledge of the system. Cost of the alloy is used as the third objective, where a weighted summation model of the elemental constituent is developed with the recent cost of the alloying additions. The biocompatibility issue is taken into account as a constraint in the optimization process. Two separate evolutionary concepts, viz. Non-dominated Sorting Genetic Algorithm II (NSGA II) and Reduced Space Searching Algorithm (RSSA) are employed for the multi-objective optimization problem. Pareto optimal solutions provide several compositions of β Ti alloys, which are analyzed further to gather additional information about the complex system.

Molecular dynamics simulation of mechanical and thermal properties of Zr and Ti based alloys

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Mechanical and thermal properties of materials are of pivotal importance in their selection for any technological application. Mechanical properties, e.g., elastic constants and stress-strain relations give a deep perception of mechanical strength of the materials; whereas thermal properties, e.g., thermal expansivity gives an intuition of thermal shock resistance and thermal stress generation at higher temperatures. Classical molecular dynamics (MD) simulations of mechanical properties and thermal properties have been carried out for Zr and Ti based alloys, viz., Zr-Al and Ti-Al systems. We have determined the elastic constants of Zr-Al and Ti-Al alloys as a function of both composition and temperature and their thermal expansion coefficients as a function of composition in a given temperature range.We have used classical MD code LAMMPS for all our simulations. Periodic boundary conditions are used to avoid the surface effects. Interatomic Potentials (force-fields), viz., Zope and Mishin eam potential for Ti-Al system and Finnis-Sinclair potential for Zr-Al system have been thoroughly tested before proceeding for the main calculations. For the calculation of elastic constants, the unit cells were appropriately deformed and the stresses corresponding to the deformation have been calculated. To calculate the elastic constants at above 0K, NPT ensemble has been employed. Thermal expansion coefficients have been calculated from the slope of lattice parameters, L, and temperature, T plots in the NPT ensemble in the temperature range between 300K to 700K. We have computed the stress-strain relations at various temperatures for some ordered intermetallics, viz., Zr_3Al, Zr2Al, TiAl, $TiAl_3$ as well as for disordered solid solutions at various composition. In this presentation, we will present our MD simulation results and compare with those obtained experimentally.

Session 2: Nov 13, 2014:[13:30 - 15:30]

MD simulation of tensile properties of copper nanoparticles

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Molecular dynamics (MD) simulations have been carried out to evaluate the stress-strain behavior of copper nanoparticles under tensile loading of ultra-high strain rates. The engineering stress-strain curves have shown initial liner elastic part, followed by plastic region involving local necking deformation. However, the engineering stress-strain curve does not show any uniform plastic deformation region under the ultra-high strain rates considered in the present simulations. Moreover, the tensile deformation has evidenced significant temperature rise of the nanoparticles, which is a natural consequence of any plastic deformation process. The yield strength, Young's modulus and % elongation of copper nanoparticles have been evaluated based on the engineering stress-strain curves generated by a series of MD simulations and correlated with strain rate, initial temperature, particle size, etc. The estimated values of yield strength and Young's modulus extrapolated for the strain rate used in conventional tensile testing have shown several orders of magnitude enhancement of these properties compared to the bulk copper. It was also found that with decrease in particle size the yield strength of copper nanoparticles increases. This indicates the dominant role of surface effect of nanoparticles in enhancing their strength properties. Moreover, addition of silver to copper nanoparticles causes further improvement in the strength properties of the nanoparticles because of the alloying effect. The present study demonstrates an easy way for the estimation of mechanical properties of nanoparticles under tensile loading conditions using MD simulations.

Use of solver in computational thermodynamics

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According to the 2nd law of thermodynamics, the criterion of equilibrium is given, in terms of Gibbs free energy (G), by $\Delta G = 0$ or $dG'_{P,T} = 0$. The first condition is used in the classical method of solving the equilibrium state of a system. The second condition, according to which the (Gibbs) free energy of a system is minimum at equilibrium at a given temperature and pressure, is the basis of all forms of computational thermodynamics. Solver, a numerical optimization tool offered by Microsoft, can be adapted to find the minimum value of the Gibbs free energy of a system, subject to the constraint of elemental balances, in an Excel spreadsheet, giving the equilibrium state of the system. In the current work, Solver has been used for a variety of equilibrium calculations. The stability diagram of the Fe-C-O system involving FeO, Fe, C, and Fe3C (all solids) and the gas phase containing CO and CO2 has been constructed. The diagram shows the coexistence of 4 solids and 1 gas phase at a particular temperature (1037 K) and pressure (10.04 atm), consistent with the zero degree of freedom allowed by the Gibbs phase rule. This work will also explain why cementite (Fe3C), often considered as a metastable phase, is not so above 1037 K. In addition, binary phase diagrams of different types such as isomorphous, eutectic with limited solid solubility, eutectic with intermediate line compounds, have been drawn. The Al-Ge binary

system has been reassessed with the help of two solution parameters (ω L and ω S) and new data on the partial excess entropies and enthalpies of mixing in the two terminal solid solutions are generated. Also, finding the equilibrium products of the carbothermic reduction of different oxides like Fe2O3, SiO2, and TiO2 at different temperatures is in progress.

Thermodynamic assessment of Mo-Nb-Ti system

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Equilibrium phase relationships in the Mo-Nb-Ti system are computed using the CALPHAD approach. SGTE recommended lattice stability parameters of pure elements are employed. To enable the thermodynamic description of binary systems, the results from a previous evaluation are adopted for the Mo-Nb, Mo-Ti and Nb-Ti systems. A thermodynamic assessment of the ternary system is performed based on the experimental data. Phase diagrams of limiting binaries, liquidus projection and two isothermal sections are computed.

A thermodynamic measurement and assessment of Nb-Ti system

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The cluster expansion and cluster variation methods (CE-CVM) are used for representing configurational enthalpy of mixing and configurational entropy of mixing for mateials systems respectively. CE-CVMaccounts for the short range ordering accurately, which is often neglected in the most widely used CALPHAD method. CE-CVM acts as very powerful technique to compute phase diagrams using phase equilibria data, thermo-chemical, thermo-physical and structural data. For Nb-Ti system only phase equilibria data are available in literature. It is also desirableto have thermodynamic and structural data for simultaneous optimization, in order to better represent the thermodynamic behaviour of the system. Hence, the thermodynamics of Nb-Ti binary alloys have been studied using differential scanning calorimetry (DSC). The data thus obtained have been utilized for simultaneous optimization using CE-CVM to identify the model parameters called effective cluster interactions, using which, the phase diagram of the system could be computed. The cph phase present at low temperatures in the system is modeled using the tetrahedron-octahedron approximation, while the bcc phase at higher temperatures is modeled using the irregular tetrahedron approximation of CE-CVM. A sub-regular solution model has been used for the liquid phase. The observed data are found to in good agreement with the calculated values.

A comparison of Gibbs energy models representing SRO in binary fcc systems

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The present investigation attempts a critical comparison of models for the Gibbs energy of binary fcc alloys which account for the presence of short range order (SRO). The frequently used models for this purpose are the cluster expansion-cluster variation methods (CE-CVM), the cluster-site approximation (CSA) and Monte-Carlo simulations (MCS). Among these, CE-CVM offers an explicit and theoretically sound representation of the Gibbs energy. On the other hand, MCS is credited to give the most accurate results, which are used as standard for comparison of various approximations of the models.The frequently used tetrahedron-octahedron (TO) approximation of CE-CVM includes pair interactions up to second neighbours and consists of 9 correlation functions as microscopic state variables for a binary fcc system. The quadruple tetrahedron (QT) approximation consists of 82 correlation functions, the results of which closely compare with those of MCS. We have recently developed a modified cluster variation method (M-CVM) based on TO approximation for improved accuracy without increasing the computational burden. The results of all these models are compared for the case of first neighbor pair interactions for selection of the most appropriate model for the Gibbs energy of fcc systems.

Development of thermodynamic database for Ti-Al-Mo-V-Cr system

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Ti alloys are used in aerospace applications because of their high specific strength. Microstructure evolution and thermodynamic modeling of multi-component Ti alloy system require a consistent thermodynamic database to be developed for the multicomponent system. Thermodynamic database contains parameters for Gibbs free energy equations for different phases as a function of temperature and compositions. A validated thermodynamic database can be used to extract all sorts of thermodynamic and phase diagram data for a multicomponent system. Commercially available thermodynamic databases (PanTi, TTTi etc.) however are valid only for a few alpha+beta alloys, e.g., Ti-64, Ti6242 etc. However, there are many near beta Ti alloys (Ti-10-2-3, Ti-5553 etc.) and other alpha+beta Ti alloys like Ti-550 which are of considerable interest for applications in aerospace industries, but available thermodynamic databases are not valid in the composition ranges of these alloys. It is therefore important to develop thermodynamic database that would be valid for the extended composition range. The present effort is towards developing such database for Ti-Al-Mo-V-Cr system, which would be valid for extended composition ranges. To develop this quinary database, firstly consistent descriptions of phases for different binary, ternary and quarternary systems have been made in terms of sublattice models and then using CALPHAD methodology a consistent database has been developed. Using the database, beta transus temperature, phase fractions, phase compositions, partition coefficients, phase boundaries have been predicted for various important multi-component Ti alloys. In this paper, development of the database and predicting thermodynamic properties using the database in PandaT software would be described. Validation efforts would also be presented.

Artificial neural network modelling for Indian coal characterization

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For characterization of coal, proximate and ultimate analyses are performed to obtain chemical composition and coal rank based on gross calorific value (GCV). Effective combustion of coal and minimum generation of flue gases are the primary factors for determining energy efficiency of coal fired power plants. Therefore determination of coal quality, proper blending of various ranks of coal and firing sequence are of great importance. It is often required to obtain the percentage of C,H, N, S and O obtained by ultimate analysis from the results of proximate analysis i.e. percentage moisture, ash, fixed carbon and volatile matters since the latter is less expensive. However, ultimate analyses give better appreciation of coal quality in terms of energy efficiency. In order to predict results of ultimate analysis from the proximate analysis data, artificial neural network modeling has been carried out. A back propagation feed forward neural network has been used with a single hidden layer. For training the network 180 data samples have been used and for simulation and prediction 80 data points are considered. For the training samples, the correlation coefficient is found to be more than 0.9 whereas the same was less than 0.8 during prediction. The predictive modeling was made based on the multivatiate regression analysis (MRA) reported by Bureau of Energy Efficiency, India regarding the inter-relationships of C, H, N and O with percentage moisture, ash, fixed carbon and volatile matters. In this case, the reason behind weak correlations observed between predicted and actual results is due to the fact that that the correlations are applicable for coal with more than 15% moisture content only. However, MRA has been carried out to obtain a new set of equations for the present variety of coal which are presented here with various correlations obtained by ANN modeling.

Session 3: Nov 13, 2014:[15:30 - 17:30]

Multi-scale modeling of effect of substrate bias on crystallinity of NixTi1-x thin films deposited using magnetron sputtering technique

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Crystallinity of NixTi1-x thin films deposited on a substrate using magnetron sputtering technique is strongly influenced by substrate bias voltage. The substrate bias induces higher flux and momentum of Ar+ ions striking deposited NixTi1-x thin film. Using Computational fluid dynamics approach on COMSOL platform, Ar+ flux in the entire sputtering chamber and on the deposited film was computed. Based on the Ar+ flux, Monte Carlo approaches at the atomic scale computed: (a) The flux of Ni/Ti adatoms reaching the thin film as well as (b) The vacancy generated due to re-sputtering. Finally Classical Molecular Dynamics based model predicted the stability of crystalline and amorphous phases based on the fraction of vacancies predicted using Monte Carlo approach. Crystallinity predictions were experimental validated using high resolution transmission electron microscopy characterization of NixTi1-x thin film deposited under different substrate biases.

In silico designing of novel age-hardenable Al alloys with improved strength and ductility

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Several computational intelligence based methods are used in order to design age-hardenable aluminium alloys with improved mechanical properties. Improving the strength and ductility simultaneously is a multi-objective optimization problem, as conflicting objectives exist. In such cases a set of solutions, known as the Pareto set, provides the best possible compromises between the objectives. The Pareto set thus offers a number of equivalent optimum alloy compositions, out of which a decision maker can easily pick and choose the most suitable ones. To implement this approach multi-objective genetic algorithm (MOGA) is used in this study. To develop objective functions for the optimization, several data driven modeling techniques, from the domain of computational intelligence, are used. The modeling methods used are artificial neural network (ANN), rough-fuzzy hybrid system and genetic programming (GP). The database generated for developing the models are taken from all three common age-hardenable Al alloy series, and thus models go beyond the limit of any particular alloy series. As a result the design solutions thus generated are also not constrained within the limit of any series. The database, used for the above models, contains the composition and processing parameters as the inputs and the mechanical properties, viz. yield strength, tensile strength and ductility, as the outputs. The computational exercise revealed that to achieve the targeted performance, the requirement of alloying additions vary significantly with variation in the testing temperature. Thus the data is divided based on the testing temperature as sub-zero, room and high testing temperature regime and then modeling is done. The result obtained from different modeling methods applied on the data brings out the important compositional variables and discards the redundant ones. Prior knowledge and the Pareto solutions obtained from the optimization studies are used for final decision making, and designing of alloys.

Microstructure evolution by spinodal decomposition &subsequent percolation analysis of formed clusters

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Our work involves studying the phase separation by spinodal decomposition by solving the Cahn-Hilliard Equation numerically. We implement explicit, implicit, and semi-implicit methods on different Models and compare these methods on the basis of Accuracy, Stability and Efficiency. The decomposition is triggered by introducing noise in the homogeneous system as the initial condition. We employ Periodic Boundary Conditions throughout.We begin with the Double Wells Potential model which is found extensively in the literature. We solve for a one dimensional binary system and then move on to the two dimensional binary system. The microstructure evolution shows cluster formation.The Cahn-Hilliard Equation is solved again for Regular Solution Model which gives surprisingly different results as compared to the Double Wells Model. Here also the microstructure evolution is studied.Once the clusters are formed after spinodal decomposition, we use the Hoshen Kopelman Algorithm to label the clusters and find their size. The actual ingenuous of Hoshen Kopelman algorithm is realised when we try to understand another very important phenomenon known as percolation. When a cluster extends across the area under observation in any direction, then it is said to be percolating in that direction. HK Algorithm is extended further to check the clusters for percolation and find the number of percolating clusters in each direction. We find the critical percolation concentration by plotting probability of percolation against concentration. The critical

percolation concentration is defined as the concentration after which percolation always occurs. The work can further be extended by replacing regular solution model with cluster variation method.

Residual stresses in laser welded 9Cr-1Mo(V, Nb) ferritic/martensitic steel: A computational study

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Ferritic/martensitic steel with nominal composition Fe-9Cr-1Mo-0.22V-0.07Nb-0.1C is a high temperature material of interest to ultra-supercritical power plants and advanced nuclear reactors. Laser welding of this material has recently attracted considerable interest. Experimental work on the laser welding of these materials need to be supported by computational work. Thermo-metallurgical and thermo-mechanical computations have been carried out by simulating laser welding of 9 mm thick plate of this steel at 8 kW laser power and 1.5 m/min welding speed on a 3D mesh model using a finite element based software SYSWELD. A combination of 3D Gaussian and double ellipsoid heat source and temperature dependent physical and mechanical properties of this material have been used in these computations. Solid state phase transformation of tempered martensite to austenite and from austenite to martensite has been incorporated in these computations. von-Mises yielding criteria and isotropic hardening model have been used. The results showed very short residence time of the material in the fusion zone (<0.3 s) and the heat affected zone (< 0.5 s). Residual stress profile showed a trough in the fusion zone and a peak on the either side of it in the parent metal bordering the heat affected zone. Temporal evolution of the stress fields clearly showed that solid state transformation of austenite has a very significant compressive effect on the residual stress. These results have been presented and discussed in the context of low heat input associated with laser welding process, high aspect ratio of the fusion zone and transformation of austenite into martensite.

A FEM model for predicting orientation dependent strain stress behavior of Ti-6Al-4V

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Titanium and its alloys are important class of material for aerospace, biomedical and chemical industries because of its unique combination of high specific strength and high corrosion resistance properties. Out of total Ti alloy market Ti_6Al_4V has more than 60% share. Texture and orientation of the grains play very important role for deciding the properties of material. In present work nanoindentation experiments are carried out to obtain load-displacement behavior for individual orientations of Ti_6Al_4V. A FEM model of nanoindentation is developed to replicate the experimental results. The constitutive equation obtained from nanoindentation is then used to predict the behavior of the other orientations of Ti_6Al_4V alloy. This model is useful for predicting the plastic behavior of single crystal Ti_6Al_4V alloy for various applications and it can be further extended to polycrystalline alloy with incorporating the interaction among the orientations.

Finite element modelling and analysis of brake squeal

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It is well-known fact that automobile brakes generate several kinds of noises like squeal, groan, chatter, judder, moan, hum and squeak. Squeal is the most prevalent, annoying and can be reduced by variations in geometry, parameters such as coefficient of friction, stiffness of material. The brake squeal generally occurs in the range of 1-16 kHz.Basically two methods are available to study the disc brake squeal, namely complex eigenvalue analysis and dynamic transient analysis. Complex eigenvalue analysis is the standard method used for squeal analysis. Analytically it is very difficult to solve because of complex brake mechanisms. Experimental and numerical techniques have been developed by various researchers in order to study brake squeal. Experimental techniques are unable to predict brake squeal at the early stages of design process and also very costly due to associated design iterations. Therefore, finite element analysis has emerged as a viable approach for brake squeal analysis.This work presents Finite Element modelling and modal analysis of disc-pad assembly using high end software tools. Linear non-prestressed modal analysis and full nonlinear perturbed modal analysis is applied to predict frequency at which squeal occurs. Real and imaginary eigenfrequencies of unstable modes are obtained. Analysis is performed by varying the coefficient of friction and outer diameter of disc-pad assembly. Increasing friction coefficient has no desirable effect on squeal frequency while squeal propensity decreases as the outer diameter of disc is increased.

Prediction of thermal conductivity of the Copper-cubic Boron Nitride composites by Finite Elemental Method

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Heat is generally generated whenever an electronic device is in operation. If the heat is not allowed to dissipate, the temperature of the device increases which may result in the breakdown of the device. Hence it has become necessary to address the issues related to dissipation of heat from these devices. Researchers are trying to find new materials to be used as a heat sink to absorb the heat from the source & dissipate it to the environment more effectively than the existing conventional materials. An attempt was made to study some of the thermal properties of copper – cubic boron nitride composite which can be a potential material for sinking the heat efficiently.In this work the micrograph of a Spark Plasma Sintered copper-cubic boron nitride composite sample, was obtained. This micrograph was fed into OOF2, a finite elemental microstructure modelling software and the thermal properties of the material was estimated through finite elemental analysis considering practical boundary conditions. The temperature distribution as well as the thermal conductivity of the composite was determined. Variation of thermal conductivity with temperature was also determined.

Morphologies of growing precipitates during solid-solid phase transformations: a phase-field study

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During solid-solid transformations, the morphology of a precipitate growing from a supersaturated matrix (among other things) is (also) influenced by the anisotropies in the interfacial energy and atomic mobilities. We have formulated a phase field model in which both these anisotropies (the interfacial and the mobility) can be incorporated. Using a numerical implementation of such a formulation (based on semi-implicit Fourier spectral technique), we have studied the growth morphologies of precipitates in systems with cubic anisotropy in atomic mobility and interfacial energy. These anisotropies lead to interesting microstructural features. While interfacial energy anisotropy typically leads to facetted morphologies, kinetic anisotropies lead to dendrite-like branching. Our studies on the changes in aspect-ratios of the branched precipitates as a function of supersaturation and time indicate that the purely kinetically driven morphological features, though more prominent in the early stages, become less dominant when the driving force for growth (namely, supersaturation) is exhausted. Finally, we will also show some preliminary results on the combined effect of the anisotropies in interfacial energy and atomic mobility on the morphology of growing precipitates. We believe that these results might be of relevance in understanding the formation of solid-state dendrites in Fe-Si-B systems and the formation of metallic precipitates in oxides.

CORROSION AND ITS PREVENTION

Invited talks

Session 1: Nov 14, 2014: [08:30 - 10:30]

Biofouling and corrosion tendency of titanium in sea water

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Titanium is extensively used as condenser tube and heat exchanger plate materials in nuclear power plants, owing to excellent mechanical and corrosion resistance properties in sea water. It is however amenable to severe biofouling in sea water and biofilms formed on titanium surfaces impede its heat and mass transfer properties. Investigations were carried out to study biomineralization of manganese on titanium exposed to sea water. Viable counts of marine organisms including manganese oxidizers and sulfate reducing bacteria were made on biofilms formed on sea water-exposed titanium specimens. Formation of biogenic manganese oxides on titanium surfaces interfered with heat and mass transfer efficiencies as tested on heat exchanger and condenser tube materials. The role of manganese oxidizing bacteria and Sulfate Reducing Bacteria (SRB) on the electrochemical behavior of titanium was studied to understand the role of biofouling on its corrosion behavior. Open circuit potential measurements and potentiodynamic studies in presence of the above marine isolates provided information on potential ennoblement and passive film impairment due to bacterial activity. The role anaerobes and aerobes in modulating the passive behavior of titanium is discussed.

Nano-structuring NiTi alloys by selective dissolution and on the novel tool for concurrent electrochemical-fatigue studies of NiTi wires

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NiTi is a special class of alloys, exhibiting unique properties like shape memory effect and pseudo-elasticity, hence used in variety of applications. The use as biomaterial demands, good mechanical stability (i.e. fatigue resistance, large reversible strain, and high tensile strength) and biocompatibility (i.e. non-toxicity and corrosion resistance in a body environment). The fatigue behaviour has been a topic receiving widespread interest. Similarly, the corrosion behaviour of NiTi and NiTi-based alloys has been in the focus of researchers. Even though, these two fronts have been addressed independently, there is only very few work relating the two fields. Studies are being carried out to tailor the mechanical, surface, and electrochemical aspects of these alloys by adding ternary elements(X= Nb, Cu, and V etc.). Also designing nano-structures in alloys (NiAl-X) using directional solidification and subsequent electrochemical

processing has been reported in literature.This presentation would address two topics. The first topic would discuss on the engineering of nano-structures in NiTi-V ternary alloy by metallurgical treatment followed by electrochemical processing. The directional solidification of the NiTi-V pre-alloy rendered self assembled nano-structures (rods/lamella) of minor phase V in NiTi-V matrix. The influence of processing parameters on the structure, the selective chemical and electrochemical processing to obtain nanostructures would be elucidated. Secondly, the design and fabrication of an in-situ bending rotation fatigue machine and its potential use for concurrent studies on the electrochemical and fatigue behavior of NiTi shape memory alloy (SMA) wires would be discussed

Session 2: Nov 15, 2014: [11:00 - 13:00]

Hydrogen and cyclic load interactions in steels

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Hydrogen interacts with cyclic loads to bring about accelerated degradations of materials. The work done on hydrogen interaction with cyclic stresses, the influence of the latter on hydrogen diffusivity, the effect of different cyclic loading parameters on hydrogen embrittlement, is discussed. The special case of overloads during cyclic loading and the modulation of crack growth in the presence of hydrogen after overloads will be presented. The generalization of the hydrogen-cyclic load interaction in steels through analytical solutions is presented. The combination of stress range and time are found to be the most definitive parameters in hydrogen enhanced fatigue.

Role of chemistry of service environment in determining engineering failures

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Failure of engineering components remains an important issue for any industry, despite the substantial advantages in design, choice of materials and advances in non-destructive inspection. The analysis of every single failed piece-either following stipulated life or premature failure- is important for the prevention and prediction of similar situations. The objective of failure investigation is to determine and explain the causes leading to failures. This makes failure analysis an important feedback to the engineers who design any component with careful tolerances and provide factors of safety. The causes for failures could be either due to one or combination of the following: (i) Improper choice of material, (ii) poor quality of the material, (iii) production-induced abnormalities, (iv) unexpected service environment. Among the above, the role of service environment in initiating corrosion depends on its chemistry which may get altered during service, either by known reasons or unknown factors. The change in the atmosphere that is in contact would make conditions favourable to initiate different forms of corrosion. It is observed that change in atmospheric content of halide ions results in either pitting or stress corrosion if the component. On the other hand, generation of oxidizing atmosphere or carburizing atmosphere results in superficial layers that may lead to softening or hardening of the component at the surface. Similarly if the liquid metal in contact were to change its state to oxide or hydroxide, caustic embrittlement ensues if the

component were in elevated temperature service. Based on author's experiences in the failure analysis of engineering components exposed to liquid metal sodium, the present talk would discuss some case studies highlighting the chemistry of the environment and its effect.

Session 3: Nov 15, 2014: [14:00 - 16:00]

Advanced Corrosion Resistant materials for sustainable future

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The demand for corrosion resistant materials is forecast to continue its steady growth over the next few yearsworldwide. It is realized that this will be mainly be driven by the growth and development of newer stainless steels offering good value. Following are the megatrends that drive growth in stainless steel and high performance alloys.Economic & population growth, Increasing urbanization and Climate change and limited resources.These drivers have significance globally but have been the key factors for pushing up the demand in the Asia pacific region and other developing countries.Stainless steel grades range from standard to high-alloyed austenitic stainless steels,to ferriticand to martensitic stainless steelsfor normal to aggressive environments at ambient as well as high temperatures. Also the relatively new category is the duplex grades of stainless steel which are becoming very popular for typically more severe environments.All these grades of Stainless steel are developed to meet different needs of the customers. The presentation brings out details on some of the new grades developed in the last few years, which can be cost effective alternative suitable for substitution to various applications across industries including the most demanding industries viz.,chemical, petrochemical , fertilizer and power.Here we present some of these new developments .The new lean duplex grades like LDX2101 and LDX2404 which offers very high mechanical strengths along with their very good corrosion resistance.These grades have become very popular globallyfor storage tanks & vessel constructions in wide spectrum of industries specially food, beverages, chemicals etc.The new development of formable duplex range FDX25 & FDX 27, which improves the formability characteristics of duplex range. Typical application is plate type heat exchangersThe latest addition is 316^{plus}– a variant of 316L with enhanced corrosion and mechanical properties at lower cost. This is still under approval stage with ASTM/UNS standard committee (EN approval completed- 1.4420). This will be a cost effective alternative for standard 316L in the future for industrial applications, for e.g. pressurevessels, heat exchangers.The new developments in ferritic grades – viz. 441, 443 & 444 which are at par in terms of the corrosion resistance properties of austenitic grades like 304 & 316. A cost saving alternative adopted by many for building, facade and cladding applications, to the costly Ni bearing austenitic grades like 304 & 316.

Contributory papers

Session 1: Nov 14, 2014: [08:30 - 10:30]

Corrosion studies of near amorphous Ni-W alloy coatings

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Nickel-Tungsten alloys have many applications out of which the corrosion properties are noteworthy and a good substitute for hard chrome coatings. The current work deals with the influence of pulse parameters and thereby the effect of Tungsten (W) content and Grain Size (GS) on the corrosion behaviour of near amorphous Ni-W alloy coatings. The Ni-W coatings were produced on mild steel substrate by pulse electro-deposition method (PED). The corrosion behaviour of the coating was studied in 2N H_2SO_4 solution using electrochemical impedance and polarization techniques. The study showed that there is a critical grain size for which corrosion rate has a minimum value. Such critical grain size appears to be between 4 and 6 nm. On the other hand Ecorr, Epassivation and break-down potential (Eb) and the passivation region (Eb-Ep) of the coatings were also found to have a critical grain size for which they have a maximum value. Interestingly, this critical grain size also appears to be between 4 to 6 nm for all the quantities. With the variation in the tungsten content, there was a striking similarity in the trend observed for the corrosion rate (C.R.), Icorr and Ipassivation. The corrosion products were characterized by scanning electron microscope (SEM) and showed that the surface was covered by tungsten oxide in some places indicating that the oxygen present in the corrosive medium had preferentially reacted with the tungsten in the Ni-W alloy coating. Cracks were also observed on the surface of the coating which maysuggest that the Pilling-Bedworth ratio(PBR)of Ni-W alloy is high (greater than 2).

Influence of high chloride concentrations and temperatures on passivity of AZ91D Mg alloy

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The advantages of Mg alloys relative to other engineering materials are due to its superior mechanical properties, electrical thermal conductivity relative to plastic, higher specific strength and ductility relative to aluminium and lower machining and casting costs relative to steel. However, their applications are still limited due to their relatively low corrosion resistance. In this study, the corrosion and passivation behavior of magnesium alloy (AZ91D) in different concentration of sodium chloride solutions and temperatures were investigated. Electrochemical impedance spectroscopy test of AZ91D was performed in 3.5 wt% NaCl solutions of 0.4, 0.6, 0.8 & d 1.0 molar (M) after very fine alumina polished conditions. The passivation behavior was also observed at 45, 55 and 65 °C in 3.5 wt% NaCl solutions. Nyquist plots have shown a sharp reduction in semicircle radius and lower polarization resistance (R_p) on addition of higher chlorides concentrations and temperatures. The total polarization resistance was calculated after best fitting of experimental plots and noted about 5530, 4030, 2465 and 2000 Ohms.cm^2 in 3.5 wt% NaCl solutions of 0.4, 0.6, 0.8 & 1.0 M respectively indicates that the film stability is reduced at higher chlorides concentration. A similar trend was observed on increasing temperatures of 3.5 wt% NaCl solutions at 45, 55 & 65 °C and found 970,600 and 300 Ohms.cm^2 respectively.

Effect of amount of available salt on hot corrosion of Ni-base superalloy CM-247LC

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Hot corrosion is an analogue of aqueous corrosion except that it is associated with higher temperatures and leads to much higher corrosion rates. It takes place in the temperature range of 650 °C – 1000 °C in

the presence of molten sodium sulphate (Na_2SO_4) salt with or without other species such as vanadium pentoxide (V_2O_5) and sodium chloride (NaCl). Hot corrosion can be a major issue in advanced gas turbine engines that are being developed by DRDO, especially those operating near sea shore. The present study examines the hot corrosion behaviour of CM-247LC superalloy with respect to the amount of Na_2SO_4 salt available for corrosion at a temperature of 950°C. The hot corrosion study of the above alloy was conducted by depositing salt and exposing these salt coated samples at 950°C in air. The durations of exposure varies up to a maximum of 200h. Change in weight of the samples was measured intermittently. To study the effect of the amount of salt available at beginning of the test on the hot corrosion process, three levels of Na_2SO_4 salt on the samples namely 3-4 $mg.cm^{-2}$, 7-9 $mg.cm^{-2}$ and 12-14 $mg.cm^{-2}$ were tested. The microstructural characterization of the corroded samples was carried out by using SEM, and XRD. The results showed that the above superalloy has very poor hot corrosion resistance. This is evident from the enormously high weight gain after hot corrosion exposure as compared to that obtained under pure oxidation. The weight gain after hot corrosion exposure was also found to increase with increase in the initially deposited amount of salt. SEM analysis of the hot corroded samples revealed a multilayer structure of the corrosion product. This structure contains porous oxides of alloying elements, an inter-granular corroded region, an alloying element depleted region and a hot corrosion front with sulphide particles.

Corrosion of AISI 202 and AISI 304 austenitic stainless steels in various H_2SO_4solutions using polarization method

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Austenitic stainless steels especially AISI 304 are widely used in a variety of industrial applications. However due to rising cost of nickel, nickel-free or low-nickel austenitic stainless steels similar to AISI 202 are being looked upon as a substitute to AISI 304for some applications. Present investigation, part of which is reported here, deals with studying corrosion behavior of AISI 202 as compared to AISI 304 in various solution of H_2SO_4. Potentio-dynamic polarization method was used in the investigation. Both the stainless steels showed passivity-like limiting current region in anodic polarization plot. The E_{corr} for 0.1M H_2SO_4 and 0.5M H_2SO_4 was almost same both showing shifts in noble direction in 1M H_2SO_4 solution. It was observed, that AISI 304 passivates instantaneously whereas, AISI 202 undergoes free corrosion for some time before the current density (c.d) decreases and attains limiting current condition. The current density at limiting current region was in the range of 6 to 8 mA for both steels. This c.d was quite high to be taken as passivity. Therefore, this feature can be termed as pseudo passivity. Break down potential for AISI 202 was higher than that of AISI 304 for all the test solutions. This showed that for these solutions AISI 202 was almost equivalent to AISI 304. The polarization resistance was also examined EIS (Nyquist plots). It was observed that increase in the concentration of H_2SO_4 decreases the polarization resistance in both the stainless steels. However, polarization resistance of AISI 202 was less than that of AISI304.

Corrosion behavior of AISI 304 and AISI 2205 SS: role of surface mechanical attrition treatment (SMAT)

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Stainless steels are well known for their non-corrosive behavior which suits their application in the corrosive atmosphere. At the same time they have poor wear resistance due to their low surface hardness, which limits their use in the diverse field of applications. Surface mechanical attrition treatment (SMAT) is one of the surface severe plastic deformation processes in which repetitive multidirectional ball impact (ball size ~ 2-10 mm, velocity~1-20m/s) on the surface induces accumulative strain, which results in grain refinement up to nano scale level as well as high hardness compared to core matrix without modifying chemical composition. In this study, SMAT was performed on the AISI 304 and AISI 2205 stainless steels using 3 mm ball diameter with three different processing times of 15, 30 and 60 minutes. Surface hardness was found to be increase about two times (400 $HV_{0.1}$) along with surface roughness (R_a ~ 0.7µm) for both stainless steel grades after SMAT. The increased hardness was found to be due to the formation of strain induced martensite formation. Electrochemical corrosion behavior was examined for AISI 304 SS and AISI 2205 SS after and before SMAT using Tafel extrapolation and cyclic polarization in solutions of 3.5 wt % NaCl. Electrochemical corrosion kinetics parameters such as corrosion potentials, corrosion current densities, corrosion rate and passive current density were calculated from experimental polarization plots. The results show that corrosion resistance reduced due to SMAT for AISI 304 and 2205 SSs and were highly dependent on surface finishing of alloys when compared with higher surface finished conditions. AISI 304 SS showed more deleterious effect on the corrosion resistance over AISI 2205 SS. A high passive current density was noted for AISI 2205 SS at higher SMATing time of 90 and 120 min.

Effect of cold rolling on sensitization susceptibility of 216L stainless steel

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Stainless Steel is often the material of choice for making components of various industrial equipments as they have good weldability, fabrication and high temperature mechanical properties. Among the various grades, austenitic stainless steels posses low yield stress and comparatively high ultimate stress with respect to plain carbon steel of the same carbon content. Recently, the 216L grade has been developed where a part of nickel (316L) has been replaced by manganese. Despite their advantages, the steel undergoes sensitization when exposed to a temperature range of 500 ^{0}C to 850 ^{0}C. Cold working of these steels introduces several strained regions which serve as nucleation sites for the precipitation of chromium

carbides. The objective of the work was to understand the sensitization susceptibility of 216L, especially under the influence of deformation prior to sensitization. The material was received in the form of 6 mm thick sheets. Coupons were cut using SERVOCUT-M250 abrasive cutter and subjected to unidirectional cold rolling upto 25-80% reduction in area at room temperature. Rolled plates were then cut in the dimension 15mm X 10mm cold rolled sheets including as-received. Specimens were subjected to heat treatment at temperatures 600^0C for 100 hours followed by water quenching. These samples were metallographically prepared by grinding using belt grinder, emery paper from 180 to 1200 grit and subsequent polishing on METASERV 3000 cloth polishing wheel using alumina (5µm) as abrasive at 350 rpm. Samples were degreased with acetone followed by masking with epoxy resin to expose a known surface area to the test solution. The test was conducted at room temperature using a solution of 0.5 M H_2SO_4 + 0.01 M KSCN by GAMRY Reference 600 Potentiostat/Galvanostat/ZRA. After DL-EPR test, the specimens were seen under LEICA DM 2500 M optical microscope for qualitative analysis and comparison of the microstructure.

Corrosion behaviour of galvanized steel in different corrosive environment

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Corrosion accounts for colossal loss of materials occurring everywhere and every moment involving billions & billions of rupees annually. It is one of the major technological problems of modern society. Although, it is a natural complex phenomenon, it can never be completely eliminated but can be delayed by utilizing adequate corrosion protection methods that lasts for decades even in the harshest environments. For more than a century, hot dip galvanized steels are used extensively to combat corrosion in major industrial environments including transportation, petro-chemical and public utilities. The zinc of the hot dip galvanized coating is more corrosion resistant than bare iron and steel. Similar to steel, zinc also corrodes when exposed to the atmosphere; however it corrodes at a rate approximately $1/30^{th}$ of that for steel. Corrosion rate depends upon its environment. So, performance of hot dip galvanized steel varies from environment to environment. Environment includes atmosphere, soil, fresh water, sea water, chemical solution, treated wood, concrete, extreme temperature etc. The exposed samples were also analyzed by using various characterization techniques such as XRD, SEM etc.

Stress corrosion cracking studies on 304L borated stainless steel in acidic chloride medium

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Borated stainless steels (SS) are used as neutron absorbers in nuclear industry. The corrosion behavior of borated SS is significantly different due to the presence of secondary phases in the form of Fe_2B or Cr_2B. In the present work, uniform, pitting and stress corrosion cracking (SCC) behavior of borated 304L SS grade B was studied and its performance compared with the conventional 304L SS in as-received, sensitized (675 ^oC/1 h) and solution-annealed (1150 ^oC /2 and 4h) conditions. Potentiodynamic anodic polarization experiments were carried out in 0.5M H_2SO_4 and 0.5M NaCl solutions to study the general and pitting corrosion resistance. Degree of sensitization (DOS) was determined by electrochemical potentiokinetic

reactiactivation (EPR) tests. SCC tests were conducted and time to failure and strength parameters were evaluated. Borated 304L SS in the as-received condition showed irregular boride particles dispersed in the matrix. While borated SS showed better passivationcharacteristics in 0.5M H_2SO_4 compared to non-borated 304SS, the former also showedhigher pitting potential (E_{pit}) values in 0.5M NaCl compared to the latter. After sensitization, borated 304L SS retained its passivity, whereas non-borated 304L SS showed total loss of passivity and started to pit at E_{corr} itself. Specimens solution annealed at 1150 ^oC/4 h showed improved uniform and pitting corrosion behavior compared to that at 1150 ^oC/2 h. Extensive pitting corrosion was observed in borated 304L SS along particle/ matrix interface due to fine Cr- depleted zones along these interfaces. The time to failure values for SCC in case of as-received, sensitized and solution annealed (1150 ^oC /2 h and 4 h) borated 304L SS specimens were found to be much higher than those observed for 304L SS, suggesting improved SCC resistance for borated 304L SS. The mode of failure was found to be transgranular in all the conditions, except in the sensitized condition wherein the specimen showed a mixed mode of failure due to Cr-depleted grain boundaries.

Mechanism and kinetics of reinforced steel bar corrosion in concrete

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Thermomechanically treated steel bar rolled from low alloyed steels are popular for use in concrete structures, due to improved mechanical properties, weldability and bonding with concrete. Under normal conditions, these steels in concrete form a compact passive film on its surface in concrete pore solution with high alkalinity (pH 12.5-13.5), increasing its corrosion resistance. However, the passive film can be locally damaged and the localized corrosion can initiate when pH and chloride concentration at the steel/concrete interface reaches critical values for corrosion. The pH value of concrete pore solution is one of the most important parameters affecting the corrosion behaviour. The most important causes of corrosion initiation of these reinforcing steel are the ingress of chloride ions and carbon dioxide to the steel surface. Corrosion products such as iron oxides and hydroxides are usually deposited in the restricted space in the concrete around the reinforced steel. Their formation within this restricted space sets up expansive stresses, which may crack and finally spall the concrete cover due to their porous nature. In spite of the extensive studies of corrosion behaviour of reinforcing steel, the exact mechanism of its depassivation is still unclear. In this study, electrochemical tests of thermomechnical treated steel bar used in reinforced steel has been carried out in different corrosive environment to know the corrosion behavior of the steel. Also the corrosion products formed on the surface of the materials were extensively studied by various characterization techniques using SEM XRD and OCP. It is observed that some of the corrosion products e.g. ferrous hydroxide $Fe(OH)_2$lepidocrocite (γ-FeOOH), goethite (α-FeOOH), akaganeite (β-FeOOH) and magnetite (Fe_3O_4) are protective in nature as its remain stable and thin. However, some of the products are porous in nature that results in further proceeding of corrosion process and grow with time.

Corrosion of rebars and its prevention

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Rebar steel is an important building material which is basically used for construction works. This metal is reinforced with concrete for construction of bridges and houses. Such types of bridges and houses face corrosive problems over the period. It is one of the main causes for the limited durability of steel reinforced concrete. In a hardened concrete structure, diffusion of oxygen, carbon dioxide, ions (primaliry chlorine) and moisture from the concrete/environment to the concrete/rebar interfaces takes place through the pores which results in the failure of the passivation provided by the alkalinity of the cement to the rebars. The corrosion product resides at the interface between steel rebars and concrete, thus degrading the bond between steel rebar and concrete. This involves billions of dollars of loss through repair and maintenance needed to keep the facilities functional. The aim of the present investigation is to evaluate the effect of manufacturing process upon the performance of reinforcing steel bars against atmospheric corrosion and chloride environment. Reinforcing steel bars produced by different manufacturing methods were exposed to the atmosphere and in salt spray for different periods of time. The progress of corrosion was evaluated by measuring the thickness of the corrosion layer formed on the surface of the bars and also by weight loss method. The rate of corrosion was also measured by Electrochemical Impedance Spectroscopy (EIS) study. The morphology of the corroded layer was studied by optical and scanning electron microscopy (SEM), while X-ray diffraction analysis was used to identify the mineralogical composition of the corrosion products. It was found that the surface condition and the initial oxide layer on the steel bars, which is closely related to the manufacturing process, played a major role in the development of corrosion both in atmosphere as well as in chloride solution.

Study on pitting defect in grade E46 HR coils

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E46 grade Hot Rolled coils of thickness 7mm are used to make Long Members (LM) of truck bodies. Surface pitting in long members is one of the critical issues at customer end. Work was carried out to study the formation of pitting defects in grade E46 HR coils supplied to customer. A cluster of dents, shallow in nature, were observed on the surface of the Long Members. When the issue of pitting was first reported, the defect was caught at TSPDL, Bara and the source of pitting was traced back to Stripper Car Roll at Hot Strip Mill. Subsequently, the roll was changed, but the pitting issue continued to be reported. Rejection of LMs due to pitting was done only after the forming process at customer end. Various touch points of strip path were studied to understand the possibility of formation of pitting on coils. Pitted samples were collected from the customer and were subjected to metallographic analysis to understand the root cause of the defect. Similar issue was reported at another customer also from a similar grade of HR Coil, only of a lesser thickness. A sample was collected and analyzed and the results of both these analysis were compared. At another customer sample, dent was repetitive with a pitch matched with the leveler roll circumference. In case of previous customer sample, presence of embedded scale could be spotted in some dents at higher magnification. Presence of scale in the dent region showed that this defect could

arise from embedded scales on straightener rolls or due to embedment of loose scales on the surface during levelling/pressing operation. Based on the study, it was recommended to look into the possibility of reducing loose scales by controlling the coiling temperature and to strengthen the strip path management (w.r.t wrapper rolls, ROT rolls, stripper car rolls) at Hot Strip Mill.

Recreation of stable rust on constructional steel for corrosion protection

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Various accelerated tests are designed to assess atmospheric corrosion in laboratories to understand indicative corrosion behavior in service condition. Both mild steel and weathering steels (WS) were exposed for long duration at places like industrial and normal marine environment. Through rust analyses, stable oxides/hydroxides phases were found on WS whereas mild steel did not show any stable phases. To get stable rust on WS, about 3 years are required and during this period material suffers loss. Hence, attempt has been made to create stable rust early so that corrosion loss may be prevented. Experiments were designed to investigate the passivation behaviour by slow polarization and to see whether such oxides/hydroxides phases at all formed or not. Na_2SO_4 solution was taken to create SO_2 insitu for industrial environment and passivated oxide layers were found only on WS. Raman spectroscopy showed γ FeOOH along with stable Fe_3O_4 and α Fe_2O_3, which are very similar to the rust of WS exposed and morphology of the simulated rust compared very well when observed in SEM. Trials were also done to simulate the normal marine environment with 0.01 (N) KCl (pH 8.8) to create Cl in situ. Raman characterization showed the presence of γ FeOOH along with α Fe_2O_3, γ Fe_2O_3, δ FeOOH and α Fe_2O_3, α FeOOH, which were very similar to the rust formed. EIS analyses of WS showed that the effective resistance of the rust for 45mins passivation was greater.

Electrochemical corrosion behaviour of mild steel pipe in alkaline soil

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This study discusses corrosion of mild steel pipelines buried in soil. For this, mild steel pipe coupons were buried in three different soils for a period of seven months. Soil samples were collected from three different locations covering approximately 20 km distance from Pune. The collected soil samples were tested for soil resistivity, soil pH, soil moisture content, soil redox potential and chemical composition. Corrosive properties of soil samples were in the order of soil 1 > soil 2 > soil 3. The corrosion potential and corrosion rate of buried coupons were studied by EIS and TAFEL polarization technique. Characterizations of corroded specimens were done with the help of XRD, that showed most commonly found corrosion products such as iron oxide hydroxide, Lepidocrocite (γ-FeOOH), Goethite (α-FeOOH), Feroxyhyte (δ-FeOOH), Hematite (α-Fe_2O_3), Magnetite (Fe_3O_4) and Maghemite (γ-Fe_2O_3). Scanning Electron Microscopy attached with Energy Dispersive Spectroscopy was used to observe surface morphology of corroded specimen and elemental analysis. SEM micrograph showed typical structure of geothite and lepidocrocite. From the SEM images it was observed, compared to the surface morphology of steel pipe specimens buried in soil 1, soil 2 specimens were more prone to cracking and flaking. However, corrosion products

were uniformly distributed on steel pipe specimen surface buried in soil 3. Mild steel coupons buried in soil sample 1, showed more corrosion than the other two soils due to more corrosive properties of soil. Corrosion rate of steel pipe specimen in soil 1, soil 2 and soil 3 after seven months of soil exposure were 64.21 mpy, 53.22 mpy and 16.35 mpy respectively.

Characterisation of naturally and artificially set Ag-Sn-Cu dental amalgam

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Dental amalgams, alloy of Hg and Ag-Sn-Cu-(Zn) are being used for the past several decades as filling/restorative materials to dentistry. These dental amalgams are susceptible to wear, damage and often breaking during mastication and chewing and also undergo corrosive attack in the hostile oral saliva environment which is bombarded with chemical, physical stimuli and metabolism of about several species of bacterial counts. Further, the pH of the oral saliva may vary from 2 to 11 and the temperature also does vary from 0^0C - 60^0C while coping with cold of ice to hot coffee and soup. Dental amalgams are prepared with approximately 1:1 ratio of Hg and single composition lathe-cut Ag-Sn-Cu alloy powders. After trituration and condensation, amalgams are allowed to set naturally at room temperature and followed by isothermal ageing at temperature 60 ^{0}C (artificial setting) as well for varying time. The microstructural changes upon natural and artificial setting have been assessed by SEM, XRD and by hardness measurement. Further, to assess corrosion of amalgams, electrochemical polarization study has been performed in artificial saliva. Attempts have been made to explain the hardening behaviour and the electrochemical behaviour of amalgams with the help of microstructural features and formations of phases with natural and artificial ageing.

Evaluation of corrosion resistance behavior of boron containing dual phase steel

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Dual phase (DP) steels are named on the basis of crystallography. Their microstructure consisting of a soft ferrite matrix containing islands of martensite as the secondary phase (martensite increases the tensile strength). DP steels possess better formability characteristics than that of ferrite-pearlite steels of similar strength making them more suitable for applications such as automotive bodies and construction industries. The mechanical properties of DP steels can be manipulated by heat treatment or addition of boron in small quantity. In the present work, DP steels with and without boron content were evaluated for their corrosion resistance properties through potentiodynamic polarization test, electrochemical impedance spectroscopy and open circuit potential measurement. The corrosion behavior of boron containing DP steel was compared with that of the ferrite-pearlite steels and DP steels. Boron containing DP steel shows better corrosion resistance than DP steel without boron in it. Microstructural characterization of DP steels and Boron containing DP steel was carried out using optical microscope.

Characterization of the corroded surfaces was accomplished using scanning electron microscopy and electron probe micro-analyzer.

Hot corrosion studies on Ni-base superalloy at 900°C

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Ni-base superalloys have been usedas gas turbine engines components such as combustion chamber and linersin marine environments. However, these alloys are vulnerable to attack of molten salts such as Na_2SO_4, NaCl and V_2O_5 in marine environment. The aim of the present investigation is to study the cyclic and isothermal oxidation behaviour of GTM-SU263 alloy at 900°C with 75wt.% Na_2SO_4 + 22 wt.%NaCl + 3 wt.%V_2O_5 salt mixture (3SM) for a duration of 100, 200, 300, 400 and 500h. Cyclic oxidation tests with this salt mixture were compared with 90wt%Na_2SO_4 + 10wt%NaCl (2SM) and without salt. Isothermal oxidized samples with 3SM were characterized by detailed XRD and SEM studies to evaluate the influence of salt mixture on hot corrosion behaviour.

Thermophysical behavior of yttria stabilized zirconia based graded thermal barrier coating developed by plasma spray deposition technique

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The present study concerns the evaluation of thermal properties (thermal expansion, thermal diffusivity, and thermal conductivity) of the Yttria stabilized zirconia (YSZ) based compositionally graded thermal barrier coatings developed by thermal spraying route and understanding the role of defects content on it. Effect of CoNiCrAlY and Al_2O_3 addition to YSZ on the coefficient of thermal expansion and thermal conductivity of the graded coating was studied in detail. Variation of coefficient of thermal expansion and thermal conductivity of the CoNiCrAlY/YSZ and Al_2O_3/YSZ composite coatings with temperature were also studied. Coefficient of thermal expansion of the composite coatings was found to increase with increase in temperature of exposure. The thermal conductivity of Al_2O_3/YSZ composite coatings was found to decrease with increase in temperature. However, in CoNiCrAlY/YSZ composite coatings, the thermal conductivity was found to increase after attaining 400 °C. An attempt was also made to investigate the effect of porosity and its shape on the effective thermal conductivity of the composite coatings by analytical modeling.

Session 2: Nov 15, 2014: [11:00 - 13:00]

The stress-corrosion cracking susceptibilityof Ti-6242S alloy in presence of hot salt

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The near α titanium alloy, Ti-6242S (Ti-6Al-2Sn-4Zr-2Mo-0.1Si), possesses creep and oxidation resistance up to 520 °C, and is being used in the initial stages of the high-pressure compressor components of aero gas turbine engine for which the temperature exceeds 350 °C, where Ti-6Al-4V cannot be used because of creep considerations. However, the titanium alloys suffer from hot salt stress corrosion cracking (HSSCC), when exposed to halides in the temperature range of 200 to 500 °C. Literature on Ti-6242S alloy in relation to HSSCC is scanty, even though this work was started during early 70s. Hence, this study was undertaken. The hot salt stress corrosion cracking susceptibility of Ti-6242S alloy was evaluated using a slow-strain-rate test technique in the temperature range 250-400 °C. The alloy showed mere susceptibility to HSSCC at 300 °C, the susceptibility increases significantly with increase in test temperatures. The cracks found initiate at the salt attack regions and along the slip lines; and grew transgranularly in primary α and across the α+β lamellae in transformed β regions.

Susceptibility to hydrogen embrittlement of tempered type 420B martensitic stainless steel

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Martensitic stainless steels (MSS's) find applications where good mechanical properties and moderate corrosion resistance are required. These steels are used for turbine components, steam generators, valve parts of pressure vessels, cutlery, machine parts and offshore platforms for oil exploration. In this study, the influence of hydrogen on the hydrogen embrittlement (HE) of tempered 420B MSS were studied by slow stain rate testing (SSRT) at a strain rate of 10^{-6}/sec during hydrogen charging with different current densities and also on hydrogen precharged tensile specimens at room temperature. The electrochemical hydrogen charging was done in 0.05 M sulphuric acid solution. The specimens were austenitized at 1020 °C for 0.5h followed by oil quenching and subsequently tempered at 300, 550 and 700 °C for 2.5 h followed by air cooling. The mechanical properties of the tempered specimens were evaluated by SSRT and fracture surface analysis was done to explain the mode of fracture. The SSRT studies on tempered specimens showed that 700 °C tempering treatment is the optimum for mechanical properties in terms of yield strength and ductility. The SSRT studies on 550 and 700 °C tempered tensile specimens which were hydrogen precharged for 15 h showed that the strain to failure was more in 700 °C than 550 °C tempered specimens. The fracture surface analysis showed intergranular cracking along the prior austenitic grain boundaries in both tempered specimens. The susceptibility to HE of 700 °C tempered specimen with different current densities of cathodic hydrogen charging showed that the strain to failure was increased with decreasing current density. The fracture surface analysis showed that the mode of failure was brittle

with higher current densities (20 and 5 mA/cm^2) and it was mixed mode (ductile -brittle) in lower current density (0.01mA/cm^2).

Effect of hydrogen on fatigue crack growth in a high strength aluminium alloy

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High strength aluminium alloys of 7xxx series (Al-Zn-Mg-Cu) have found extensive use in aerospace structural components such as wing spars of aircraft. The fatigue behaviour of Al alloys is greatly influenced by the environmental conditions. Al alloys, especially high strength alloys are susceptible to environmentally assisted crack growth. The peak-aged (T6) temper high-strength Al alloy, 7075 T6 AA, are highly susceptible to either stress corrosion cracking or hydrogen embrittlement, thereby resulting in intergranular fracture. The stress corrosion and corrosion fatigue of high strength Al alloys have been extensively investigated. A small number of work reported in literature dealt with hydrogen induced fatigue of Al alloys in chloride environment. It is necessary to establish the mode of hydrogen transport near the crack tip, the hydrogen availability and surface coverage and the contribution of hydrogen at crack tip towards crack growth. Unlike the crack initiation event, the crack growth mechanism and its driving force are not well understood for hydrogen embrittlement (HE) conditions. In this study, the fatigue crack growth rate (FCGR) for high strength Al alloy (7075 T6 Al alloy) was studied with in-situ hydrogen charging. Hydrogen induced fatigue tests were carried out in 3.5% NaCl solution in a servo-electric machine at room temperature using three point bend specimens. Hydrogen was introduced by cathodic charging at a constant current density with the help of a galvanostat. The fatigue crack growth rate tests were conducted with constant stress intensity factor range (ΔK) protocol with load ratio (R) and frequency variation. The effects of different parameters, like stress intensity factor range (ΔK), load ratio (R), frequency and stress values have been investigated. Fracture surfaces of hydrogenated specimens and crack morphologies were studied using scanning electron microscope (SEM) analysis.

Ageing response and electrochemical behaviour of 2xxx series Al-Cu-Mg alloys of various tempers

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Artificial and natural ageing study of AA2024 and AA2014 aluminium alloys showed characteristic precipitation hardening phenomena. Potentiodynamic polarization studies carried out on both the alloys of various tempers in 3.5 wt.% NaCl and in 3.5 wt.% NaCl in presence of a small amount of H_2O_2 solution exhibited shifting of corrosion potential (E_{corr}) towards more negative potential with the increase of aging time. The E_{corr} values for the alloy tempers have shifted anodically in presence of H_2O_2 in 3.5% NaCl solution. Further, passivity phenomenon has been observed in all the alloy tempers when tested in 3.5wt.% NaCl solution at pH 12. Optical micrographs of the corroded surfaces of polarised samples showed general corrosion, extensive pitting and intergranular corrosion as well. Further, potentiodynamic cyclic polarization curves displayed wide hysteresis loop indicating that the alloy tempers are susceptible to pit

growth damage. Attempts have been made to explain the variation of observed electrochemical behaviour of various alloy tempers and the electrolyte conditions with the help of microstructural features.

Influence of surface mechanical attrition treatment on the corrosion performance of AA7075

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Aluminium alloy 7075 (Al–Zn–Mg–Cu alloy system) consists of intermetallics ($MgZn_2$, $CuAl_2$) which result in heterogeneity of microstructure. The microstructural heterogeneity essentially influences the corrosion properties of the alloy. Aluminum alloy 7075 when artificially aged possesses high strength, but is highly susceptible to stress-corrosion cracking. This can be avoided by retrogression and reageing (RRA) heat treatment without affecting mechanical properties. Various conventional surface modification techniques are developed for improving corrosion resistance. This led to development of novel surface mechanical attrition treatment (SMAT) technique the so called ultrasonic shot peening which gives nano grain structure at surface. Aluminium alloy 7075 was shot peened to produce nanograins at surface and the corrosion behavior was studied in 3.5wt% NaCl solution. SMAT was carried out by peening of the surface with steel balls of 1 and 3 mm diameter, at an ultrasonic frequency, for a time interval of 5, 15 and 30 minutes. As-SMATed sample with 1 mm balls exhibits better corrosion resistance as compared to that of un-SMATed sample. In contrast, the samples treated with 3 mm balls showed poor resistance to corrosion. A decrease in corrosion resistance with increase in peening time was observed. The decrease in corrosion resistance in shotpeened samples with balls of larger diameter may due to increase in microstrain and defect density.

Corrosion behavior study of novel Mg-Li-Al based alloys in dilute chloride solution

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This study investigates new findings on the influence of multiphase structure on corrosion behavior of thermomechanically (TM) processed Mg–Li–Al based alloys, namely Mg–9wt%Li–7wt%Al–1wt%Sn (LAT971R) and Mg–9wt% Li–5wt%Al–3wt%Sn–1wt%Zn (LATZ9531R), in dilute sodium chloride (0.01M NaCl) solution. Understanding the corrosion mechanism of multiphase Mg-Li-Al based alloys, which is important for its wide application and has not been adequately reported in the literature, is finally articulated based on the early stages ex-situ surface morphology evolution of both Mg–Li–Al based alloys after 20sec, 60sec, 300sec, 1200sec and 3600sec immersion in 0.01 M NaCl. Present paper also describes the corrosion mechanism of Mg-Li-Al based alloys in chloride solution utilizing scanning electron microscope (SEM) and energy dispersive spectrometry (EDS).

Investigating the formation and corrosion inhibition imparted to an aluminium alloy AA2024-T3 by a trivalent chromium process (TCP) coating

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Aluminium alloys are susceptible to corrosion when exposed to the ambient environment. TCP coatings are formed by immersion and are available from several commercial suppliers. The various commercial coating baths have distinct chemical compositions such that it is important to understand the formation, structure and corrosion inhibition of each. In this work, we used electrochemical methods to study the corrosion inhibition of a Metalast TCP-HF conversion coating on an aluminium alloy (AA2024). Measurements for 2 different method of sample preparations were made in aerated electrolyte at room temperature: 0.5 M Na_2SO_4 (mild).The first one was that of commercially used Alodine-5200, while the other one was as standardised for Metalast coating. Generally speaking, the conversion coating is 50-100 nm thick and consists mainly of hydrated zirconia ($ZrO_2.2H_20$) and a trivalent chrome salt. The coatings were characterised using Scanning electron Microscope and Atomic force microscopy. The electrochemical parameters studied were the open circuit potential, impedance-frequency spectra at the open circuit potential, linear sweep voltammetry to determine the polarization resistance, cathodic and anodic polarization curves to determine the corrosion currents and Tafel slopes and a comparison between the two methods of preparation was done. Presence of Cr(VI) bonds in the coatings were evaluated by Raman Spectroscopy.

Effect of salt coating on hot corrosion behavior of superalloy IN 718 at 600°C

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Nickel based superalloys are used in components of gas turbines and their performance is drastically affected by environmental conditions. Low temperature hot corrosion tests were conducted on superalloy IN718 (Peak aged) at 600°C for 1000 hours by coating three different salt mixtures (100% NaCl and 75% Na_2SO_4+25% NaCl and 90% Na_2SO_4+ 5% NaCl+ 5% V_2O_5). It was observed from the XRD and EDS analysis that there was no oxidation of samples without salt coating as no new phase was observed, but in case of salt coated samples Cr_2O_3, Fe_3O_4 and NiO were the main oxide phases found in addition to other oxides. Weight gain/loss measurements were carried out to determine corrosion kinetics. NaCl coated sample was found to be most damaging among the salts studied.

To study on synthesis and hot corrosion behaviour of lanthanum cerate ($La_2Ce_2O_7$) coated with V_2O_5+Na_2SO_4 at 650°C and 950°C

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The hot corrosion behaviour of sintered lanthanum cerate ceramic in V_2O_5+Na_2SO_4 environment has been investigated over the temperature range 650-950 °C. The hot corrosion products were identified using X-ray diffraction and scanning electron microscopy. The corrosion products depend mainly upon corrosion temperature. At 650 °C, the corrosion products were $LaVO_4$ and $CeVO_4$ along with unreacted Na_2SO_4. However, at 950 °C, the reaction is almost complete and formed $LaVO_4$ and CeO_2.

Hot salt stress corrosion cracking behaviour of Ti-6Al-4V and IMI-834 alloys in presence of metallic coating

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Titanium alloys are prime choice of material for the compressor components of gas turbine engines because of their high specific strength. But, titanium alloys are generally prone to hot salt stress corrosion cracking(HSSCC), when exposed to halides at elevated temperatures As these alloys are generally used in metallic coated condition which gives abrasion, wear, fretting, erosion and oxidation resistance during service, it is important to study how they behave in the coated conditions. In this study, an attempt was made to study the HSSCC behaviour of alloys Ti-6Al-4V and IMI-834 in metallic coated condition. For this, Ti-6Al-4V alloy specimens were coated with Metco 58NS and Metco 450-101NS, while the alloy IMI-834 with Metco 58NS and Metco 450-307NS, respectively. In order to study the diffusion effect of salt, a time dependent phenomena, salt coated specimens were exposed for a duration of 500-2000 h in tubular furnace at 400°C and then subjected to slow strain rate tests at a strain rate of 1.4×10^{-6} $s^{-1}$and at 400 °C. No significant variations in mechanical properties in presence of salt environment were observed. The fractographic analysis using scanning electron microscopy revealed the presence of dimple features, characterizing ductile failure. Our study on alloy Ti-6Al-4V and IMI-834 showed that the coating offer resistance to HSSCC for both the alloys which are susceptible to HSSCC in absence of metallic coating in the temperature range of 300°C - 450°C.

Lanthanum hexaaluminate as advanced thermal barrier coatings: thermo-physical and mechanical properties, hot corrosion behaviour and thermal cycling life

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Neodymium doped lanthanum magnesium hexaaluminate (LNMHA) was synthesized through aqueous sol gel processing. The thermo-physical and mechanical properties of lanthanum magnesium hexaaluminate were altered by neodymium doping. The thermal conductivity of doped material was decreased whereas thermal expansion coefficient was increased significantly. The mechanical properties were also enhanced manifold as doping transforms the basic microstructure of porous interlocking type to a self-reinforced structure. This improved material showed higher thermal cycling life measured at 1400°C. The hot

corrosion resistance was also found to be much better than the parent system at 1050°C for 100h under molten sulfate-vanadate salts. The improved features of LNMHA ensure its potential use as advanced thermal barrier coatings.

Electrochemical and corrosion behaviour of β-Phase intermetallic (Mg_2Al_3) using spatially resolved droplet cell

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Aluminium-Magnesium alloys are used in ship building due to their weldability, formability and corrosion resistance. The solubility of Mg in Al is 3.5 wt.%. Higher Mg addition above the solubility limit helps in solution strengthening, but leads to deleterious precipitation of β phase (Mg_2Al_3) also called "Samson phase". The β-phase tends to increase the susceptibility for intergranular corrosion (IGC) and stress corrosion cracking (SCC). In order to study the corrosion behaviour of β-Phase, Al and Mg are cast with appropriate proportions following the phase diagram. For bulk/global corrosion studies, a larger area of the sample must be of a single β-phase, which involves longer duration of heat treatments. However in the present work, we utilize the concept of spatially resolved micro electrochemical technique to directly screen the β-phase particles which are of few hundred micrometers in size. We would discuss on the fabrication and challenges of such a tool to study corrosion behaviour of these precipitates. The microstructure of the specimens revealed several other phases, which could be tested for corrosion. The work would also highlight the effect of environmental friendly inhibitors on the corrosion rate these precipitates.

Study of cathodic protection in various engineering applications

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Deterioration of a material's properties due to its interaction with its environment is known as corrosion. Corrosion is a chemical or electrochemical oxidation process in which the metal transfers electrons to the environment and undergoes a valence change from zero to a positive value. Anode, Cathode, Electrolyte and an electrical conducting path between the anode and cathode, these are four necessary elements in order for corrosion to occur. Thermodynamics is used primarily to determine, mathematically, the tendency for corrosion to occur. The amount of Gibbs free energy in a system represents the proximity of the system to equilibrium. That is, the lower the free energy, the closer the system is to equilibrium and conversely, the higher the free energy, the less stable the system is. Corrosion protection methods are used to additionally mitigate and control the effects of corrosion. Forms of corrosion protection include the use of inhibitors, surface treatments, coatings and sealants, cathodic protection, and anodic protection. Cathodic protection is a highly adaptable and effective means of preventing corrosion on a variety of underground or underwater structures, such as canal gates, condensers, submarines, water tanks, marine piling, offshore oil – drilling structures, chemical equipment, bridge decks, parking garages, and other reinforced concrete structures, etc. There are basically two types of systems: namely, galvanic and impressed current. Each has characteristics which make it more adaptable' under given circumstances. Pipeline integrity management programs are used by pipeline operators to determine the locations in which corrosion defects pose a threat to safe operation. Cathodic protection designs can differ

considerably depending upon the coating, the configuration of the structure, the environment and the presence of neighboring structures. When a system is designed, installed and maintained properly, cathodic protection is one of the most effective and economical methods of preventing corrosion.

Organic coatings developed by nanotechnology - review

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Nanotechnology is regarded as one of the key technologies of the future. The term “nanotechnology” is referred as the manufacturing, analysis and use of structures, for example particles, layers or tubes of less than 100 nanometers (nm) in at least one dimension. Artificially produced nano-sized particles and nanoscale system components have new properties which are of importance for the development of new products and applications. This paper reviews different types of coatings, different types of pigments used in paint formulation and a special focus is set on the uses of nanomaterials /fillers in coating application. More importance is given to the development of nanocomposite coating.

Degradation behaviour of detonation gun sprayed Cr_3C_2-25NiCr coating on 304L in presence of Na_2SO_4-82%$Fe_2(SO_4)_3$ salt deposits at 900 °C

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The present work evaluates the hot corrosion resistance of detonation gun sprayed Cr_3C_2-25NiCr coating deposited on steel substrate (304L). Thermogravimetry technique was used to study the high temperature hot corrosion behavior of bare and coated specimens in molten salt environment, Na_2SO_4–82%$Fe_2(SO_4)_3$ at 900°C under cyclic conditions. Each cycle consisted of 1 hour heating at 900°C in a silicon carbide tube furnace, followed by 20 min cooling at room temperature (25°C). The oxide scale was characterized using the combined effect of X-ray diffraction, scanning electron microscopy/energy-dispersive analysis and X-ray mapping. The as sprayed coating showed dense microstructure consisted of uniformly distributed chrome carbide-rich ceramic grains attached with nickel chrome-rich binder phase with porosity less than 1%. The microhardness of coating was found to be in the range of 790–1060 Hv. Hot/accelerated corrosion occured on the bare substrate resulting in intense spalling of its oxide scale, where as coated specimen showed negligible spallation at the edges. The coating was adherent to substrate in aggressive environment during cyclic oxidation at elevated temperatures. The Cr_3C_2-NiCr coating provided necessary resistance to hot corrosion, attributed to the formation of oxides of active elements in the surface scale. The EDS analysis of the scale revealed the possible formation of Cr_2O_3 and NiO as the principal phases. The better corrosion resistance behaviour of the coating in aggressive environment was also enhanced due to low porosity and homogeneous distribution of fine grains of the coating.

Effect of corrosive environment on austempered ductile iron weldament

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The microstructure of Austempered Ductile iron (ADI) is unique ausferritic structure consisting of ferrite needles and high carbon enriched austenite. As at one end the transformation to martensite is prevented, on the other hand due care is taken that no pearlitic transformation should take place during its transformation. Even after being researched so heavily and studied so deeply, ADI is unable to replace convincingly any of the material to whom it has superior mechanical and microstructural properties because its weldments were not studied in corrosive environments. The present work deals with the study of ADI, prepared after ductile iron is austenitized at 900°C for 1 hour followed by austempering at 270°C and 370°C for 2 hours to get lower and upper bainite microstructure respectively. For the present study, two corrosive environments were taken i.e. NaCl and HCl at four different concentrations of 5%, 10%, 15% and 20%. The samples were compared for their weight loss and analyzed for mechanism of corrosion. Analysis of corrosion products was done through X Ray diffraction and SEM to analyze the mechanism of corrosion. The results revealed that the microstructure of ADI also plays an important role to avert corrosion at lower concentrations of corrosive environment.

The influence of halide ions on the passivity of NiTi alloys: changes in oxide properties

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Nitinol (NiTi) is an attractive biomaterial because it possesses unique shape memory and superelastic properties and biocompatibility. The presence of aggressive halide ions in the body environment corrodes NiTi by releasing Ni^{2+} ions that act as an allergen. Hence passivation or surface treatment is required to improve the surface properties of nitinol. In the present work, the influence of halide ions on the corrosion and passivity behavior of nitinol was investigated using electrochemical techniques. Potentiodynamic polarization studies were carried out on NiTi at varying concentrations of halide ions to identify the corrosion behavior. NiTi alloys display wide range of passivity under different halide concentration. The results showed that NiTi alloy was primarily susceptible to passivity breakdown, when exposed to a solution containing halide ions and post-corrosion surface characterization confirms the susceptibility to pitting. The potentiostatic growth of passive films in the range 0.10 - 0.40V (SCE) was performed until the current transients attained steady-state conditions. Electrochemical impedance spectroscopy (EIS) and Mott schottky analysis were performed to characterize the passive film nature and to derive dopant type and donor/acceptor concentration. Passive film thickness estimated from capacitance measurements are comparable with the results obtained from *ex-situ* ellipsometry measurements. Also, other kinetic parameters for ionic conduction like, the electric field strength, the diffusivity, flux and the transport number were derived from the electrochemical measurements and the influence of halide ion concentration on these parameters is elucidated.

Study of cadmium equivalent plating

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Ferrous components are highly susceptible to corrosion, wear and tear. Protecting them from all possible deleterious sources like industrial and corrosive atmosphere, solar radiations, friction, humidity, salt laden air, etc assumes significance. Advantages of cadmium plating are well-known. Its ability to confer both barrier as well as sacrificial type of protection together with high corrosion resistance at relatively lower coating thickness and galvanic compatibility, ensures that it is still one of the favored coatings. Irrespective of newer techniques like PVD, CVD, Plasma/Thermal spray, quite a few countries, even today, specify cadmium as the coating of choice. This is particularly observed for threaded assemblies and fasteners and mainly in sectors such as aerospace, nuclear and defense. The fact that cadmium is a potent toxicant and hazard to health and threat to environment cannot be overlooked, though. Cadmium plating operation generates toxic waste which contains not only cadmium but also cyanide, as cadmium-cyanide plating bath is used at large. Strict disposal norms complicate the problem further. It cannot be overlooked that cadmium compounds are also carcinogenic to human beings. Keeping all this in mind, in-depth studies towards finding alternatives to cadmium plating become the need of time. Present study makes an effort to compare cadmium with zinc plating; electroless nickel plating and the dual Zn-Ni alloy coatings. Coated test samples of each of the coating were analyzed for properties which included coating thickness, coating composition, salt spray resistance and electrochemical tests for corrosion rate determination. The experimental techniques included XRF, SEM-EDS, PMI, etc. Main emphasis of this study was on the corrosion protection properties of the coatings. Findings of the study led to conclusion that the Zn-Ni alloy coating (with organic top-coat) as being the closest offering the advantages comparable to cadmium.

Session 3: Nov 15, 2014:[14:00 - 16:00]

Electrochemical characterization of oxide film formed on the carbon steel: effect of cold work

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Flow accelerated corrosion (FAC) of carbon steel piping occurs when the protective oxide film formed, rapidly dissolves in the flowing stream of water or wet steam causing extensive thinning of pipes. FAC occurs mainly at the locations like elbow, reducer/expander and bends and is generally attributed to high flow turbulence at these locations. These regions invariably undergo cold working during fabrication process. The aim of the present work is to examine reasons other than turbulence, like changes in the nature/thickness of the oxide film formed at cold worked regions and if these contribute to increased FAC rate at these locations. In this context, carbon steel (AS106 Gr B) plates were cold worked to different extent by rolling to 5%, 10%, 20% and 30% reduction in thickness. Micro hardness measurements showed an increase in the hardness from 120 HV to 210 HV by 30% cold rolling. Cold worked specimens were oxidized by exposing to a solution of pH 10 (LiOH) in an autoclave at 270°C for 72 h at a pressure above its

saturation pressure. Weight changes before and after the exposure were measured. Oxide films were characterized by Raman Spectroscopy and XRD. The oxide films were also characterized by electrochemical impedance spectroscopy (EIS) followed by Mott–Schottky analysis. EIS were performed from 10 kHz to 10 MHz in the potential range of -0.3 V to 1.0 V in borate buffer solution of pH=9.2. Raman Spectra and XRD confirmed the formation of magnetite film on all the samples. In Mott-Schottky analysis, the capacitance values at each potential were plotted with respect to applied potential and defect densities were determined from slope of this curve. It was observed that the defect density of the oxide film increased from 1.1×10^{22} to 2.07×10^{22} with 30% cold working. This increased defect density may also be contributing to the high FAC rate at locations of elbow, reducer/expander, bents etc.

Oxidation behavior of some high entropy alloys.

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High entropy alloys (HEAs) are multicomponent systems with equiatomic or near equiatomic composition possessing mixing entropy $\geq$ 1.61R. The information and understanding generated on these alloys indicate that these alloys usually possess simple solid solutions, exceptionally high strength and hardness, good thermal stability where as some scattered investigations appear to indicate that these alloys exhibit good oxidation and corrosion resistance too. However, studies on cyclic oxidation behavior of these alloys have not received any attention till date. This investigation aims to examine the oxidation behavior of four HEAs (i) $Al_{23}Cr_{23}Cu_{11}Fe_{21}Ni_{21}$ (HEA-NCo), (ii) $Al_{16}Co_{17}Cr_{20}Cu_{9}Fe_{19}Ni_{19}$ (HEA-LCo), (iii) $Al_{8}Cr_{17}Co_{17}Cu_{8}Fe_{17}Ni_{33}$ (HEA-LAl) and (iv) $Al_{23}Cr_{23}Co_{15}Cu_{8}Fe_{15}Ni_{15}$ (HEA-HAl). The alloys were prepared by vacuum induction melting and characterized in terms of their composition, microstructure and properties like Young's modulus, Poisson's ratio, hardness and compressive strength. Cyclic oxidation behavior of the alloys was studied at 800˚C and 1000˚C for 24h duration. The oxidized samples were then characterized using XRD and SEM-EDX analyses to reveal their nature, morphology and thickness. The microstructure of HEA-NCo consisted of bcc Al-Ni and bcc Cr-Fe-Ni phases. Cobalt addition replaced the Al-Ni phase by bcc Fe-Co phase. The alloy HEA-LAl was found to exhibit a mixture of ordered Al-Ni and disordered Fe-Cr fcc solid solutions while HEA-HAl showed a similar mixture of the phases but as bcc solid solutions. The HEA-LAl indicated the highest oxidation resistance while HEA-NCo showed the lowest oxidation resistance at 1000ºC. The oxidation resistance of HEA-LCo was the highest while that of HEA-NCo was the lowest at 800 ºC. All the selected HEAs were found to show negligible mass changes per unit surface area during cyclic oxidation at 800˚C/24h. At 1000˚C, an initial mass gain followed by continual mass loss was observed; mass loss during oxidation of these alloys was attributed to volatilization of chromium oxide and spalling of the oxide scale during cyclic oxidation.

Corrosion behaviour of zirconium–stainless steel metallic waste form in simulated geological repository environments

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To optimize SS-content in metallic-waste-form, potentio-dynamic polarization studies were conducted on as-cast zirconium (Zr)–stainless steel (SS) alloys (i.e. Zr–5, 8, 12, 16, 20 and 25 wt.% SS) at pH = 1 (aggressive), 5 (mild acidic) and 8 (mild basic) simulating geological repository environments. With the

raise of SS-content in these alloys, intermetallic share in the matrices increases that showed considerable influence on their electrochemical behavior. In addition, variation in electro-chemical response in these Zr–SS alloys was depended upon pH environments: such variation was significant at pH 1 and 5, although, was negligible at pH 8. Electrochemical behavior showed lower pitting potentials, at pH = 5 and 1, for the alloys which had more than 16 wt.% SS and their microstructure analysis by scanning electron microscopy also showed localized attack at micro-cracks and matrix-intermetallic interfaces. On the other hand, Zr–Zr_3(Fe, Cr)/Zr_3(Fe, Ni) interfaces were mainly prone to attack in the Zr–5, 8, 12 and 16 wt.% SS alloys. Zr–8 and 16 wt.% SS holding less Zr_3Fe-type of phases demonstrated better corrosion resistance as compared to Zr–5 and 12 wt.% SS, respectively.Since, corrosion resistance of Zr–8 and 16 wt.% SS alloys are comparable in normal environments (i.e. pH 5 or 8) as well as in aggressive environment (i.e. pH 1), occurrence of additional Laves phases (potential host phases for radio-nuclides) makes the later more attractive for MWF.

Passivity breakdown studies on CarElso 70 SOHIC carbon steel in chloride-containing borate buffer solutions

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70 SOHIC is a special high strength C-Mn steel adapted for pressure equipment. This article describes an attempt to predict and interpret the pitting corrosion of the steel in terms of the point defect model (PDM) in deaerated borate buffer solution at different ph (6, 8, 9, 10) at a constant chloride ion concentration ($[Cl^-]$ = 1M). The genesis of the research is to support the development in Belgium for the safe disposal of processed or un-reprocessed high level nuclear waste (HLNW) in specialized "supercontainers" for periods of over 100,000 to 1,000,000years. Though the real environmental parameters to study the corrosion during the disposal should be pH 13.5 at 100 °C, the present study was necessary to generate data for extrapolating the critical breakdown potential from high to low pH in order to describe the passivity breakdown in actual repository condition. The objectives of the work is to: (i) determine the breakdown potential (V_c) as functions of pH, chloride concentration and potential sweep rate (υ); (ii) analyze the relationship between V_c and $[Cl^-]$, pH and υ in terms of PDM to extract parameter values for passivity breakdown and (iii) assess the ability of PDM to account for passivity breakdown. The near normal distribution in breakdown potential is in satisfactory agreement with the analytical prediction of the breakdown potential distribution obtained from PDM. The linear dependence of breakdown potential on the square root of potential scan rate and polarizability dependence of the barrier layer/solution interface upon potential and pH are 0.83 and 0.01 respectively, as predicted by the PDM, yields an estimate of the critical areal concentration of condensed vacancies at the metal/film interface ($\xi = 4.9\times10^{14}$ cm^{-2}) that leads to passivity breakdown. These provide convincing evidence for the validity of PDM for modeling passivity breakdown on 70 SOHIC carbon steel.

Improvement in pitting corrosion resistance of duplex stainless steels in 0.1N NaCl solution at 80°C

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Development of duplex stainless steels began due to the limitations of corrosion resistance of austenitic stainless steel grades like 304/316 particularly to stress corrosion cracking in chloride atmosphere with high chloride content. The corrosion resistance property of duplex stainless steels is described in terms of the PREN number. For 2205 grade, the PREN is generally maintained at over 35 to get the desired corrosion properties along with mechanical strength. Our process for manufacture uses the UHPF(EAF) Convertor VOD triplex process route for stainless steel making. This process ensures a product with consistent heat chemistry and uniform properties for the steel. In order to characterize the features of our duplex stainless steel, the effect of sigma phase on corrosion properties was studied. The results obtained through microscopic measurement and estimation of phases from constitution diagram are presented and discussed. To understand the corrosion resistance quality, the critical pitting potential was also evaluated by Potentiodynamic method in a 0.1N NaCl solution at 80°C. It was observed, that the critical pitting potential was affected by nonmetallic inclusions and the sulphur level in the steel even though the PREN requirements were met. Based on EDS analysis of these inclusions, specific actions were implemented to improve the steel cleanliness and thereafter achieve the desired corrosion characteristics. In subsequent heats made, the pitting potential was observed to be 350 – 450 mV against the specified minimum value of 250 mV. This paper discusses the test results of pitting corrosion and the improvement actions initiated to control inclusions and improve the pitting potential of our duplex stainless steel grade 2205.

Electrochemical corrosion behavior of electroless nickel plating

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Electroless nickel plating can be applied on nonmetals like ceramic hence useful for electronic applications. Resistors used in electronic circuits are normally prepared using carbon or metal films formed by dry processes like sputtering or vacuum deposition on non conducting ceramic substrates. The diameter and length of the substrate varies depending on the resistance and wattage required. In order to reduce the temperature coefficient of such resistors, usually a non metal is incorporated in the film. In the present study, electroless nickel-phosphorous alloy was coated on ceramic resistors. Because of co-deposition of 6-14% phosphorous in case of electroless nickel phosphorous alloy plating, the temperature coefficient values of the resistor films become low. The different parameters affecting electroless plating such as HF etching pretreatment, composition of chemical solution, bath temperature and post heat treatment temperature on the temperature coefficients of Ni-P film resistors are discussed. Scanning electron microscopy (SEM) attached with energy dispersive X-Ray analysis (EDS) was used for surface morphology & elemental analysis. With the help of X-Ray diffraction crystalline phases of electroless plated Ni-P on ceramic substrate were examined. A conventional three electrode cell was used with graphite as counter, saturated calomel electrode (SCE) was used as reference, electroless Ni-P plated ceramic rod as a working electrode and electrolyte in 3.5wt.% NaCl solution. To understand the corrosion behavior, electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization tests were performed on electroless Ni-P plated ceramic rod with three different phosphorus content, such as low P, medium P & high P. The results obtained will be discussed in detail.

Invited talks

Session 1: Nov 12, 2014: [16.00 - 18.00]

Discrete cohesive zone modeling to simulate and predict fracture of 2d triaxially braided carbon fiber composites

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Results from an experimental program to investigate the propagation of damage and energy dissipation in 2D triaxially braided carbon fiber textile composites (2DTBC) under static conditions are reported. A methodology is presented in which classical concepts from fracture mechanics are generalized to address damage growth in an orthotropic and heterogeneous structural material. Along with results from the experimental program, a novel numerical technique that employs ideas from cohesive zone modeling, and implemented through the use of finite-element analysis, is also presented. The inputs that are required for the discrete cohesive zone model (DCZM) are identified. Compact tension specimen fracture tests and double notched tension tests were carried out to measure the fracture energy (GIc), and the maximum cohesive strength (rc), of the 2DTBC. The DCZM modeling strategy was independently verified by conducting single edge notched three-point bend tests using a modified three-point bend test fixture. The experimental and numerical analyses were carried out for two different types of 2DTBC made from the same textile architecture but infused with two different resin systems to validate the proposed methodology.

Contributory papers

Session 1: Nov 12, 2014: [16.00 - 18.00]

Study on mechanical properties evaluation of coir-glass hybrid epoxycomposites

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Coir, the so-called coconut fiber from south or eastern India is one of the most common agro-fibers having high tensile modulus and low elongation at break. The low density of this fiber is taken in to consideration, and then its specific stiffness and strength are comparable to the respective quantities of glass fibers. The fiber has a high aspect ratio, high strength to weight ratio, low in energy conversion and has good insulation properties. Preparation of coir-glass hybrid epoxy composite with orientation of fiber at different stacking sequence has been performed. Study of mechanical properties of coir-glass hybrid epoxy composite for different stacking sequence has been investigated through SEM analysis for structure,

property co-relationship. The coir fiber composites can be very cost-effective material especially for building & construction industry, packing, automobile & railway coach interiors and storage devices.

Development of controlled size polymer nano-cap via coffee ring effect

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A coffee droplet on a table is ostensibly a simple physical system, but, it inspired countless studies to understand the preferential accumulation of solute at the contact line due to irregularities of the table. The manifestation of this phenomenon is called ring stain or coffee ring effect. The phenomenon of deposition of solute at the contact line is inevitable in ink jet printing technique, which has been conceived as an undesirable effect. Since, this technique received considerable attention for real time patterning to develop miniaturized and advanced functional electronics, considerable effort has been expended to eliminate undesirable effect of stain. In contrast, we have exploited the coffee ring effect to demonstrate the spontaneous assembly of controlled size polymer nano cap over the gold nanoparticles. In this context, we have exploited polyvinyl alcohol (PVA) for the development of nano-cap by using ink jet printing techniques. Depicted is the schematic representation of PVA nano- cap formation on PVA matrix on the same polymer substrate to provide enhanced affinity between cap and polymer substrate. As shown, the process was performed by printing an aqueous solution gold nanoparticles on PVA substrate from cartridge A, followed by drying under UV light for 3 min. Subsequently, cartridge B prints on the top of the previous printed droplets of the Gold nanoparticles under the same printing parameters at room temperature, followed by similar drying operation. Due to the versatility and flexibility of the technique, our work has demonstrated an excellent and facile alternative route for encapsulation and the fabrication of unique biocompatible nanocomposites for biomedical application.

Carbon nanotube/ultra high molecular weight polyethylene composite for hip joint – influence of CNT morphology on wear behaviour

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Ultra high molecular weight polyethylene has been extensively used as one of the bearing surfaces in total joint replacements due to its excellent energy absorption, high impact strength and other constructive mechanical properties. The wear debris generated between metal counter face during articulation motion invites macrophage and giant cell activation, which leads osteolysis and makes aseptic loosening of implant. Therefore a drive always exists to enhance the wear resistance of UHMWPE lining material without increasing the coefficient of friction. In this framework carbon fiber reinforcement is ultimate choice to reduce the risk of osteolysis. In the present work, different morphological multiwall carbon nanotubes (CNTs) reinforced UHMWPE composite are synthesized by compression molding followed by

curing at 160° C for 60 minutes to improve the tribological and mechanical properties. Ball-on-disc tribometer and nano indentation test were performed to assess the tribological and mechanical property of the composite surface. The study showed that addition of different morphological MWCNTs up to 0.1 wt % lowered the specific wear rate and co efficient of friction significantly as well increased the hardness and elastic modulus slightly compared to UHMWPE due to CNTs excellent in plane strength and load transfer between the polymer matrix and reinforcement. Microstructural analysis of worn surfaces provides insight into the wear mechanism dominating the tribological behavior of CNT reinforced UHMWPE composite. Mechanical and tribological behavior of the composite is found to show a significant dependence on the morphology of CNT, which has been investigated.

Novel approach towards the processing and feasible preparation of stable electrospun chitosan nanofibers

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Chitosan (CTS) is an N-deacetylated product of chitin, the second-most abundant natural polysaccharide next to cellulose. It displays beneficial therapeutic properties such as biocompatibility, biodegradability, non-toxicity, antimicrobial activity and wound healing. Electrospinning of Chitosan adds interesting characteristics such as fine diameters (nanometer range), large surface area per unit mass and high porosity, which assist in drug release and adsorption, proving to be potential candidates for wound-healing applications. The structure of nanofibers is characterized by Field Emission Scanning Electron Microscopy (FESEM), Fourier Transformed Infrared Spectroscopy (FT-IR) and X-Ray Diffraction (XRD). The electrospinning of Chitosan nanofibers is attributed to physical parameters like molecular weight, viscosity, concentration, surface tension and conductivity. The complexity and intricacy of Chitosan solution to be drawn into nanofibers, restricts the convenience of its widespread use. The present study demonstrates a facile method of obtaining electrospun chitosan fibers by enhancing the processibility of Chitosan, using a plasticizing effect of Poly Vinyl Alcohol (PVA). Formation of small micro size droplets, *sans* PVA, was observed by variation of solvent concentration (1-90%acetic acid) , viscosity(250-400 cP) , chitosan concentration (1-10wt%), Molecular Weight (60,000-3,00,000). Successful attempts have been made on regulating and calibrating chitosan nanofibers by varying the concentration of PVA in Chitosan solution (3:1, 5:1, 1:1), electrospinning with optimum parameters and thereafter, treating with NaOH solution to remove PVA. These nanofibers possess a stable morphology and posses potential applications in effluent treatment as adsorbent for toxic heavy metal ions.

Effect of solvents and lithium salt on P(VDF-HFP) based composite polymer electrolytes

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The poly[(vinylidene fluoride)-co-hexafluoropropylene] (P(VdF-HFP)) based gel polymer electrolytes having different combinations of lithium salt ($LiClO_4$) and plasticizer (PC+DEC) contents were synthesized by solution casting technique and the effect of the such combinations on the electrolyte characteristics was investigated. The electrolytes were characterized using FESEM for morphological analysis, X-ray diffraction

(XRD) for structure determination, Fourier Transform Infrared Spectroscopy (FTIR) to reveal the formation of polymer-salt complexes and thermal analysis (TGA) to estimate the residual contents. Electrical conductivity of the electrolyte samples was studied using ac impedance analysis technique. The maximum conductivity (10^{-5} Scm^{-1}) at room temperature (30˚C) is resulted with the electrolyte composition of 0.6P(VDF-HFP)-0.15(PC+DEC)-0.1$LiClO_4$ (wt%) and its temperature dependent conductivity data follow Arrhenius behavior.

Improving mechanical and swelling properties of biodegradable composites via chemical route

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Cellulose in different forms is used to reinforce polyvinyl alcohol (PVA) matrix. The hydrogen bond between hydroxyl groups of PVA and cellulose enhance interfacial adhesion and hence resulting in a good composite. We demonstrate incorporation of covalent bond between PVA and cellulose that can further improve the mechanical properties of the composites. Click chemistry route to covalently link cellulose and polyvinyl alcohol (PVA) is demonstrated in a three step process. First, polyvinyl acetate azide (PVAA), was synthesized via chloroacetylation and subsequent azidation of poly (vinyl alcohol). In the second step, propiolic ester of cellulose was synthesized via esterification of cellulose by propyl acid chloride. Azide and alkyne derivatives on PVA and cellulose respectively were then coupled using click chemistry, resulting in covalent linkage between cellulose and PVA. Successful synthesis of the azide and alkyne derivative was established by FT-IR, mass spectroscopy and ^{13}C NMR techniques. Covalent crosslinking of cellulose and PVA by alkyne and azide derivatives respectively was confirmed using FTIR. Click chemistry reaction between azide and alkyne was also confirmed using FTIR.

Review of red mud derived geo-polymer (a green construction material)

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Red mud is a solid waste residue of the digestion of bauxite ores with caustic soda for Alumina production. Disposal of large quantities of red mud generated at the Aluminum plants all over the world possess an increasing problem of storage, land cost & availability and pollution. Because of the complex physio-chemical properties like relatively complex composition, coupled with its fine particle size distribution, high pH, poor settling properties and some toxic rare metal associations make it a very challenging task for the designers to find out the economical utilization and safe disposal of red mud.In this study the geopolymerization process for obtaining construction materials based on red mud was used. Red mud can be used as a source of alumina, silica and alkalis for geopolymerization. Geopolymers are resistant to fire, corrosion and organic solvents. They are nontoxic, “green” materials since their production saves energy and does not produce CO_2 emission, unlike traditional Portland cement. This paper reviews the

fundamental chemical and structural characteristics of geopolymers derived from red mud, fly ash and slag in terms of the effects of raw material selection on the properties of geopolymer composites. The factors affecting goepolymerization reaction, mechanical properties of geopolymers and the problems related to its practical applications have also been discussed in this paper.Geopolymers can be a cementitious material to replace Portland cement in infrastructure applications, such as roadway construction, building materials, as well as waste containment and capsulation, which bring economic and environmental benefits.

Aligned multiwalled carbon nanotubes/chitosan electrospun nanofibrous scaffold for neural tissue regeneration

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In recent years, the utilization of naturally occuring polymer and nanomaterial composites in the field of neural tissue engineering has brought revolutionary change in the approach to treat nerve-related abnormalities and diseases. MWCNT/polymer scaffolds are favourable option as substrates for neuronal growth due to the electrical conductivity offered by the reinforcement phase. Carbon nanotubes provide interface for improved electrical conduction in cultured neural cell networks, while chitosan provides biocompatibility. So, in this study, we fabricated novel aligned MWCNT/chitosan nanofibrous scaffold through electrospinning technique. MWCNT/chitosan nanofibrous scaffold consists of singular MWCNTs wrapped inside chitosan polymer. CNTs as core to the fibers, allow the dimension of the fiber to be in couple of hundreds nanometer range and alignment of fibres provides increased conductivity and better nerve cells organization. Field emission-scanning electron microscopy and transmission electron microscopy were used to characterize aligned and randomly oriented electrospun fibers. Mechanical properties of scaffold is also evaluated using small scale tensile testing machine. HT22 mice hippocampal neuronal cell line was used to perform in-vitro biocompatibility and cytotoxicity tests of scaffolds. These studies give an insight on the effect of CNT reinforcement, as well as alignment on the mechanical, electrical and biological behaviour of the scaffold.

Creating mold for fabrication of microfluidic devices for biological applications using Polydimethyl Siloxane polymer by FARM technique

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Microfluidics, as such deals with science of fluid flow at micro-scale (micrometer size) in the channels with dimensions ranging from single micrometers to tens or hundreds of microns. These, so called microfluidic-channels are used for fabricating various devices such as on-chip electrophoresis, smart biosensors, gradient generation devices, micro and nano emulsion generation devices, micrototal analysis system, micro chemical factories, cell-loading devices etc. The most commonly used polymer for fabricating these microfluidic systems is PDMS (Polydimethyl Siloxane), which is a mineral organic polymer of Siloxane

family. These Siloxane bonds allow a flexible polymer chain with a high level of viscoelasticity so that after cross-linking, PDMS becomes a hydrophobic elastomer.FARM (Foil Assisted Replica Molding) technique, as such involves devising master patterns for soft-lithography of microfluidic devices by using patterned Al foil with the help of XY-plotter by importing the design file (.plt extension) from COREL DRAW software. This master is then used to mold against a PDMS elastomer & cross-linking agent mixture in the ratio of 10:1 thereafter keeping the degassed sample in a hot-air oven for 2 hrs (min) to obtain a replica of micro-channels. Now, to allow the injection of fluids for future experiments, the inputs and outputs of the micro-fluidic device are drilled with a needle or a punch of the size of future outer tubes. Finally, plasma oxidation at the surface of PDMS device is done to covalently bond it on oxidized glass surface by creation of a Si-O-Si bond thereby providing a base to the micro-fluidic chip.Hence, PDMS finds wide-scale application in microfluidics as it is transparent at optical frequencies thereby, facilitating observation of contents of micro-channels visually or under the microscope as well as has low auto-fluorescence and is bio-compatible. Also, its gas-permeability allows cell-culture by controlling the amount of gas through fill channels.

FAILURE ANALYSIS

Invited talks

Session 1: Nov 14, 2014: [08:30 – 10:30]

Why does the material behaves the way it does?

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Despite the great strides that have been made in technology, failures continue to occur, often accompanied by great human and economic loss. The sources of such failures are attributed to deficiency in design, material selection, and or in following proper processing route, error in assembly, improper service condition etc. Failure analyst uses various analytical tools to analyse, reaches correct fact – based conclusions on the cause of metal failure. Numerous analytical methods are being used to establish the cause of failure. Almost every mechanism of failure left behind specific signatures, and hence analyst has to gather those features, correlate with event and establish his hypothesis. Once arrive s at conclusion, a failure analyst often suggests design and or material/process improvement to prevent recurrence of failure any more.This talk highlights the comprehensive coverage of both the "HOW" and "WHY" of metal failure with specific emphases for the failures in aerospace components. First part of the presentation covers portrait of a crack which includes close-up on crack, concentrator converting into crack growth, moral principles, talkativeness of a crack and finally its stoppage. The second part covers in general metal failure (condition & storage), practices followed by failure analyst, type of failure with emphasis on environmental effect on failure. Few failure case studies reconstructing the sequences of cracking have also been discussed.

Contributory papers

Session 1:Nov 14, 2014: [08:30 – 10:30]

Failure analysis and design modification in plunger used in CNG cylinder valve assembly of passenger vehicles

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Compressed Natural Gas (CNG) is one of the potential fuels in modern automobile industry because of its low emission characteristics. Plunger used in CNG Cylinder valve found broken in the field vehicles lead to problems in CNG-fitted cars. The fractured surface of the plunger was analyzed using Scanning Electron Microscopy. Computational fluid dynamics was performed on the entire valve to understand the temperature surrounding the failed plunger part. It was found that sudden temperature drop at the solenoid valve during filling of CNG has caused the plunger to undergo brittle failure. Also, decrease in

fracture toughness value of the ferritic steel plunger with decrease in temperature was confirmed experimentally. Modified design of the plunger was further proposed and various endurance trials were conducted to ensure the long term use of the new plunger in the same environment.

Metallurgical investigation of air intake casing of Gas Turbine Starter Unit (GTSU) engine of a military aircraft

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One of the struts of the part “Air Intake Casing” meant for GTSU engine of a military aircraft had cracked during the qualification test of the engine. The part was newly fitted into the engine before the vibration test and the crack in the part had developed just within 1 hour and 30 minutes of the test. The casing was made of Al-Cu-Ni cast alloy of specification MSRR 8009, was in solutionized and aged condition and was manufactured by Foundry and Forge division of HAL.Radiography inspection did not show any significant defects in the casing. The chemical composition and heat treatment condition of the casing met the specified requirements. However, micro examination revealed presence of numerous microshrinkage porosities in the casing. The casing was free from other casting defects like hot tears. The crack in the forwarded part was opened-up for fractographic studies. Scanning Electron Microscopy revealed presence of fatigue striations on the fracture surfaces of the casing. The orientations of the striations indicated that fatigue failure had originated at multiple locations from the surface of the strut of the casing. SEM analysis also revealed presence of microshrinkage porosities at the fatigue failure origins.It was concluded that the “Air Intake Casing” has failed by fatigue that had originated from microshrinkage porosities present on the strut surface of the casing. Al-Cu cast alloys have poor castability due to long freezing range that results in casting defects like microshrinkage porosities and hot tears. Therefore, laboratory had recommended changing the material used for the casing to Al-Si premium grade cast alloys or Al-Cu wrought alloys subject to fulfilling the design requirements.

Failure analysis of waste heat boiler during service

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Waste Heat Boiler is critical high pressure equipment in which hot synthesis gas is flowing on tube side and steam is generating on shell side. In one of the waste heat boiler (vertical) with U-tube bundle, catastrophic failure of 120 mm thick circumferential seam between tube sheet (material of construction:SA182 F 21) and hemispherical head (material of construction:10CrMo910) occurred during operation within the short duration . A circumferential crack of 1650 mm length was observed on the outer surface of the weld seam between tube sheet and hemispherical head on channel side. Circumferential weld seam having mentioned failure was on the process gas outlet side. Detailed analysis such as fractography, chemical analysis, material characterization, metallography examination etc. was performed to determine the root cause of failure.This paper covers the detailed investigation carried out to find the root cause of failure of waste heat boiler.

Failure Analysis of an intermediate shaft

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Structural integrity evaluation is prerequisite for any engine components prior to its actual usage. To validate engine components like fan drum, compressor drum, etc. an intermediate shaft was being tested in no load condition to 25000 rpm level under cyclic spin test. During testing, the shaft failed prematurely from the fillet region at a lower level of rpm. Other segments of the assembly namely journal bearing, high speed pinion, etc. were also found to be damaged. The shaft was manufactured from martensitic stainless steel grade [AISI 420] material. The finished component was supplied in hardened and tempered condition.The main objective of the work was to analyze the damage mechanisms that promoted the failure. Fractographic analysis of the components was conducted using stereo zoom microscope, followed by scanning electron microscope. Microstructure analysis and micro hardness evaluation were also carried out using optical microscope and micro hardness tester respectively. Fractographic analysis of all the failed pieces revealed features as chevron marks, cleavage steps and dimples on the fractured surface. These observations suggested that shaft failed due to overload with crack origin at the interface of the bearing surface and the geared coupling [near fillet region]. Nonmetallic inclusions were observed at the site of crack origin. From the evidences, it was corroborated that the shaft failure was due to usage of inferior material grade.Suitable standard samples both Low Cycle Fatigue and tensile were also extracted and prepared from the failed intermediate shaft. Effect of the presence of nonmetallic inclusions on the mechanical properties has been studied and results presented. Fractographic studies on the tested samples have also been carried out. The mechanical properties of the extracted samples were found to be inferior as compared to the typical values for the alloy.

Failure of the cooling turbine fan of an Aero-engine: A case study

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Failure of the cooling turbine fan (ventilator) of an aero-engine has been reported approximately after 50h of service life following the engine overhaul. Subsequent visual inspections revealed that the two blades at diametrically opposite side of the ventilator are broken at the tip and at one of the broken blade sides shows severe “rub” marks (visible from the back side of the ventilator).. Through detailed investigation involving fracture surface and microstructural examination, the contributing factors for such failure have been established. Failure of the ventilator blades is primarily attributed to the combined effect of pitting corrosion and stress corrosion cracking. Formation of corrosion pits acted as stress raisers and the cyclic loading during service led to the fatigue crack initiation process. After the crack has had grown to a significantly large size, the load bearing capability of the individual blade has been reduced greatly, so that the remaining part gets fractured by overload mode creating sharp “shear-lip” regions. Failure of one side of the ventilator blade has caused severe imbalance in the system. As a result, the ventilator gets tilted on the opposite side of the blade leading to severe “rubbing” of the component. In summary, the most probable sequence of critical events leading to the failure has been highlighted.

Failure analysis and prevention action of high-chromium iron hot strip mill work rolls

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The investigation of failed samples of high chromium (HiCr) iron work roll spall in finishing stand 3 (top roll) in a hot strip mill. The work roll spall was analysed by means of visual examination, chemical analysis, hardness measurement and optical & scanning electron microscopy (SEM). The spalling can be possible due to the applied mill load and the localized flattening of the rolls at their contact point. The maximum resultant shear stress (commonly referred to as "Hertzian Stress") is located at a short distance below the roll surface. Theother possible cause of spallingmay be of poor material quality whichcan be identified by the presence of a concentric fatigue pattern (fish eye) on the fracture face.The visual examination shows fracture is instantaneous and brittle in nature and the chemical analysis of the sample shows chromium on thelower sidein range of the specified chromium percentage (3-7%). The measured hardness in HSS work-roll sample is in the range of 625.2-650.3 BHN with average hardness of 632.8 BHN (59 HRC & 80 Shore C), which is higher than the specified (72 - 76) hardness. The microstructure observation of nital-etched spalling roll surface revealed the presence of secondary carbides of Cr, Mo & V distributed in the matrix of martensite. The SEM fractrograph of thesample shows that the cracks tend to propagate along the carbide networks, and also it is a brittle failure. If the test certificate had shown the actual composition of work roll, would have been rejected and the roll failure would have been prevented. The roll spalling resulting from a sub-surface defect can be prevented by ultrasonic inspection techniques using a straight beam transducer on every roll after completion of the grinding operation.

Session 2: Nov 15, 2014: [11.00 - 13.00]

Investigation of stainless steel lance failure at lime calcination plant

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LCP is designed to convert Limestone ($CaCO_3$) into lime (CaO) by heating at a temperature at around 1000°C using a fuel. JSW Steel, Dolvi is using a technology where pulverized coal (PC) is used as a fuel. PC is injected through Stainless Steel lance (SS310) into the lime kiln shaft where it is burnt and tip of the lance reaches to temperature of around 950-1050°C. Even though SS 310 is recommended for its high temperature corrosion resistance and structural stability at these temperatures, lance pipes are failing prematurely within 6-8 months, against their expected life of 2-3 years.Present work discusses the premature failure of lance pipe and detailed investigation of lance failure. The investigation includes visual observation & microstructures using optical and scanning electron microscope. Visual observation shows that failed location is at a distance from the tip and thinning of the lance pipe is clearly visible. Outer layer of the lance shows presence of non continuous porous oxide layer. Microstructure and SEM analysis indicates significant presence of Sulphur at the failed location along with concentration gradient of Nickel & Chromium. Various characteristics features of Sulphidation corrosion are compared with actual observation on the failed sample and various possible alternatives are discussed to counter this problem.

Failure analysis of a charge roll of reheating furnace of a bar rolling mill

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Failure of a charge roll of a reheating furnace has been discussed. Nine in-furnace charge rolls are used to guide cold steel billets into the furnace at a bar rolling mill of a steel plant. One such roll failed in service. On inspecting the site of failure it was noted that the roll used to be exposed to a furnace atmosphere where the temperature reaches to about 700°C and was run by motor connected to it by a shaft. Also, to counter the effect of heat, industrial cooling water (ICW) was continuously circulated within the roll. Visual observation of the failed roll revealed bulging at the middle part of the roll surface and cracking at the edge. The failure was investigated using metallographic techniques. Microstructural investigation followed by characterization using SEM-EDS, hardness testing etc. was carried out. It was confirmed that due to overheating for prolonged period evolution of microstructure in the form of pearlite phase disintegration had occurred which resulted in a time dependant deformation of roll at high temperature. Some deposits were found on the inner side of the failed roll and were present in abundant quantity. Analysis of the deposits revealed that these were oxides of calcium and magnesium, indicating that hard ICW was the source of these precipitates. The deposits led to ineffective heat transfer between the roll and ICW which resulted in overheating of the roll. As such, the choice of material composition of the roll did not play any role in facilitating the failure. Based on the observations, recommendations such as acid-cleaning during maintenance shut-downs and routine quality-check of ICW were made to incorporate in maintenance practices for the rolls.

Failure analysis of AISI 309S lance tube used for coal injection in a blast furnace

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The failure analysis of a coal injection lance tube in a blast furnace has been presented. Most of the modern blast furnaces are having facilities for injecting pulverized coal as an auxiliary fuel. The coal is injected in front of the tuyeres through lance tubes by nitrogen as a carrier gas and the injected coal is burnt in contact with the hot blast (blast temperature ≈ 1225°C) enriched with oxygen and humidity. The injection lances are failing pre-maturely within two months of their service. The material of the lance tube is found to be AISI 309S grade of austenitic stainless steel. Visual observation shows multiple branched out cracks on the tube wall. Fractography of the crack surface under scanning electron microscope (SEM) shows cleavages indicating brittle fracture. Microstructural examination reveals branched out transgranular cracks initiating from the outer surface which is in contact with the hot blast. Energy dispersive spectroscopy (EDS) on the cracks near the outer surface shows presence of corrosive elements like chloride and sulphur in the corrosion product; potential source of chloride and sulphur is the raw materials like coal and coke used in the blast furnace. Sulphidation is also observed at the inner surface. Analyses of the failure, particularly, multi-branched transgranular cracks of austenitic stainless steel suggest stress corrosion cracking in chloride environment; hot blast conditions like temperature, humidity

and oxygen enrichment aggravate the cracking. A suitable material was recommended for the application after the root cause analysis.

Metallurgical investigation of failed canned motor pump impeller

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Nickel aluminium Bronze (NAB) is known for its excellent corrosion resistance in seawater, good mechanical strength and better erosion-corrosion resistance; and has been most extensively used for impellers, propellers and shafting in circulating seawater systems. Premature failure of an in-service NAB made canned motor pump impeller was reported for causative analysis for which NMRL carried out metallurgical investigation. Studied NAB impeller was completely corroded with widespread perforations along with thinned edges and green colour corrosion deposits. Chemical composition of the impeller was found to be similar to NAB but the manganese content was lower than specified. Manganese addition helps strengthening the alloy thereby preventing mechanical damage due to erosion within the permissible limits of impingement velocities. Corrosion behaviour of these alloys is strongly dependant on the formation of a protective film of copper and aluminium oxides on the underlying metal. However, this protective oxide layer can be disrupted at high impingement velocities due to suspended fine sand particles present in seawater; which could cause extensive localized pitting. NAB alloy can withstand flow velocities from 4.3 to 5.0 m/sec with intermittent maximum of 10 m/sec and peripheral velocity of maximum 23 m/sec. As per pump design specification received, the peripheral impingement velocity of impeller was given as 27 m/sec; slightly higher than safe limit of 23 m/sec for NAB. This in combination with lower manganese content could be the probable reason for cavitation erosion and premature failure of the impeller. It was also found that NAB pump was coupled with graphite bushing and titanium base pipings without adequate insulations, which may have resulted into severe galvanic corrosion of the pump impeller made of less noble NAB as compared to both graphite and titanium which are good conductors.

Failure analysis of overhead crane bolts

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One 20MT overhead crane failed due to bolts failure in foundry shop. A portion of bolts 20 mm diameter was found broken. The shop was having crane that operates on rails supported by columns and beam. Visual inspection of the entire bolt predicts failure is during the course of operation. All the analysis like hardness, metallography, and visual inspection supports that failure is fatigue induced by the overhead movement of the crane .The crack was developed and then it was propagated gradually under repeated stress .The nature of crack shows that bolts failed under high tension. The fracture was absolutely ductile. The bolt should be tightened properly and inspected periodically to avoid such type of failure.

Failure of hot bundle tubes used in tunnel furnace of a thin slab caster

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Failure investigation of hot bundle tubes used in a tunnel furnace at a thin slab caster of an Integrated Steel Plant is presented. Tunnel furnace which is placed between the slab caster and the rolling mill heats the slab for homogenization before rolling. The combustion air for the tunnel furnace is preheated through the heat exchanging process. The hot bundle tubes carrying the combustion air at ambient temperature comes in contact with the hot waste gas at 900°C during the heat exchanging process. Cracking as well as thinning of pipes were observed. The composition of pipes was closer to UNS S 43035 grade which is a Standard Specification for Seamless and Welded Ferritic Stainless Steel Feed water Heater Tubes. Detailed characterization using chemical analysis, optical and scanning electron microscopy, EDS analysis was carried out. Microstructural examination showed severe oxidation and grain boundary corrosion at the pipe surface. Thick layer of scale containing sulphur was detected on the outer surface. The source of sulphur was detected to be the waste gas. Recommendation was given after root cause analysis to avert any similar failure in future.

Failure analysis of axles of seal valves in blast furnace and their condition monitoring through non-destructive testing

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In-service failures of components in blast furnaces result in huge losses due to downtime for maintenance shutdowns and component damages in addition to inducing operational anomalies. It is thus imperative to prevent any in-service failures. The upper and lower seal valves together form a key part in the top charging system of blast furnaces. The axles in these valves are an integral part in the sealing mechanism of the valve flaps. In the recent past there have been several accounts of failure of these axles in service. It is in this view that failures of these axles were studied to arrive at their root cause. Failure analysis revealed that the failures occurred in the fatigue mode. In addition, metallographic examination of the failed axles revealed material anomalies which may have accelerated the fatigue crack propagation. The analysis proved to be instrumental in taking necessary measures for implementation of suitable NDT technique for condition monitoring of the axles which ultimately led to prevention of further in service failures.

Failure analysis of pistons due to thermal fatigue: A review

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Pistons operate in an environment where both repeated heating and cooling take place during each operational cycle. Such loading condition is responsible for thermal fatigue phenomena on pistons. Fatigue due to mechanical loading is also present during the operation of engine. Thermal fatigue along with mechanical fatigue synergize well for the deterioration of mechanical properties of the material. Hence, an understanding of this phenomenon is very important with respect to automobile industry**.** In this article, case studies are presented to explore the failure zones and the reasons behind their occurrence. Metallurgical aspects of thermal fatigue phenomena at the micro-structural level for piston alloy 319 have also been discussed. Furthermore, the extent of deterioration in material properties due to in-phase and out-of-phase loading has been explored. The areas of future research to improve the quality of pistons have been highlighted towards the end.

Qualifying criteria analysis in rails due to residual stresses & its measurement using strain-gauge method

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In recent years, analysis of residual stress in rails is of prominent importance due to several modern railway advancements i.e. higher axle load, increased traffic density and faster speed of trains.All modern rails are hot-rolled products having cross-sectional profile approximating an I-beam having asymmetric distribution about the horizontal axis: a broad head to resist wear so as to give a good ride, an interconnecting web and a flat- bottomed foot to suit the fixing system (fishplates). These rails develop residual stresses due to uneven cooling after hot-rolling and also during finishing operations, such as roller-straightening/levelling.Development of these stresses on the surface and inside the rolled products (such as rails) has serious implications in fatigue damage in service applications. Residual stress significantly influences the structural and dimensional stability as well as life span of different rolled products like rails. These stresses when combined with external static and dynamic loads may cause catastrophic premature failure of rails, leading to high casualty and loss of property.Several methods are employed for measurement of residual stresses in rails: the most easy and accurate being “Transverse Template Cutting Method” which involves use of a sensing element to measure surface strain, known as Strain-Gauge.In this method, a sample of one-meter length is taken from the rail and a slice of 50-60 mm thick is cut in the center of the sample. Stresses in the slice are measured before and after cutting. The difference of two measurements gives residual stress in the sample.The stress σ is calculated as given below:$\sigma=E\varepsilon$ where, $E=2.06\times10\hat{}5$ N/mm^2ε= Measured value in με, (micro-strain) .The strain is measured by using strain balancing method (principle of Wheatstone bridge) and digital strain indicator which is considered as reference (zero strain). The experimentally obtained residual Stress values in rails is now validated using software analysis by first obtaining the exact plot of cross-sectional rail profile in CATIA and then importing the plot in ANSYS to get the stress distribution in rail profile and hence determining the value of maximum residual stress (which shouldn’t exceed 14% UTS value of rail) and position where it occurs thereby estimating the region of crack-initiation & its propagation in rails.

Failure analysis of grate bars at 6 MTPa iron ore pelletizing plant at tata steel india

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Grate bars used in 6MTPA capacity pellet plant at Tata Steel exhibited severe damage in the form of deep directional surface cracks and bending. Damaged grate bars were causing pellet leakage and gas channeling, leading to improper pellet firing and frequent machine breakdowns. Fuels used in pellet firing found to comprise different corroding elements like sulfates and chlorides that can potentially result in hot corrosion of chromium rich grate bars. The objective of the present study is to find the root cause and mechanism of failure and provide potential solutions. Accordingly, laboratory corrosion simulation tests were carried out on fresh grate bar coupons at 850°C using chemicals viz., (i) Na_2SO_4 (ii) NH_4Cl (iii) chloride rich water associated with coal tar. The results were compared with sample from (i) corrosion simulation test with no chemical exposure (ii) failed grate bars from plant. Different characterization techniques used work includes XRF, AAS, SEM-EDS and X-ray mapping.Present study showed that NH_4Cl and coal tar water caused severe damage to the samples by forming weakly adherent porous granular scale on surface while Na_2SO_4 caused mild uniform corrosion. No damage was observed on sample tested without any chemical exposure. Microstructures of samples exposed to NH_4Cl, coal tar water were found to be similar to that of damaged grate bars from the plant which confirms that chloride assisted hot corrosion is the primary reason for grate bar failure. Sp. grate bar consumption reduced from 120 to 40 g/ton of pellets by reducing coal tar water content.

Session 3: Nov 15, 2014: [14.00 - 16.00]

Creep behavior of commercial P91B steel

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This work is focused on testing and evaluation of creep properties of P91B base material and welded joints as well. Creep tests are performed at 600°*C*, 625°*C* and 650°*C* for different loading conditions. The obtained creep data shows that creep is associated with the high and distinct values of creep damage tolerance factors in the low and high stress regimes indicating dominance of microstructural degradation in the steel. At higher stress and temperature the failure is due to formation of extensive necking, where as in case of lower stress, it is due to cavity along with necking within the gauge. A decrease in creep ductility in terms of % elongation to rupture and % reduction in area with increasing creep exposure is observed in welded joint than that of base metal. The creep strength of welded joints is lower than that of the base metal due to Type-IV failure in the region of ICHAZ. It is revealed from an experimental examination such as microstructural analysis and density measurement that the creep damage is due to the formation of creep voids. Also it is found that the increase in cross sectional area within the gauge length of the interrupted creep test of base specimens. In the microstructural studies, density

measurement and oxidation test suggested that the increase in cross sectional area may be due to formation of creep voids along with the formation of oxide layer within the gauge length. The creep properties as results of weld strength reduction factors (WSRF) of welded joint specimens of P91 and P91B steel have been evaluated. It showed that the weld strength reduction factors of P91B steel are higher than that P91 steel.

Analysis of thermal fatigue behaviour of H13 hot work tool steel

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Dies for aluminium alloys die-casting fail because of number of different and simultaneously operating factors. Thermal fatigue cracking is a major mode of failure of dies. Crack initiates due to the large thermal shock experienced by the die surface when it is rapidly heated and quenched to 700°C and 150°C respectively. This thermal fatigue loading propagates the crack until gross failure occurs or die becomes unusable. XRD stress measurement can be a powerful tool for failure analysis or process development studies. Quantifying the residual stresses present in a component, which may either accelerate or arrest thermal fatigue, is frequently crucial in understanding the cause of failure. H13 steel is the most popular material for aluminium die casting dies. Immersion test apparatus was developed, which enables simulation of conditions during die casting and enables controlled thermal fatigue testing. Special specimens were developed for the test. The steel was treated by austenitising at 1030°C and quenched at cooling rates of 30, 43 and 56°C/min followed by multiple tempering with final hardness of 46-48HRC. Induced microstructures are revealed using optical microscope and phase compositions are assessed by XRD technique while mechanical characteristics are investigated based on hardness and toughness standard tests.After heat treatment, material exhibits the tempered martensitic structure with wider laths of martensite connected to a complex carbides mainly of M23C6 and M7C3 chromium carbides type. The intense martensite tempering is observed on the die surface, while the core material remains unchanged. A considerable hardness change is measured from the material surface to core up to three millimetres distance. Toughness property is found to be higher for increasing quenching rate from slower to higher. Results of residual stress analysis shows the dependency of tool manufacturing process and thermal fatigue conditions. Lower thermal gradients keeps the stresses in lower level.

Characterization of low cycle fatigue behavior of Inconel 718

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Inconel 718 is one of the most popular disc alloys for gas turbine engines. It has to withstand frequent startup and shutdown. Therefore high temperature low cycle fatigue resistance is one of the main criteria for the selection of materials for such an application. This paper presents low cycle fatigue test data on a commercial grade of solution treated and subsequently aged Inconel 718 alloy. The tests were conducted on INSTRON servo electric testing system at 600C under strain controlled mode at different strain ranges. With increase in strain amplitude the fatigue life found to be detoriated.The experiments revealed that initially with gradual increase in cycles the material shows cyclic softening behavior followed by constant curve characteristic and finally leading to fatigue failure. Cyclic hardening/softening curves, cyclic stress-

strain curves, and fatigue life were obtained at 600°C. A criterion based on15% drop in load during such a test was used to determine fatigue life time under all test conditions. The analysis of the life time data revealed that it follows Coffin-Manson relation having two distinct slopes. Microstructure of the alloy has been examined under SEM and TEM. The material before the test was found to have relatively coarse Ni3Nb (delta phase) at grain boundaries and fine coherent gamma double prime within the matrix. The grain boundaries were found to be serrated at several locations. Similar examination after LCF tests showed that the grain boundaries are the potential sites that are susceptible to initiation of fatigue damage. Barring this the microstructure of the alloy was found to be fairly stable during the long hours of high temperature exposure during the test.

Failure analysis of wire breakage during crimping operation

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Crimped wire is used for manufacturing of wire mesh. This wire mesh has a wide application in sugar mills, paper mill, coal and mining industries. Medium and high carbon steel wire rod is commonly used for making the wire mesh. Crimping is extensively and commonly done in just about all major manufacturing industries. After drawing operation, the wire passes through coupled dies and these dies are involves pressing the wires to get them in shape. A successful crimping operation depends on quality of wire and dies, lubrication, die pressure etc. This paper deals with failure analysis of wire mesh during crimping operation. Visual observation under stereo microscope revealed figure nail type of failure. Die marks were observed at some of the location of the failed wire. In the macrostructure, it was clearly revealed that, the thickness was not uniform in most of the sections. This observation indicates that the thickness reduction occurred due to localized pressure generated on certain location during crimping operation. In microstructural examination, micro voids were observed near the fractured end as well as at locations where size reduction was observed. The micro void formation was more in bend portion of thinner section compared to the thicker section. This indicates that, during crimping operation, die pressure was not uniform in all the bended locations. The void was formed by higher pressure of die. Thus the failure occurred due to manufacturing process (crimping).

Failure analysis of tyre bead wires during drawing

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High carbon steel (HCS) wires are vital material for various applications such as bead wire, spring, railway sleepers, slabs and steel cord for reinforcing pneumatic tyre. The bead is that part of the tyre that contacts the rim on the wheel. Drawn patented wires are used for tyre bead applications to achieve specific properties that must have good-mechanical properties so that the tyre bead can sustain the tyre safely, and yet retain adequate ductility to buckle easily around the wheel. Wire breakage during cold drawing is a complex phenomenon; wire breakages may be induced by improper wire-rod quality, inappropriate heat treatment, and/or lubrication breakdown on the wire surface. A comprehensive metallurgical investigation of failed/broken wire samples is therefore essential for understanding the origin of failure.The present work highlights failure investigation of wires of High Carbon grade during cold drawing operations. Failed wires samples were collected from wire drawing mill and in-depth investigation was carried out to find the

geneses of failure. The fracture surface was of finger nail type for the representative case considered for this work. Crow feet like defects including button like surface abnormalities were observed on the broken wire samples. The surface defect was observed near the fracture end and in localized manner. The etched microstructure revealed presence of hard phase appearing as a brown layer in the vicinity of the defect. From microstructure analysis, the brown layer appeared to be of martensite (which was further verified by micro hardness value). Presence of hard phase in the wire samples led to breaking. The surface condition indicated that improper lubrication during drawing led to the formation of hard phase.

Failure analysis of fiber reinforced polymer composite cross-ply laminate

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Composite laminates due to their high strength and stiffness to weight ratio are widely used in different load carrying structures. In order to obtain high stiffness, laminates with complicated lay ups are used. Laminates during the service are subjected to complex thermo-mechanical loading that causes damage accumulation. In the presented study, damage accumulation in the layers with fiber orientation transverse to the loading direction is studied. The first mode of damage is matrix microcracking or transverse cracking.In present study, the effective elastic modulus of the composite are determined by finite element analysis of the representative volume element (RVE). It is very crucial in such analyses that the correct boundary conditions be imposed such that they simulate the actual deformation within the composite. In the present analysis, the appropriate boundary conditions on the RVE for various loading conditions are determined by judicious use of symmetry and periodicity conditions and effect of damage mechanism on elastic modulus is studied. Also, in this study, transverse cracking initiation on a fiber/matrix level is investigated. The measure of the damage level in laminates containing 90^0 layer is crack density in that layer. With increasing the crack density in transverse layers, elastic properties of whole laminate is decreasing. FEA models are developed in order to predict the level of elastic properties degradation. These models are verified by comparing prediction with experimental results available in literature. FEA results show good agreement with experimental results taken from the literature survey. Stress distributions in layers are studied on meso level and same stress is applied to RVE to predict the exact damage mechanism responsible to initiate the failure.

Modal analysis of automotive seating system

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The purpose of this study is to carry out the modal analysis of automotive seating system. A simple novel procedure has been used to solve the vibration problem of seat and determine its effect on human comfort through magnification factor or dynamic amplification factor. The aim of this present work is to find out the natural frequencies and magnification factor of seat component. The magnification factor shows the amplification of static amplitude in dynamic condition. This has been calculated at different excitation frequencies for fundamental frequency of the component. It has been observed that some of the components are critical from resonance point of view and shows higher dynamic amplification factor at the lower excitation frequency range. The lesser the magnification factor the better is the seat from

dynamic point of view. We aim here to keep the magnification factor as low as possible. The natural frequencies of seat components have been found out by using Hypermesh and Nastran, and the dynamic amplification factor has been calculated with the help of analytical method. It is concluded that higher the natural frequency lower is the resonance between human body and seat components. This leads to increase the comfort of the occupant while riding.

Effect of processing parameters on the quality of adamite grade castings

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Adamite grade castings are high carbon steel base castings, with about 0.8-1.6% carbon, 0.8-1.2% chromium, and 0.2-0.4% molybdenum and some silicon, manganese. Severity of defects in this type of casting increases with its size and the volume. Percentage carbon, temperature, lining/coating, method of pouring and re-poring time etc. are the important parameters to be controlled. Through this paper it has been tried to cover one case study of transverse crack in adamite grade castings & a practical approach to overcome the problem. Various problem solving tools like pareto, 4M analysis, why-why analysis are used to deploy standard conditions. Many tools techniques are adopted to control each process parameters.

Condition monitoring technique for fault detection of rolling element bearings

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Rolling element bearings are frequently encountered in rotating machine due to their load carrying capacity and low friction characteristics. The complexity of loading mechanism in bearing shows its effect in the form of distributed and local defects. Defects in bearing causes catastrophic failure and Hertzian contact stresses, which increases the vibration level in the machine. Efficient functioning of machine critically depends on good health of employed rolling bearings. Hence, health monitoring of rolling element bearing through their vibration responses is a vital issue. There are various techniques for vibration analysis such as wear debris analysis, oil analysis, temperature method and condition monitoring. Most of the work attempted in various papers was condition monitoring done by time domain and frequency domain method. Some of the work focused on time domain analysis, they compared statistical parameters of time domain signals with shaft speeds. In few papers the work is focused on characteristic fault frequency of bearing in frequency domain analysis. The proposed work is to develop the experimental test rig to acquire vibration levels of defective bearing and also the frequency domain analysis is carried out by FFT analyzer to compare the vibration amplitudes on characteristic fault frequency of bearing. The effect of rotational speed, defect position and load on the diagnostics of rolling element bearing defects is investigated. Vibration signatures of these parameters are compared and sensitivity of vibration analysis is studied.

Investigation into severe incrustation in single stage wet scrubber of a BOF steelmaking gas cleaning circuit

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In Durgapur steel plant there are three converters for production of steel through BOF route. The particulate matter in the gas generated during blowing is cleaned through a system primarily consisting of a wet particulate scrubbing circuit with cooling skirt, hood, stack and ID fan as well as clarifier -thickener. Recirculating water is to carry the total scrubbed particles to the thickener clarifier via the mixing tank. In the thickener-clarifier, the sludge settles at the bottom and clarified water is recycled in scrubber. While the suspended solids level in recycled clarifier overflow is well within operational norm of 100 mg/l, significant particulate material gets deposited primarily in the scrubber zone. This in turn affects the gas flow to such an extent that converter has to be stopped for manual cleaning of various stages of scrubber unit. Besides, this also causes serious problems in associated pipe lines, nozzles which have also led to tripping of ID fans, affecting production level. With increased demand of production the problem gets accentuated. A study to establish process/control parameters for addressing the problem and identify a viable solution was undertaken. Detailed time series data has been generated in terms of nature of deposition, deposition characteristics and circulating water characteristics in scrubber clarifier circuit. It has been hypothesized and established that in terms of nature, deposition material does not lead to rock like encrustation; Based on time series data it has also been concluded that a number of controllable variables like particle size range of charge mix, water hardness, pH and alkalinity and certain non-controllable attributes (e.g, basic design of scrubber) is responsible for deposit formation. A combination of improved operational practice and introduction of a minimally invasive physico-chemical system has been conceptualized and proposed to address the challenge.

Design and analysis of leaf spring for tanker trailer suspension system

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A leaf spring is a simple form of spring, commonly used for the suspension in wheeled vehicles. Leaf Springs are long and narrow plates attached to the frame of a trailer that rest above or below the trailer's axle. In the pursuit of Design and Analysis of Leaf Spring for Tanker Trailer Suspension System an attempt is made to obtain the values of safe stress and deformation by carrying out finite element analysis for software tools. The objective was to find safe stress and deflection of leaf spring by applying safe load. After carrying out analysis and experimentation work, the safe design stress and deformation were found to be in close agreement with analytical and experimentation results.

Analysis of bend test specimen

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The bend test is performed for determining the ability of metallic materials to undergo plastic deformation in bending. There is no standard for interpretation of bend test results.Bend test was carried out on 4 samples of Hot rolled medium and high tensile strength steel. Results are discussed, interpreted and critically reviewed by considering acceptance criteria of various standards such as ASTM, Indian Standards (IS), Japanese Industrial Standards (JIS), and International Standard organization (ISO). After investigation, it is concluded that cracks observed during bending are not generated during the bend test. It also reveals that they were nothing but the processing defects such as seam, lap etc. which were opened up during bend test.

FUEL CELLS AND BATTERIES

Invited talks

Session 1: Nov 13, 2014: [15:30 - 17:30]

Fuel cells: Potential in India and CSIR's efforts to realize it

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Hydrogen is a clean fuel with the highest known energy density. A fuel cell converts the chemical energy of hydrogen into electricity through an electrochemical oxidation process, which is more efficient than combustion. FCs offer an efficient, reliable, cleaner, silent and decentralized energy generation alternative to today's widely used technologies such as combustion of diesel or coal. Fuel cells are being used in commercial, residential, industrial and strategic applications in several countries. In India, fuel cells are likely to find wide range of applications driven by our unique geo-political demography. However, the poor awareness and non-availability of this technology in India, as well as the high capital costs of FCs and the cost of hydrogen are major barriers for its wide-spread implementation. CSIR has embarked on a major technology development initiative on FCs for surmounting many of these barriers so that this technology will be able to find its rightful place among the several alternative energy technologies that are coming up in a major way in India. In this talk I will summarize CSIR's efforts in this mission mode technology development program, highlight some of our achievements so far and discuss the roadmap ahead.

Recent advances in nanomaterials for electrochemical energy conversion devices at CFCT, ARCI

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Conversion of energy from one form to another is an important aspect in energy management as energy is required in a specific form at the point of use. The characteristics of the energy conversion devices depend on the properties of the materials used in these devices. Batteries of various types, Super capacitors, and hydrogen fuel cells have been subject of intense investigations in recent years as they play a very critical role in the energy storage domain and electric vehicles. Materials with nano structures are expected to play significant role in improving the efficiency of these devices. This aspect is reflected in the number of publications and report that keep appearing in open literature. Centre for Fuel Cell Technology (CFCT) at International Advanced Research Centre for Powder Metallurgy and New Materials (ARCI) has been engaged in developing several hydrogen technologies and electrical energy storage devices during the last ten years. The development activities cover wide range of subjects from materials to system development with eventual field trials. During this period, CFCT developed and demonstrated (i) Grid Independent Power Systems (GIPS) based on low temperature PEM Fuel cell in the range 1- 20 kW. These GIPS system

include, besides the fuel cell stacks, required inverters/converters and control systems. (ii) Integrated and demonstrated indigenously developed PEMFC in transportation applications as range extender in 2, 3 and 4 wheeler electric vehicles. (iii) developed and demonstrated a 1 NM^3 hydrogen generator. As part of these advances CFCT has also developed new process know-how for making low cost bipolar plates, electro catalysts, electrolyte membrane etc., The Centre has recently demonstrated a 600 watts high temperature PEM Fuel cell. It is now well established globally, that hydrogen and Fuel cells could contribute significantly to provide clean energy solution and reduce air pollution as they can be used in diverse application domains. However, widespread applications of fuel cells depend on the cost of the fuel cell system and availability of low cost hydrogen. Fuel cell stack cost dominates the fuel cell system cost. To reduce cost, a number of issues need to address which include efficient manufacturing techniques and identification of new high performance low cost materials / low cost components. One of the approaches is to develop high performance catalysts which can reduce the active area of the electrode. Performance of the catalyst depends on a number of properties. Many of these properties are contra-indicating. The catalyst should show not only high performance but also stability under the environment of fuel cells. The high catalyst activity measured in half cell experiments need to be translated into the full working electrode performance in fuel cells. The design of the electrode as well as the properties of the other components of the electrodes are also important. The journey of using of electro catalyst for practical low temperature fuel cells which started with the use of unsupported platinum (Pt black) in the 1960s has now arrived at using ultra low loading of catalysts supported on carbon but retaining high performance. These developments went through a large number of various approaches which include developing carbon supported catalysts, use of carbons of different morphologies including the recent sensation graphene etc. These catalyst sizes were in the range 2-6 nanometers.In transportation applications which involve frequent start-stop cycles, it has been observed that the carbon supported catalysts used in the fuel cells tend to lose their efficacy owing to carbon corrosion. This carbon corrosion leads to catalyst agglomeration and thus loss in electrochemical active surface area. The degradation rate is found to increase with higher operating voltages. One approach to overcome this problem is to develop catalysts supported on materials with better corrosion resistance and strengthening metal–catalyst support interactions. These approaches include functionalization of carbon and use of oxides and carbides as supports. The other approach which is gaining importance is the use of unsupported catalysts with nano structure. Today there are methods available to made unsupported catalysts with different morphologies. Metal catalyst with high surface area can be realized with novel nano structures. Use of such catalyst would reduce the catalyst quantities and thus reduce the cost. Unsupported catalysts with meso and nano porous structures have also been reported.In the author's laboratory, several of these approaches have been investigated and a large number of unsupported metal catalysts with novel nano structures and as well as catalyst supports with nano structures, graphene supported catalysts which have high potential to overcome the corrosion issues and also reduce the cost of the fuel cell stacks have been developed. One of the major issues with these catalysts is that many of them are not amenable for large scale synthesis. In the author's laboratory a new process has been developed which allows large scale synthesis of these nano materials. In addition to development of nanomaterials for fuel cell applications, CFCT has also developed materials with nano structures which have been used to build super capacitors, batteries and used for storing hydrogen gas. The talk would highlight some of the recent developments at the author's laboratory in developing novel materials with nano structures for use in various energy conversion devices.

Lithium-ion Battery Advances Now and in Near Future

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The popularity of the Li-ion battery is due to the advantages offered over other secondary like lighter in weight for a given capacity, delivers a high open-circuit voltage, low self-discharge rate (about 1.5% per month) and environmental benefits being rechargeable character and reduced toxic landfill. However Li-ion batteries have also struggling with issues such as poor cycle life, particularly in high current applications (EV applications), rising internal resistance with cycling and age, safety during overheated or overcharged condition and current applications demanding more from Li-ion battery capacity. To realize such high demand, one need to develop or introduce new technology may be based on fabrication or material development in this battery system. It is also equally important to study the existing electrode materials from their basic properties. Lithium interaction materials without further doubt can be classified into three categories; layered structure ($LiCoO_2$, $LiNiO_2$, etc., Spinel frame work structure ($LiMn_2O_4$, LiV_2O_4) and Olivine structure ($LiFePO_4$, $LiMnPO_4$ etc.). This talk will touch upon the basic requirement and current development in electrode chemistry including few existing electrode materials and introduce a new class of layered electrode materials for high energy density lithium ion battery.

Graphene electrodes for next generation lithium-ion batteries

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Conventional graphitic anodes in lithium-ion batteries provide a maximum specific charge storage capacity of ~372 mAh/g. Moreover graphitic anodes cannot provide high power densities due to slow diffusivity of lithium ions in the bulk electrode material. In my talk, I will describe novel thermal and photo-thermally reduced free-standing graphene paper as high-energy and high-power density capable electrodes for lithium-ion batteries. These materials are also structurally robust and deliver stable performance for thousands of cycles of charge and discharge. I will explain the fundamental mechanisms that enable the superior performance of graphene electrodes over their graphitic counterparts. I will also discuss how defects in the graphene lattice can be used to initiate the plating of lithium metal in the interior of porous graphene networks. I will show how the nano-porous nature of the graphene electrode prevents dendrites from forming in such structures. Using this principle, I will demonstrate a full-cell configuration where graphene based electrodes is utilized as both the anode and the cathode and Lithium metal is reversibly plated and contained within the porous graphene structure. Such all-carbon electrode chemistries could potentially offer an environmentally friendly solution that does not utilize hazardous materials such as cobalt in battery manufacturing.

Session 2: Nov 14, 2014: [08:30 - 10:30]

Nanoscale Electrode Assemblies for High Energy and Power Density Batteries

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It has been widely recognized that electrochemical devices such as battery and fuel cells can be utilized as energy devices in large scale applications such as electric power grid and also be integrated with renewables e.g. solar, wind. Rechargeable battery performance and stability are completely determined by the participating electroactive materials in the reversible chemical reaction and storage mechanism.

The materials composition and structure ascertain the rate and yield of electron and ion transport in both bulk and across various interfaces. Tailored materials assembly and composition strategies (including (composites, inorganic-organic hybrids) at the nanoscale have been demonstrated to be highly effective in developing high energy and power density batteries. Synergistic and confinement effects play a critical role in determining the materials physico-chemical properties. also provide multitude of avenues for generation of advanced functional materials for efficient energy storage. Exploration of novel materials in these lines may pave the way for the development of integrated electrochemical devices of varying sizes for varied applications.

Defect Reaction Mechanism in CeO_2 Doped $SrTiO_3$

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Donor doped $SrTiO_3$ materials are potential candidates for anode in solid oxide fuel cells due to their high electronic conductivity and good structural and chemical stability at high temperatures and under a wide range of oxygen partial pressures. However, so far the fundamental understanding related to the defect structure of donor doped $SrTiO_3$ has not been clear. The knowledge of defect chemistry is essential in order to obtain a reliable and meaningful correlation between the physical properties and composition. In the present work, a defect reaction mechanism will be presented for CeO_2 doped in $SrTiO_3$ under oxidizing conditions. We had performed a systematic study to examine the phase formation in the nominally cation-stoichiometric series, i.e. $Ce_xSr_{1-x}TiO_{3+x}$ when calcined in air. Conventional solid state route was used to synthesize dense ceramic pellets of $Ce_xSr_{1-x}TiO_{3+x}$ (x = 0, 0.02, 0.04, 0.07, 0.10 and 0.15). SEM performed on the sintered pellets of doped compositions revealed the presence of residual phase of CeO_2 in the dark color matrix of perovskite phase. However, the lattice parameter of $Ce_xSr_{1-x}TiO_{3+x}$ was observed to decrease linearly up to 15 mol.% CeO_2 obeying Vegard's law. The phase quantification analysis was performed in order to estimate the mole fraction of fluorite and perovskite phases, and verify the validity of the proposed defect reaction mechanism. The comparison of the obtained results with the theoretical model based on the proposed defect reaction will be discussed. In addition, the conductivity behavior of CeO_2 doped $SrTiO_3$ measured using impedance spectroscopy from 300-700°C will be presented.

Session 3: Nov 15, 2014: [11:00 - 13:00]

Classical molecular dynamics and quantum ab-initio studies on lithium-intercalation in interconnected hollow spherical nano-spheres of amorphous silicon

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A high concentration of lithium, corresponding to charge capacity of ~4200 mAh/g, can be intercalated in silicon. Unfortunately, due to high intercalation strain leading to fracture and consequent poor cyclability, silicon cannot be used as anode in lithium ion batteries. But recently interconnected hollow nano-spheres of amorphous silicon have been found to exhibit high cyclability. The absence of fracture upon lithiation and the high cyclability has been attributed to reduction in intercalation stress due to hollow spherical geometry of the silicon nano-particles. The present work argues that the hollow spherical geometry alone cannot ensure the absence of fracture. Using classical molecular dynamics and density functional theory based simulations; satisfactory explanation to the absence of fracture has been explored at the atomic scale.

Session 2: Nov 14, 2014: [08:30 - 10:30]

High platinum cost: obstacle or blessing for commercialization of fuel cell technology

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Polymer electrolyte fuel cells (PEFCs) have tremendous potential for transport and portable applications and it is in the early stage of commercialisation. Like other new technologies during pre-commercialization phase, PEFC technology is also facing several challenges and uncertainties. Use of the platinum catalyst is considered as one of the key obstacles due to its cost and scarcity. In particular, cost and durability of the fuel cells are some of the prime challenges towards their commercialization. Current cost of PEFC technology are estimated to be at least five fold greater than the target cost, even with the consideration of cost saving by mass production. Generally, high cost of platinum, is blamed and considered as the main hurdle towards commercialization, especially since it is not possible to bring down the cost by mass production of fuel cells. In the present work, a life cycle cost analysis is carried out based on the inflation and discount rate of the market for different economic zone and it is shown that the high platinum cost could be beneficial and should promote the commercialization of the fuel cells if proper business model is in place.

All lanthana based SOFC: component properties and design

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Solid oxide fuel cell (SOFC) is an electro-chemical energy conversion device, and getting popularity because of its fuel flexibility, high efficiency and long lifespan. Though YSZ material is commonly used as few component materials (electrolyte and anode), its high operating temperature limits its usages. Alternatively, ceria based materials are less reliable because of their electronic contribution (deleterious effect in electrolyte) in reducing atmosphere. However, doped $LaGaO_3$ at La and Ga sites with different dopants develop promising component materials with improved performances (like conductivity, stability, etc). Since these properties are also process (synthesis) dependent, Pechini method with a specific heat

treatment schedule was adopted. Moreover, the thermal expansion coefficient has also been tailored to produce acceptable SOFC component system.

The effect of electrode thickness on charge/discharge hysteresis of lithium-ion cell

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Li-ion batteries (LIB) are the most promising energy storage units in electric and hybrid electric vehicles because of their high energy density and slow self-discharge. LIBs are made up of electrochemical cells connected in series and/or parallel. The Li-ion cell consists of three active components; negative and positive electrode coated on copper and aluminium current collectors, respectively, and a separator soaked with an electrolyte.Fabrication of LIB cells involves various crucial steps like (i) slurry preparation; (ii) coating of slurries on current collectors;(iii) calendaring of the electrodes; (iv) slitting; (v) winding;(vi) tab welding;(vii) electrolyte filling; and (viii) sealing of the can by laser welding. The qualities of the electrode coatings determine the electrochemical performances such as capacity, cyclic stability and impedance; and also the safety aspects of the battery. The quality of the coating is determined by uniform dispersion of the active material in the slurry. Rheology plays a vital role in optimizing the slurry formulation. The main ingredients of the slurry are active material, conductive carbon, binder and solvent. The slurry of anode and cathode (15 L) were prepared using dual shaft vacuum mixer. The investigations on active materials like $LiFePO_4$ and graphite to NMP ratio on the rheology of the slurry have been studied. Anode and cathode electrodes of various thicknesses (50-100 µm) were prepared by reverse comma coating technique on Cu and Al foils, respectively. The electrodeshave been characterized by various physicochemical and microscopic techniques. Preliminary electrochemical investigation indicated a linear increase in the hysteresis of charging and discharging voltages with respect to coating thickness. The detailed study on electrode preparation, coating thickness versus electrochemical performance will be presented.

Advances in metal supported SOFCs - cells, stacks and systems at NFTDC, Hyderabad

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We hope to discuss the progress in developing Metal Supported Intermediate Temperature Solid Oxide Fuel Cells, at NFTDC, in Hyderabad. This project is in collaboration with University of Cambridge, UK, and funded as part of a Indo-UK initiative. Usage of Ferritic Steels as anode supports is an old idea originally propagated by the late Prof. Brian Steele of Imperial College, UK; an idea that eventually saw the birth of Ceres Power, UK. The excellent compatibility of Ferritic steels with YSZ/GDC and such ceramic electrolytes, in terms of Coefficient of Thermal expansion, along with low costs of the metal relative to ceramics like YSZ and GDC, makes the concept very attractive. It does introduce several limitations in operating temperatures of the Cell (< 700°C), constraints in cell manufacturing processes (sintering atmospheres, temperatures), and a compromise between efficiency versus operating life of the Cell/Systems. In this work, we hope to present NFTDC's work towards the various processes towards building metal supported SOFCs, using SS430 as the Ferritic Steel of choice, the cell and stack assembly, stack design, performance

data and some systems design under intermediate temperature regimes (600°C). NFTDC (Non-Ferrous Materials Technology Development Center) is an autonomous R & D and Technology Center, devoted to the development of technologies and components, using metals, alloys and ceramics; with a strong focus on design, and manufacturing.

Factors controlling the oxygen reduction kinetics in Sr-doped lanthanum cobaltite and ferrite based composite cathode

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LSCF ($La_{0.54}Sr_{0.40}Co_{0.20}F_{0.80}O_{3-\delta}$) based cathode materials have good electrical conductivity as well as high oxygen surface exchange coefficient. In the present study, composite cathode based on LSCF and LSFC ($La_{0.54}Sr_{0.40}Fe_{0.20}Co_{0.80}O_{3-\delta}$) have been synthesized by mechanical mixing process. DC electrical conductivity and electrode polarization of this composite cathode have been evaluated and they are found to be 686 Scm^{-1} and 0.02 Ω cm^2 successively. The electrochemical performance exhibited by this composite cathode is remarkably high. The current density obtained from the single cell constituting Ni-YSZ anode/ YSZ electrolyte/ CoCGO interlayer/ (LSCF+LSFC) composite cathode is found to be 3.98 A cm^{-2} at operating voltage 0.7 V at 800 ^{o}C. Now, attempt has been made to explore the oxygen reduction reaction (ORR) kinetics occurring in composite cathode using impedance spectroscopy. The polarization resistance of cathode composite has been measured by two-electrode impedance methods usingsymmetricLSCF+LSFC)/ electrolyte/ (LSCF+LSFC) cells, where the electrolyte was 1 mm thick CGO (Gd-doped CeO_2) and CoCGO (Co and Gd doped CeO_2). Data have been collected under a uniform air atmosphere. The cathode ASR has been determined from raw impedance plots where the high-frequency offset is due to the electrolyte resistance and the diameter of the complete arc is due to the cathode polarization resistance. The electrode polarizations as determined have been found to be significantly low 0.020 Ω cm^2 for CoCGO electrolyte and 0.026 Ω cm^2 for CGO electrolyte at 800 ^{o}C. Linear sweep voltametry (LSV) has been performed to calculate the exchange current density of the composite cathode applying Tafel equation for (LSCF+LSFC)/electrolyte/Pt cells by two electrode mode in the voltage range 0-0.5V with sweep rate 0.5 mV and the calculated exchange current density are found to be 0.141 Acm^{-2} and 0.039 Acm^{-2} for CoCGO and CGO electrolytes, respectively, which are considered to be significantly higher for oxygen reduction reaction.

Fabrication of dense zirconia film for solid oxide fuel cell application by electrophoretic deposition technique

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Suspensions of 8 mol % yttria stabilised zirconia (YSZ) particulates in isopropanol medium are prepared using benzoic acid as dispersants. The effects of dispersant concentration on electrical conductivity, pH and stability of the suspension are studied in detail to optimize the suspension chemistry. Electrophoretic deposition (EPD) has been conducted to produce thin and dense YSZ electrolyte films onto a conducting plate for in-depth study of deposition kinetics. Good quality films on conducting substrate are obtained at an applied voltage of 25 V for 3 min. However, EPD is carried out to fabricate YSZ electrolyte films onto the

non-conducting NiO-YSZ anode substrate using a conducting steel plate on the riverside of the porous anode to construct the solid oxide fuel cell (SOFC) half cells. Upon co-firing at 1400 ^{o}C for 6 h dense YSZ films of different thickness are obtained. A suitable cathode composition ($La_{0.65}Sr_{0.3}MnO_3$) thick film paste is then screen printed on the top of the sintered YSZ electrolyte layer. Finally, a second stage of sintering of such cathode thick film at 1100 ^{o}C for 2h yield a single cell SOFC. The electrochemical performance of such single cells are then evaluated under different applied load and the measurement is carried out at 700, 750 and 800 ^{o}C using hydrogen as fuel and oxygen as oxidant. A maximum power output of 1.91 Wcm^{-2} is obtained at 0.7 V when measured at 800 ^{o}C from a single cell having an electrolyte thickness of ~ 5 µm.

Development of LSCM protective coating on ferritic stainless steel for interconnect application in IT-SOFC

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Low cost ferritic stainless steels are candidate interconnect materials for intermediate temperature SOFC. However, these interconnect materials are prone to growth of non-protective oxide scale and evaporation of chromium species into the cathode, leading to increase in cell resistance and polarization resistance. A protective layer of conducting oxides can prevent oxidation of the metallic interconnect and diffusion of chromium species to the cathode. Protective $La_{0.75}Sr_{0.25}Cr_{0.5}Mn_{0.5}O_{3-\delta}$ (LSCM) layer was developed over the ferritic stainless steel by screen printing method to overcome these issues. Fine LSCM power synthesized by solution polymerization method has been used to form a dense layer over the interconnect material. LSCM has been evaluated for its functional properties. Samples of the interconnect material with and without the protective coating were exposed to oxygen atmosphere at 800°C for 300 h and 6 thermal cycles from room temperature to 800°C. The area specific resistance with protective coating was 5 $m\Omega.cm^2$, ascompared to 2 $\Omega.cm^2$ for the uncoated samples. Good adherence of protective layer and control over the growth of oxide layer has been observed from the SEM and EDS analysis of the coating interface. The results suggest that LSCM can be used effectively as protective conducting coating on ferritic stainless steel interconnects by way of preventing the growth of oxide scale and evaporation of chromium species.

Fabrication of solid oxide fuel cell components by tape-casting and screen printing approach

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Solid Oxide Fuel Cell (SOFC) is an electrochemical device in which the chemical energy of Fuel, such as hydrogen, natural gas is converted to electrical energy in an energy efficient and environmental friendly manner. Among the SOFC designs, the two very popular have been the tubular and planar configurations. SOFC has mainly three components, such as electrolyte, cathode and anode. In planar design, the majority of the components are fabricated by tape-casting and screen printing approach and they become the backbone of the whole technology. These technologies give ample scope to tune proper microstructure of each component, which can be suitably modified in the fabrication steps depending upon the requirements.In this work for electrolyte supported cell YSZ (yttria stabilized zirconia), electrolyte was

fabricated through tape casting and electrodes by screen printing approach. Large area impervious YSZ electrolyte tapes of thicknesses between 120 and 150 µm were fabricated by tape casting method. In anode supported fuel cell the Ni-YSZ anode was also fabricated via tape-casting. The electrolyte was applied either by co-tape-casting or by screen printing. By co-tape-casting approach nearly 40 µm thick electrolyte could be co-casted on tapes having thicknesses > 400 µm. In the screen printing methodology, it was possible to fabricate very thin electrolyte of nearly 4-10 µm depending upon the applied number of coatings of the electrolyte. Screen printing methodology was further applied to the fabrication of porous electrode. Thus, tape casting and screen printing methodology have been successfully applied to fabricate both electrolyte and electrodes for SOFC applications.

Doped $SrTiO_3$ based anode in SOFC

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Bi-doped materials, with La, Y and Nb present at both A and B sites of $SrTiO_3$, have been synthesized and tested as anode materials for solid oxide fuel cells (SOFC). The cell performances with bi-doped anode materials, viz., lanthanum yttrium strontium titanate (LYST) and lanthanum niobium strontium titanate (LNST), were found to be superior compared to those with single-doped anode materials, viz., LST and NST. Addition of 20 wt.% Ni-YSZ to LYST and LNST improved the cell performances from 23 mW/cm^2 to 77 mW/cm^2 and 25 mW/cm^2 to 38 mW/cm^2, respectively. LYST-Ni-YSZ anode gave the maximum power density of 70 mW/cm^2, which was stable for 60 h of operation. Characterizations, including XRD, SEM and TEM were also performed with the anode materials to understand the phase assemblages, morphologies and microstructural aspects.

Investigation of synthesis pathways of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ solid state assisted Co-precipitation

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$LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ (LNCA) has been proposed as a promising cathode material for automotive applications due to their high energy density, high specific capacity, less toxicity and lower cost. LNCA has rhombohedral layered structure with R$\bar{3}$m space group and theoretical capacity of 278 mAh/g in the range of 3.6-4.0V; against Li/Li^+. In order to obtain ordered, electrochemically active monophasic LNCA, the synthesis conditions such as annealing temperature, duration, pH and sequence of doping need to be optimized.We have synthesized LNCA by co-precipitation of transition metal hydroxides followed by solid sate synthesis with lithium source. Aluminum precursor is introduced either during hydroxide precipitation or solid state synthesis. In this presentation, we will be discussing the effect of Al precursor introduction during co-precipitation or solid state synthesis on morphology and phase purity of hydroxides and LNCA. Introduction of aluminum during precipitation of hydroxide leads to the formation of layered double hydroxide (LDH), which was confirmed by XRD and IR spectroscopy. The formation of LDH layer is due toAl disturbing the charge neutrality of hydroxide, since stable oxidation state of Al is 3+ while that for transition metals is 2+. Therefore, the presence of Al incorporates SO_4^{2-} and H_2O into the layers of transition metal hydroxides to maintain the charge neutrality. The influence of Al on the morphology of

hydroxides and LNCA are investigated using SEM and TEM.Further, the optimization in the ratio of Li to transition metal taken during the synthesis is essential in order to obtain stoichiometric LNCA and to compensate for the Li loss due to evaporation during high temperature annealing. The concentrations of Li^+were investigated using ion chromatography. Also, the influence of stoichiometry, morphology on the electrochemical characteristics of LNCA prepared by solid state assisted co-precipitation will be discussed.

Session 3: Nov 14, 2014: [11:00 - 13:00]

Understanding the lithiation/delithiation mechanism in few layer graphene

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Graphitic carbon is still the most commonly used anode material for Li-ion batteries; with recent works demonstrating that its 2-dimensional building block, viz. graphene, on its own may possess even higher Li-capacity. However, the mechanism of lithiation/delithiation of graphene is yet to be understood, and so is the mechanical integrity upon lithiation/delithiation of graphitic carbon as a whole. Therefore, using few layer graphene (FLG) as model material, an attempt has been made in this work to better understand these issues. This is further aided by monitoring the dimensional changes and stress developments *in-situ*[using multi-beam optical stress sensor] during lithiation/delithiation of FLG filmsin a custom-made electrochemical cell. The FLG (~ 7 layers) films were deposited via chemical vapor deposition at 1000°C by decomposing methane gas on copper foil. The electrochemical performances of FLG were studied via cyclic voltammetry and galvanostatic charging/discharging against Li. The FLG electrodes showed high reversible capacity of ~2 $\mu Ah/cm^2$ at a current density of 2.7 $\mu A/cm^2$, which is nearly an order of magnitude greater than that for bulk graphite. Additionally, appreciable cyclic stability and good rate capability were also observed for these thin film electrodes. Interestingly, the net in-plane stress development that was recorded upon full lithiation agreed reasonably well with the expected stress based on classical Li-intercalation in between the graphene planes (i.e. LiC_6 formation). As will be discussed, these observations tend to support the hypothesis that the excess capacity might be associated with the surface, edge planes or defect sites. Another interesting observation has been that of stress release at potentials between 0.5-0.25V (against Li/Li^+), which got accrued at the higher current densities. Such observations might throw some light into the mechanical integrity/degradation of graphitic/graphenic carbon based electrodes, especially during the initial stages of stages of lithiation and later stages of delithiation.

Controlling the nitrogen content of iron based electrocatalysts via selenium addition

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Non-precious metal electrocatalysts based on pyrolysed metal-nitrogen-carbon (MNC) are viewed as inexpensive replacements for expensive platinum-based electrocatalysts for the oxygen reduction reaction (ORR) in fuel cells. One of the drawbacks of all the reported procedures to synthesize MNC electrocatalysts is the inability to control the nitrogen content. Since the type of nitrogen present (pyridinic, pyrrolic or quaternary) and its proportion are both seminal in deciding the ORR activity of the electrocatalyst, it is important to carefully study the effects of nitrogen content on the properties of the electrocatalysts. In this study, we use selenium as the ligand that can replace the nitrogen coordinated to iron in the electrocatalyst, thereby imparting control on the nitrogen content of the electrocatalyst prepared by high pressure pyrolysis of a composite consisting of melamine, ferric nitrate nonahydrate and Ketjenblack EC-600JD carbon. Upon introducing 14 at.% selenium, the N content of the catalyst dropped to 3.7 wt.% and the ORR activity reached 7.2 mA cm^{-2} at 0.8 V vs. RHE, indicating the important role of nitrogen on ORR activity. We have also concluded the need of iron to complete the active site, by way of complexing the iron site with bipyridine, which increased the overpotential towards ORR as much as 60 mV. More importantly, from our catalysts the intermediate of ORR reaction that is peroxide generation is very low, even at very low catalyst loading of 50 cm^{-2}, whereas catalyst synthesized by other approaches produces H_2O_2 as high as 80% at low catalyst loading. It is important to mitigate the peroxide production, as the radical produced from this peroxide intermediate has potential to degrade the solid electrolyte membranes used in fuel cells or batteries. From this study we believe that high pressure pyrolysis yields a homogenous catalyst which could enhance the complete $4e^-$ reduction of oxygen.

Development of alkaline and rare earth co-doped ceria electrolytes for IT-SOFC

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Gadolinia doped ceria (GDC) has been found to be a promising electrolyte material for intermediate temperature solid oxide fuel cells (IT-SOFC). GDC can be co-doped with other aliovalent elements to enhance the conductivity. In this study, GDC was co-doped with SrO (Sr-GDC) and doubly co-doped with SrO and Pr_2O_3(Sr-Pr-GDC) and its effect on the electrical conductivity was studied. Powders of GDC and the co-doped compositions were processed by solution combustion synthesis. X-ray diffraction (XRD) analysis confirmed the formation of phase pure powders which were found to be nanocrystalline in nature. TEM observations also confirmed the nanocrystallinity in the synthesised powders. Sr co-doping helped achieving a high sintered density owing to its low melting point. The relative density of GDC, Sr-GDC and Sr-Pr-GDC were 94.2%, 96.6% and 96.1% respectively. Ionic conductivities of the sintered pellets were measured using AC two-probe impedance spectroscopy as a function of temperatures from 400ºC to 800ºC. Both Sr and Pr co-doping resulted in considerable improvements in the conductivities of GDC. The doubly co-doped composition (Sr-Pr-GDC) exhibited higher conductivity than GDC and Sr-GDC.

Fabrication of cathode supported tubular SOFC through iso-pressing and co-firing route

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Solid oxide fuel cell (SOFC) is an electrochemical device which produces electricity from the direct conversion of chemical energy to electrical energy in an efficient and environment friendly manner. In a SOFC dense electrolyte layer is sandwiched between two porous electrodes (cathode and anode) and oxygen is passed at the cathode side and hydrogen at anode side. There are two basic design configurations in SOFC, namely, planar and tubular. In each of these configurations the cell can be anode supported, cathode supported or electrolyte supported. Higher power density is normally reported in anode supported planar design. However, sealing requirement is more stringent in planar configuration while forming a stack. Tubular SOFC has better thermal shock resistance, lower start-up time and requires minimum sealing. Different fabrication methodologies have been adopted in manufacturing of such cells. In the present work, LSCM cathode supported tubular SOFC has been fabricated by a co-pressing and co-firing route. The one-end-closed tubular cathode support was initially fabricated by cold isostatic pressing (CIP) and subsequently coated with YSZ electrolyte and NiO-YSZ anode layers. The coated tube was co-pressed in CIP and co-fired at 1350°C. Microstructural investigation indicated formation of dense electrolyte coating and porous electrodes. Symmetrical cells in planar disc configuration have been fabricated to simulate the interfaces of tubular cell and area specific resistance (ASR) for interfacial polarization has been determined by electrochemical impedance spectroscopy (EIS) technique. The results suggest that the electrode-electrolyte interface of a cell fabricated by co-pressing and co-firing approach has good adherence and reasonably low polarization resistance and hence, the present technique can be a viable one for fabrication of LSCM cathode supported solid oxide fuel cell.

Issue related SOFC stack design and improved performances

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The performances of the scaled-up solid oxide fuel cells (SOFC) are severely affected by ohmic and concentration losses. The ohmic losses increase with reduction in macro and microscopic contact area, whereas the concentration losses depend upon the distribution of fuel and oxidant over the cell active area. The present work aims to investigate the influence of the interfacial resistance (ohmic), both in terms of the external compression load, i.e., the interaction at the microscopic level and macroscopic contact area. Also, the effect of the uniformity of the flow distribution on the performance of the scaled-up SOFC has been studied. To that end, the base configuration has been modified at different stages and the performance of the cell has been noted. The electrochemical performance has been observed to enhance by 62% with application of the optimal external contact pressure, as estimated based on the materials properties. A noticeable 108% enhancement was apparent due to the increment of contact area. Another 50% enhancement in performance has been observed by making the gas distribution more even, by designing appropriate flow fields. Overall, 220% enhancement in the performance has been achieved with

the modified design of the flow field and increasing contact pressure between electrodes and interconnects.

Si-CNT hybrid structure as Li-ion battery anode material

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Increasing demand of secondary batteries in portable electronics, electric vehicles and electrical storage systems have triggered the research efforts in the field of Li-ion battery. Different materials and their composites are being investigated intensively in order to achieve higher capacity, longer life span and better capacity retention ability. Silicon is known to offer maximum theoretical specific capacity of 4200 mAh/g, but volume expansion and contraction during lithiation and delithiation process hinders its commercialization. Carbon nanotube is known for its high conductivity, excellent mechanical properties, large surface area and flexible nature that make it suitable to be used as nano-matrix. The CNT based nano-matrix helps in accommodating the volume change in Si due to their flexible nature and also increases the overall electrical conductivity of electrode. Combination of these effects eventually leads to better capacity retention and longer life span. This research deals with a simple method of obtaining a Si-CNT hybrid structure as Li-ion battery anode material. CNTs were functionalized using nitric acid in order to introduce COOH functional group in the sidewalls of CNTs which provides better binding with Si nanoparticles. Electrodes prepared with this manner were characterized using FTIR, Raman spectroscopy and XRD. Electrochemical characterizations were performed with different current densities. The electrode has shown good initial specific capacity of 663 mAh/g.

Understanding the role of graphene as buffer layer for stress mitigation in silicon as anode for Li-ion batteries

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Despite possessing higher Li-capacity and improved safety aspects, Si is yet not able to replace graphitic carbon as anode material for Li-ion batteries. This is primarily due to the huge volumetric expansion/contraction (~ 300 %) upon lithiation/delithiation of Si, which leads to severe stress developments and concomitant fracture/disintegration/loss of contact with current collector. It is believed that graphene sheets, possessing good flexibility and excellent fracture strength, may act as superior buffer material to accommodate the stresses arising from these dimensional changes of Si. Unfolding and folding of wrinkles in graphene is expected to be one of the mechanisms for stress relaxation. In order to develop better insight into the possible role of graphene as mechanical buffer, in this work we have used continuous Si thin films and Si/graphene multilayered composite thin films as model materials. The stress

developments in the presence and absence of few layer graphene (FLG) have been monitored *in-situ* using multi-beam optical stress sensor (MOSS) during electrochemical cycling against Li in a custom-made electrochemical cell. The FLG (~ 7 layers) films were deposited via chemical vapor deposition at 1000°C by decomposing methane gas on copper foil. Presence of wrinkles in the as-transferred graphene films were confirmed using SEM and AFM. Subsequently, Si was deposited via hot wire CVD on the FLG films transferred onto stiff 0.5 mm thick quartz substrate coated with 100 nm thick Ni film, acting as current collector. The results obtained with galvanostatic cycling and *in-situ* stress measurements, in light of cyclic stabilities, overall stress development and some finer features of the stress developments, for the bare Si and Si/graphene thin film electrodes will be compared and contrasted. Further analytical treatments will lead to a better understanding of the effect of the presence of graphene on the stress developments in Si during lithiation/delithiation.

In situ monitoring of stress development during electrochemical cycling of Sn thin film electrodes for Li-ion batteries

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Sn is a promising candidate to replace graphitic carbon as anode material for Li-ion batteries due to its lower cost, higherLi-capacity and improved safety aspects. However,Sn experiences huge volume changes during Li insertion/extraction, which generates significant stresses, resulting in cracking and loss of electrical contact with current collector. Against this backdrop, we report here results and understandings based on real-time (*in-situ*) monitoring of the stress evolution in Sn thin film electrodes (a simple and model configuration) by monitoring the substrate curvature using multi-beam optical sensor (MOSS) during electrochemical cycling against Li in custom-made electrochemical cell. It must be mentioned here that, even though various other research groups have earlier attempted to understand the stress development via mathematical modelling and minimize the stresses by designing innovative nano-architectures, to the best of our knowledge we have been the first to monitor the stresses experimentally and in real-time for such ductile metallic anode material. The experiments have been performed with Sn thin film electrodes (~ 200 nm thick), deposited via e-beam deposition, on thicker (~ 0.5 mm) and stiff quartz substrate coated with either Cu or Ni (~ 100 nm), acting as current collector. In addition to measuring the overall compressive stress developments during lithiation and release during delithiation, we focus on some of the finer features of the stress development, which involves the stress evolution during the first order phase transformations that take place during the Li-alloying reactions (i.e. formation of the various Sn-Li intermetallic). Our results reveal that unlike in the single phase (solid solution) regimes, evaluation of the stress response during the phase transformation regimes must go beyond simple elastic behaviour. Additionally, the influences of the current collectors (Cu and Ni) on the phase assemblage, electrochemical behavior and stress developments in Sn have also been critically evaluated.

Synthesis and characterization of sodium beta alumina for Na/S batteries

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Sodium beta-alumina (SBA) electro-ceramic can act as an efficient ion conducting solid electrolyte because of its fast ionic conductivity, which arises from its unique structure in which two-dimensional conduction planes partially occupied by Na^+-ions are separated by crystalline Al_2O_3 spinel blocks. For a material to serve as a solid electrolyte for sodium-sulfur battery, it should have fine grain structure, minimum porosity and high strength at the operational temperature of 300°C. Amongst the various processing routes available, sol-gel route, which is more energy efficient, offers better composition control and product homogeneity while leading to synthesis of β''-alumina in the purest form, was used so as to attain all the attributes of synthesizing 'perfect' solid electrolyte. Sodium beta-alumina (SBA) nano-powders were synthesized by the citrate sol-gel process, with the use of glycerin as an alternative raw material, instead of ethylene glycol. Thenano-powder thus obtained was compacted uniaxially with 100 MPa pressure and sintered between 1350°Cand1500°C for 3-10 h. The structure and morphology of the nano-powders will be characterized by X-ray diffraction (XRD) and scanning electron microscope (SEM) techniques, respectively. The dielectric constant and DC conductivity has been measured using Impedance Spectroscopy. The effects of CTAB on the citrate sol-gel process and the SBA formation will be investigated.

Poly(vinyl alcohol)/sulfosuccinic acid membrane based MEA for proton exchange membrane fuel cells

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A polymer electrolyte membrane serves as a barrier to avoid direct contact between the fuel and oxidants in proton exchange membrane fuel cells (PEMFCs). In the present study,new types of proton conducting membranes based on poly-vinyl alcohol (PVA) cross-linked with sulfosuccinic acid (SSA) were prepared using a cost-effective and simple procedure. PVA membranes were prepared with varying amounts of SSA (5-25 wt.%) and cross linked for different time periods in order to achieve desirable properties for fuel cell applications. The structures of cross-linked PVA-SSA membranes were characterized using Fourier transform infrared spectroscopy, and X-Ray diffraction. Swelling of membranes due to water is a major concern in fuel cells as it significantly affects the efficiency of the cell. The water uptake and compressibility of the membranes was investigated and their dependence on the cross linking time and SSA concentration was identified. From these studies, PVA-10SSA membrane wasselected for membrane electrode assembly (MEA) fabrication and tested in a H_2-O_2 fuel cell testing fixture. PVA-SSA polymer based MEAs were fabricated with different ionomer loadings in the catalyst layer. Highest power density was achieved with an ionomer loading of 0.05 mg/cm^2. The study revealed that low cost PVA-SSA based MEAs are cost effective for low power density applications and have potential commercial application.

Synthesis and pore distribution studies of monolithic gas diffusion media for PEMFC by varying solvent type

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Proton exchange membrane fuel cells (PEMFC) are low temperature based clean energy conversion devices that aid in addressing'energy problems'. One of the components of a PEMFC is the gas diffusion medium (GDM) that transports gas and water molecules when the fuel cell is in operation. Mass transport losses through the GDM can significantly impact the performance of a fuel cell. Commercially available starting materials, such as carbon paper and carbon cloth, restrict the overall design of the GDM to work within the constraints of their structures. The present work focuses on designing a GDM starting from Carbon black, Graphite, and Teflon, in the powder form, which increases flexibility of design. The GDMs are tailored to exhibit Macro and Meso pore structure and to further possess a funnel like structure for easy seepage of water. The pore size, uniformity and pore distribution were observed in case of five solvents – IsoPropyl Alcohol(IPA), Glycerol, PEG-200, Water and mixture of IPA+Glycerol. The GDMs prepared have been characterized using SEM and Mercury Intrusion Porosimetry. Pore distribution, sizes and composition have been examined using SEM images and composition maps on a comparative basis. Optimization of conductivity, hydrophobicity and water transport have been carried out. The results obtained will be presented, discussed and compared. The advantage of a particular solvent is recognized as a part of this approach.

GLASS AND ADVANCED CERAMICS

Invited talks
Session 1: Nov 13, 2014: [9:00 - 11:00]

Challenges in fabrication of LTCC devices and their applications

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Low Temperature Co-fired Ceramic (LTCC) is a glass-ceramic, multilayer circuit and electronic package preparation process that has advantages over the other, more conventional electronic packaging technologies. This process allows very high interconnect density due to multilayering capability, can integrate passive components, handle very large numbers of Input –output (I/O) connections and at the same time has a capability to cater to high frequency (HF) circuit needs. The flexible manufacturing process of LTCC also allows its use as structural, supporting material as well as in preparing sensors and actuators themselves. The LTCC technology does, however, have some limitations, such as, high shrinkage tolerance, non-flexibility and low thermal conductivity. In spite of these disadvantages, LTCC holds great promise in development of total integrated solutions inclusive of wireless front ends, sensors, actuators and digital circuits finding applications in consumer medical, space and military electronic systems alike. Apart from the technological aspects, the talk also highlights materials limitations and current development directions.

Session 2: Nov 15, 2014: [14:00 - 16:00]

Ceramic materials: High tech applications

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High performance structural ceramics is a high strength, high hardness, a new type of ceramic material with high temperature resistance, corrosion resistance, wear and chemical properties of wear-resistant stable, in aerospace, mechanical, electronic, chemical industry, communications, energy, biomedical, environmental engineering and other fields are widely used. Due to high thermo oxidative stability and corrosion resistance the ceramic materials have an edge over conventional structural materials like metals and polymers. But, the brittle & intractable nature of ceramics restricts their application as engineering materials specially for making complicated shaped components. But the emergence of pre-ceramic polymers creates entirely new possibilities of solving the problems of obtaining a new generation ceramic materials and their fabrication to get complex design components. Pre-ceramic polymers – the precursors of ceramic materials- are products with an inorganic chain skeletal structure and organic side groupings. As a result of pyrolysis the inorganic skeletal changes into a ceramic material and organic group are removed as gaseous products. These materials are in general, fusible, soluble and capable of being shaped in either the molten or dissolved state. After the pyrolysis the shape, size & dimensions of the ceramic shaped articles thus obtained remained intact. Nowadays, silicon carbide composite components are used

in exhaust cones of advanced gas turbine engines replacing Fe-Ni metal alloy components and silcon carbide and boron carbide based composite armour plates were used to defeat high energy ammunition. But such technology is restricted to very few advanced countries like, Japan, USA German & France etc. We are working on the development of preceramic polymer for making silicon carbide based CMCs and also use of different ceramic material for armour application. This presentation will give an overview of work carried out in DMSRDE on ceramic material to be used for different high tech applications in defence applications.

Novel approaches in processing of foam materials for aerospace applications

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Carbon and ceramic foams are finding applications in thermal protection systems and light weight structural materials in aerospace and defense applications. The carbon foams are generally prepared either from pitches or from synthetic organic polymers derived from petroleum. The use of naturally renewable materials of agricultural origin as feed stock for the prep ration of advanced materials is getting importance as the fossil fuel based precursors are depleting. The present talk focuses on the preparation of carbon foams from sucrose, an abundant agricultural product. The methods developed for the foaming and setting of sucrose and properties of carbon foam etc will be discussed. The methods of improving the foam property using reinforcing additives such as carbon particles, short carbon fibers and carbon nano tubes will be presented. The talk also covers the use of sucrose in the preparation of light weight ceramic foams. The emulsion based methods for the preparation of ceramic foams using vegetable oils as pore template will also be presented.

Contributory papers

Session 1: Nov 13, 2014: [09.00 - 11.00]

Synthesis and characterization of ultrahigh temperature ceramics for hypersonic applications

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To enhance the safety, maneuverability and lowering the maintenance costs of hypersonic reentry vehicles, sharp wing leading edges (WLE) have been proposed instead of blunt shaped wings. During reentry into earth's atmosphere very high temperatures are generated on the WLE, which is inversely proportional to the square root of the radius of curvature of WLE. Conventional thermal protection systems cannot withstand such high temperature and the harsh environment. Borides, carbides and nitrides of Zr, Ti and Hf with melting temperatures of > 3000 ˚C are referred as ultrahigh temperature ceramics (UHTCs) are considered potential candidates for hypersonic applications. TiB_2 and ZrB_2 exhibit excellent properties such as high hardness (> 25 GPa), high elastic modulus (> 450 GPa), low density, low

electrical resistivity (10-30 x10^{-6} Ω cm) and high thermal conductivity (60-140 W/m K). These properties are vital for the abovementioned applications. However, the fracture toughness, oxidation and ablation resistance of these materials needs to be improved. In the present study, elemental powders of Zr and Ti were ball milled and reaction spark plasma sintered to produce 25ZrB_2-75TiB_2, 50ZrB_2-50TiB_2, 75ZrB_2-25TiB_2 composites by vol.%. Composites have also been synthesized by spark plasma sintering of ZrB_2 and TiB_2 powder mixtures. The microstructure and phase analysis have been determined using SEM and XRD. The effect of TiB_2 on thehardness, indentation fracture toughness, oxidation resistance and the coefficient of thermal expansion has been studied. The effect of reaction sintering on the microstructure and properties is presented.

Size effect studies on structural and electrical properties of PMN-PT ceramics

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A single phase, perovskite lead magnesium niobate- lead titanate (0.65PMN-0.35PT) powder has been successfully derived from mixture of oxides through columbite precursor route. Two milling methods are employed in the present study for comparison; conventional ball milling (CBM) and high energy ball milling (HEBM). The synthesized powders are then treated with HEBM for different duration 5, 10 and 15 h. The HEBM process reduces the average particle size to less than 100 nm. The milled powders were pressed into disk-shaped pellets with diameter 12 mm and thickness 1.2 mm at 100 MPa. The pellets were sintered at 1250˚C for 2 h in a covered alumina crucible. The sintering behavior, dielectric and piezoelectric properties of the ceramics were explored . For electrical measurements , silver paste is applied on both sides of specimen as electrodes and cured at 650˚C. The specimen is poled in silicon oil bath at 70˚C by applying a field of 3 kV/mm for 30 min. Compared to conventional ball milling, HEBM suppressed the formation of unwanted pyrochlore phases and furthermore resulted in finer grain microstructures with better density, dielectric and piezoelectric properties. Piezoelectric charge coefficient (d_{33}), piezoelectric coupling coefficient (k_p) and dielectric constant (ε) of modified PMN-PT ceramics having different grain sizes have been studied. Temperature dependent dielectric studies are also carried out to investigate the effect of grain size on Curie temperature and dielectric properties.The hysteresis loop of CBM and HEBM processed ceramics are compared and studied. The achieved properties like piezoelectric charge coefficient, 700 pC/N and dielectric constant, 4000 are found to be higher than those available in the literatures. These PMN-PT materials are tested as sensors for structural health monitoring applications and found to have better sensitivity.

Thermogravimetric analysis and kinetic study of formation of lithium titanate by solid state route

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The kinetics of formation of lithium titanate from the solid state reaction of lithium carbonate and titanium oxide was studied using non-isothermal thermogravimetric technique. Thermogravimetric data for the reaction of lithium carbonate and titanium oxide was obtained at various heating rates. The

methods such as Flynn-Wall-Ozawa and Kissinger-Akahira-Sunose were used to estimate the kinetic parameters from the obtained thermogravimetric data. The average activation energy for the formation of lithium titanate by solid state route was found to be 243kJ/mol.K.The reaction mechanism was determined by the method given by Malek. It was found that the three dimensional diffusion model best describes the reaction kinetics. A kinetic equation describing the reaction is proposed and reaction mechanism is discussed.

Structural, thermal & magnetic characterization of soft-magnetic amorphous $Fe_{78}Si_9B_{13}$ melt spun ribbons

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Wide and continuous amorphous ribbons of $Fe_{78}Si_9B_{13}$ alloy with good surface quality was prepared using planar flow melt spinning technique. The as melt spun ribbons were annealed in high vacuum furnace at different temperatures. The as melt spun and annealed ribbons were characterized using XRD, DTA, VSM and Coercimeter. The thickness of the ribbon was found to be uniform along the length of the ribbon whereas the width decreases along the length of the ribbon due to the solidification of the alloy on the edges of the slit bottom. The as spun ribbon crystallizes in two stages namely Fe_3Si phase and Fe_2B phases. The Fe_3Si phase decreases the saturation magnetization and Fe_2B phase increases the magnetization. The coercivity decreases on annealing which may be attributed to Fe_3Si nanocrystalline phase formation which averages out magneto crystalline anisotropy. The coercivity at higher annealing temperature is very high due to the higher magnetic hardness of Fe_2B phase formation.

Effect of solvent on synthesis of $K_{0.5}Na_{0.5}NbO_3$ (KNN) via modified solid state route

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Alkali Niobates provide an environmentally friendly alternative to PZT (lead zirconate titanate , $Pb(Zr,Ti)O_3$) which is widely used owing to its high piezoelectric properties. Most of the recent studies and research are pertaining to scalability of production of alkali niobates at industrial level which can be used in electronic devices such as high frequency transducers , sensors etc. In our study, we havedone non-stoichiometric synthesis of pure phase KNN (**$K_{0.5}Na_{0.5}NbO_3$)**via solid state route with two step calcination using potassium acetate , sodium acetate and niobium pentaoxide as precursors. Comparative study of solvent used namely water and methanol is carefully investigated. Milling time and calcination time and temperature is optimized to avoid formation of secondary phases of KNN. Obtained powder samples both by water and methanol as solvent are characterized by X-ray diffraction (XRD), dynamic light scattering (DLS) and quantitative SEM-EDS analysis before piezoelectric measurements. The first result of comparison is present here. Special focus of our study is to attain high yield powder with suitable structural and dielectric properties which require low calcination time and temperature.

Effect of W-rich phase on ablation behavior of pressure-less sintered ZrB_2– SiCvbased ultra high temperature ceramic composites

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The ablation resistance of pressureless sintered ZrB_2-SiC (10-30 vol.%) composites has been investigated. The composites have been sintered at 2000 °C in argon atmosphere using B_4C (3 wt. %) and C (2 wt. %) as sintering additives. The ablation experiments have been carried out by exposing specimen surfaces to neutral oxyacetylene flame for 10 minutes, resulting in a maximum temperature of 2200 °C. The damage due to ablation has been determined in terms of change in mass, Young's modulus, hardness and indentation fracture toughness. Results show that presence of SiC appears to aid in densification by increasing WC content by erosion of milling media, and inhibiting matrix grain growth. Both SiC and WC appear to aid in reduction of oxide impurities. In addition, the impurities enriched in W, Fe and Co obtained from milling media are found to be segregated at ZrB_2 grain boundaries and appear to assist in densification. The density and mechanical properties of the composites are found to improve with increase in SiC content. The surface oxidation products are found as ZrO_2, SiO_2 and $ZrSiO_4$, by examinations using X-ray diffraction, scanning electron microscopy and energy dispersive X-ray analyses. The W–rich phase present at the ZrB_2 grain boundaries or ZrB_2–SiC interfaces gets oxidized during ablation. The rapid volatilization of WO_3 results in higher rate of degradation of the ZSBC–30 composite compared to that of the ZSBC–20 composite.Comparison of the estimated results indicate that ZrB_2-20 vol% SiC exhibits superior ablation resistance compared to the composites containing 10 and 30 vol.% SiC.

Effect of yttria distribution on wear properties of Y-TZPceramics

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Recently, yttria stabilized tetragonal zirconia (Y-TZP) ceramics are known to be one of the potential materials for the structural application i.e cutting tools, femoral head in total heap replacement. Till to date, all research in this class of ceramics is mostly concentrated on understanding the influence of dopant content and grain size on transformation toughening. The present understandings have focused on the role of yttria distribution on transformation toughening and wear properties of Y-TZP ceramics. In our experiments, 8 mol% yttria stabilized zirconia powders were mixed with varying amount of Y-free monoclinic zirconia powders and subsequently sintered at 1450 °C for 2 hours. The microstructural-mechanical characterizations were carried out using XRD, EPMA, SEM and Vicker's Indentation. Tribological behavior was studied using fretting wear test against steel at load of 10N with duration of 100000 cycles. As results, varying yttria distribution and uniform microstructure having grain size around 0.2 μm were obtained in the sintered ceramics. While, fracture toughness, coefficient of friction and wear volume of ceramics were recorded 10.5± 0.2 $MPam^{1/2}$, 0.5± 0.12 and $3.5\times10^5 \mu m^3$, respectively. For the first time, the present research has demonstrated that the mechanical property, in particular the

indentation toughness and wear properties of Y-TZP can be improved, when sintered at 1450 ^{o}C for 2 hours. Finally, a detailed study was investigated on the influence of stress induced transformation and associated microstructural variables on the friction and wear mechanisms of Y-TZP ceramics against steel.

Modified aqueous combustion synthesis method for the preparation of ceria powder

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The ultra fine ceria powder was prepared by aqueous combustion synthesis method. The mixed fuels were used in the combustion. The fuel ratios are varied to get the adequate properties of the powder. The effects of fuel-to-nitratemolarratio on the combustion behaviour and resulting powder yield are studied.The combustion reactions become less vigorous and smouldering in nature with the decrease in fuel to nitrate ratio.The synthesis reaction with stoichiometric ratio and a fuel deficient ratio produces a fluffy white and yellow powder. It is evident from the XRD, that the crystallite size of the ceria powders increases with an increase in fuel to nitrate ratio. The morphology of the particles was determined byscanning electron microscopy.

Session 2: Nov 15, 2014: [14:00 - 16:00]

Preparation and properties of SiC_p/Al_2O_3 composites by reaction sintering

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SiC_p/Al_2O_3 composite gained interest among industries due to its excellent mechanical and tribological behavior, both in room temperature as well as at elevated temperature. Owing to inherent brittleness and lower strength, more emphasis has been given on improving the fracture toughness and other mechanical properties of such ceramic composites. Preparation of SiC_p/Al_2O_3 composite by hot pressing and hot isostatic pressing in inert atmosphere reports improved density, strength and toughness. However, this process is constrained to produce components with simple geometrical shapes. Highly dense composites can also be prepared by pressureless sintering under inert atmosphere. Utilizing hot pressing routes and inert atmosphere are quite expensive and complex. The objective of this work is to prepare SiC_p/Al_2O_3 composite in normal atmosphere with two stage pressureless sintering methodology and to investigate its properties. Initial powders are blended with various contents of SiC (5 vol. % and 10 vol. %) in Al_2O_3 and compacted to pellets. The green compacts are pre-sintered at 1300-1400 °C for two hours in normal atmosphere. During this process, the surface of SiC gets oxidized forming SiO. The pre-sintered pellets are sintered at 1600 °C in normal atmosphere for four hours. The surface SiO reacts with Al_2O_3 and forms mullite, which makes the process a reaction sintering route. Mullite is the only stable intermediate phase in the alumina-silica system at atmospheric pressure. Thus the resultant composite has SiC (size reduced) surrounded by mullite phase, which is distributed in continuous alumina matrix. The prepared composite is characterized using X-ray diffraction and scanning electron microscopy. The density

and fracture toughness of composites prepared by reaction sintering route are compared with monolithic alumina and discussed.

Development of $MgO-MgFe_2O_4$ bulk ceramic 'nanocomposites' via solid state precipitation

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The present research is aimed at designing a commercially viable processing route for developing bulk MgO-based oxide/oxide particulate 'nanocomposites'. This route, which is based on basic physical metallurgical principles of *in-situ* precipitation of second phase particles during aging of bulk super-saturated ceramic solid solutions avoids the use of nanosized starting powders, is devoid of the challenges associated with minimization of particle coarsening during densification and does not necessitate the use of any advanced sintering techniques. Here we show that it is possible to dissolve up to 7 wt.% Fe_2O_3in MgOto form bulk solid solutions during pressureless sintering in air at 1650°C(solution treatment) followed by air quenching/fast cooling. Subsequent aging treatments of the bulk solid solutions of MgO containing Fe^{3+} at different lower temperatures (800°C - 1000°C) and for different durations (up to 40 h) led to the precipitation of $MgFe_2O_4$ spinels as second phase particles uniformly within the matrix grains, as well as along the grain boundaries. The solution treatment and aging conditions were selected based on published $MgO-Fe_2O_3$ phase diagram. The aging conditions have been optimized for retaining the size of the inter/intra-granular second phase particles within the nanosized regimes. Preliminary investigation of the mechanical properties, using Vickers' Indentation, have revealed that the bulkcomposites containing the precipitated second phase $MgFe_2O_4$nanoparticles possess superior hardness (up to ~ 60%) and indentation toughness (up to ~ 35%), as compared to phase pure bulk MgO. Furthermore, abrasive wear tests have revealed that these 'nanocomposites' possess improved wear resistance, as compared to phase pure bulk MgO.

High temperature oxidation studies of TaC based ultra-high temperature ceramic composites

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Space vehicle experiences very high temperature due to aerodynamic heating and to assess its safety during landing is a big issue. Ultra-High Temperature Ceramics (UHTCs) are promising material due to potential application in thermal protection system (TPS) in extreme thermal and chemical environments. Here, we report the experimental investigations of TaC based ultra high temperature ceramic (UHTC) composites processed via spark plasma sintering (SPS) at 1850 ºC in vacuum (at 40 MPa) and holding for 5 minutes at maximum temperature. TaCis reinforced with 15 vol. % of SiC, CNT and mixture of both to improve the densification, mechanical properties and thermal conductivity of the material. In order to investigate the oxidation behavior, thermal analysis on these composites were performed from ambient temperature to 500, 700, 900, 1100, 1300 and 1500 ºC with the rate of 20 ºC/min in oxygen atmosphere

followed by isothermal holding at each temperature for 30 min. and weight gain of ~12.9-16.1% for composites at 1500 °C due to the formation oxide layers. The addition of SiC and CNT is found to suppress the formation of oxide layer, which confirmed by recording the change in weight at each temperature.During the non-isothermal studies, the formation and transformation of intermediate crystalline phases were also observed. Activation energy of oxidation has been calculated for all the compositions and it is found that TaC with 15 vol. % SiC composite can use as a potential oxidation resistance UHTC.

Synthesis and characterization of precursor derived titanium disilicide based ceramic composites

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High temperature stability (upto 1150 K), high melting point and low resistivity (15-35 μΩ cm) are the explicit features of titanium disilicide ($TiSi_2$), making it an ideal candidate for a number of applications. $TiSi_2$ thin films are widely used in the semiconductor industry as a source drain, gate contact and also as a contact material in thermoelectric devices to improve their efficiency. $TiSi_2$ primarilyexists as two polymorphs - C49 (base-centered orthorhombic) and C54 (face-centered orthorhombic). The high temperature polymorph C54 exhibits lower resistivity than C49 and is preferred in integrated circuits. The field emission properties of $TiSi_2$ is also being widely researched and employed in fabrication of microelectronic devices.The present work deals with the synthesis and characterization of $TiSi_2$ based precursor derived ceramics. $TiSi_2$ powder was finely dispersed in a solution of polymethylhydrosiloxane (PHMS) and was then ceramized in the furnace at 900 ^{0}C. The ceramized powder was then ball milled for 10 h in toluene medium. The resulting mixture was characterized using X-ray diffraction (XRD) for phase analysis and particle size was determined using a particle size analyzer. The mixture was then spark plasma sintered (SPS) at 1000 ^{0}C for 30 minutes at a pressure of 50 MPa. The SPS pellet was annealed at 900 ^{0}C for one hour and then water quenched. Various characterization techniques such as XRD, Raman spectroscopy, scanning electron microscopy, electrical conductivity measurements were carried to completely analyze the composites.

Preparation of carbon fiber reinforced silicon nitride composites by reaction bonding process

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Silicon nitride ceramics has been used for structural applications and some engine parts due to their high strength, good corrosion and oxidation resistance and better thermal shock resistance. However, the mechanical reliability of this ceramics is low due to low fracture toughness. Introducing second phase materials in the form of whiskers, fibers and particulates will improve the toughness of the silicon nitride ceramics. There are different routes to form silicon nitride composites and each method has its own advantages and disadvantages. In the present study, the reaction bonding process is used, which offers low temperature processing using low cost raw material and near net shape product. Raw silicon powder is ball milled for 10 h and mixed with pre-dispersed carbon fibers. The slurry is mechanically agitated to get

homogeneous dispersion. The mixed slurry is dried and then cold compacted at 150 MPa. The samples with different amount of carbon fibers are nitrided by using N_2-H_2 gas mixture at 1400 °C. The temperature and gas mixtures are chosen based on the previous studies. X-ray diffraction analysis shows that the composite is consisting of both α and β-silicon nitride. Scanning electron microscopy analysisreveals the homogeneous distribution of carbon fibers and the whisker like formation of silicon nitride. Using transmission electron microscopy detailed internal microstructure and interfacial structure between matrix and reinforcement are analyzed.

Prediction and synthesis of Fe base amorphous/glass forming compositions

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Fe based alloys have low glass forming ability (GFA) and most of the glass forming compositions in Fe based systems are complex in nature. Several models were proposed to estimate GFA based on nucleation kinetics. However, these models are not predictive in nature. They require an alloy to be made first and evaluate thermal analysis parameters. In-order to predict the GFA without conducting experiments, thermodynamic parameters and molecular dynamics are being used. In the present work, a new parameter P_{HSS} (i.e. product of enthalpy of chemical mixing, mismatch entropy and configurational entropy) has been used to identify the glass forming compositions Fe base systems. Results indicated that this parameter has very good correlation with GFA of Fe base alloy systems. In present study, P_{HSS} model parameter is validated for binary (Fe-B), ternary (Fe-B-Zr) and quaternary (Fe-Cr-B-Zr) systems. Enthalpy of chemical mixing was calculated using Takeuchi's approach from binary equi-atomic mixing enthalpies. Mismatch entropy was calculated using Mansoori's approach normalized by Boltzman's constant. Configurational entropy was estimated from classical statistical mechanics normalized by gas constant R. To verify model predictions, selected alloys were prepared using melt spinning as well as by mechanical alloying route. It was observed from rapid solidification processing (RSP) and mechanical alloying studies that all alloys with P_{HSS} between -0.55 kJ/mol to -6.00 kJ/mol would form glass in the Fe base system. Melt spin ribbons and mechanically alloyed powders were characterized using x-ray diffraction and transmission electron microscopy. Powders of identified glass forming compositions systems in ternary and quaternary were synthesized by mechanical alloying route. These amorphous powders were sintered using Spark plasma sintering technique at different temperatures. Among sintered samples, one composition from Fe-Cr-Zr-B system has shown very high hardness of 1200 VHN and very high corrosion resistance equivalent to cast super duplex stainless steel.

Designing composition with high glass forming ability in Zr-Ti-Fe-Cu-Al system through thermodynamic modeling

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High entropy alloys (HEA's) and Metallic glasses (MG's) are the intriguing categories of materials owing to their structural and functional properties. In order to design these materials for technological applications, a knowledge of phase stability of high entropy phases as a function of composition and temperature is

essential. Though steady progress has been made in CALPHAD method to describe the phase formation in multicomponent systems, reliable methodologies are yet to be developed in estimating Gibbs free energy of high entropy phases in concentrated alloy systems thereby hindering discovery of novel glass formers. High configurational entropy obtained through equiatomic concentration of elements obviates competing ordered intermetallic phases and stabilizes liquid like structure. Hence this method of addition of alloying elements in equiatomic levels can lead to bulk metallic glasses provided that the thermodynamic and topological conditions for glass formation are satisfied.In this paper, a new Zr-Ti-Fe-Cu-Al high entropy metallic glass composition is designed near deep eutectic regions of the sub quaternary systems. The quinary compositions are modeled using P_{HSS} parameter, which is the product of enthalpy of chemical mixing (ΔH_{mix}) calculated through Miedema's model, mismatch entropy ($\Delta S_o/k_B$) obtained through Mansoori's approach for hard spheres, and configurational entropy ($\Delta S_c/R$). Further, the glassy nature of the alloy is verified using different characterization techniques and the mechanism of amorphization is discussed.

Transparent polycrystalline ceramics

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Polycrystalline Nd: YAG Ceramics were fabricated by mixing the powders of Al_2O_3 (99.9%), Y_2O_3 (99.9%) and Nd_2O_3 (99.7%) in stoichiometric proportion. Two routes were followed for further processing. In first route wet ball milling was carried out with addition of TEOS and Ethanol for 12h at 250 rpm. Milled powder was compacted to form pellets. To lower down sintering temperature addition of sintering aids such as CaO, MgO, SiO_2, ZnO, TiO_2 and (2%Al_2O_3 +6%Y_2O_3) was made (1, 5 and 10 Wt.%). Sintering was done at 1530 ^{0}C for 10h in $MoSiO_2$ Sintering furnace. In second route Cold Isostatic pressing was carried at 103.42 MPa pressure and the pellets were formed. Sintering was done at 1650 ^{0}C for 20h.These pellets were characterized for density, hardness dielectric properties, XRD, SEM were measured.

Synthesis, consolidation and densification of aluminium titanate

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Various ceramic materials have been widely used in industries due their insulating (thermal) properties. Aluminium titanate (Al_2TiO_5) is one among them. Properties of aluminium titanate like good thermal shock resistance due to very low thermal expansion and poor wettability with molten metals can be exploited well in industries like foundry technology and in metallurgical melting. Micro-cracks are generated in Al_2TiO_5 during sintering due to anisotropy in thermal expansion. This results in the reduction of mechanical strength. Producing a crack-free aluminium titanate itself is a challenge. This can be solved either by adding some additives or by forming solid solutions with magnesia and silica. It is noticed from the work reported so far that MgO and Fe_2O_3 addition is effective in controlling the grain growth. In the present study aluminium titanate was produced from the respective oxides (alumina and titania). Both the oxides were taken in 1:1 ratio. This mixture along with MgO or Fe_2O_3 (at various wt. %) was milled for 24 hrs with ethanol and then dried in oven at 110°C for 24 hrs. The dried powders were calcined at various temperatures (900°C to 1300°C) and consolidated by uniaxial pressing with a load of 2.5 tons. The pellet was reactive sintered for 2 hrs at different temperatures from 1300 to 1600°C with a heating rate of5°C/min. It was confirmed from the XRD data that the as milled powders contained a major phase of

Al_2TiO_5 and traces of Ti_2O_3 and $Al_2Ti_7O_{15}$. The aluminium titanate with 5% MgO and 5% Fe_2O_3, sintered and fractured surface was examined under SEM. The average grain size measured was in the range of 1-5µm. Homogeneous distribution of MgO and Fe_2O_3 was confirmed by elemental mapping analysis. Efforts are being taken to vary the additive amount and to study their influence on grain growth control.

Synthesis of SiC whiskers from the mixture of coconut shell and polycarbosilane

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The SiC whiskers, because of their high strength and stiffness, are utilized as reinforcement in metal and ceramic matrix composites. In this study, the SiC whiskers are produced from a mixture of coconut shell and Polycarbosilane. The coconut shell was cut into small pieces and dispersed into polycarbosilane toluene solution and allowed to dry at room temperature. The mixture was taken in a graphite boat and pyrolyzed at 1400 °C under argon gas atmosphere. The synthesized SiC whiskers have been characterized by X-ray diffraction, scanning electron microscopy and transmission electron microscopy. The whiskers are formed predominantly on the surface layer of coconut shell and there is very minimal whisker growth at inner regions, which indicates that the whisker growth occurred by gas phase reactions. The whisker yield from this mixed raw material is high compared to that from coconut shell alone. The more whisker yield is attributed to the formation of highly reactive SiOC structure from polycarbosilane and the adsorption capacity and carbon activity from coconut shell. Transmission electron microscopic analysis shows that the nanowires have β-SiC structure, which grows along <111> direction. The SiC whiskers have striped morphology, comprising of defects like twins and stacking faults.

HEAT TREATMENT AND SURFACE ENGINEERING

Invited talks

Session 1: Nov 12, 2014: [13.30 - 15.30]

Overview of quenching: Wetting kinetics, kinematics and heat transfer characteristics of liquid quenchants

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In industrial heat treatment of steels, quenching is an important step that affects the evolution of microstructure, properties and the quality of components. Quenching is no longer an art but a very complex field involving science, engineering, technology and mathematical computation. During quenching, a wetting front forms and its movement on the hot metal surface results in simultaneous occurrence of all the three stages of quenching. The localized cooling of the component controls thermal transport and evolution of microstructure during quenching. The phenomenon of wetting during quenching has a significant influence on the rate of heat transfer and temperature distribution and thus controls the microstructure and mechanical properties of components. The simulation of quench hardening process to predict the formation and/or distribution of microstructures and residual stresses requires the use of reliable boundary heat transfer coefficients. The use of non-linear inverse heat conduction problem (IHCP) coupled with austenite decomposition enables the estimation of heat flux transients under plant conditions. The simultaneous assessment of spatially dependent heat flux transients and wetting kinematics is needed for better understanding of phenomenon of quenching. The relevance of wetting kinetics, kinematics, temporal and the spatial variation of heat flux transients to industrial heat treatment of components are highlighted.

Session 3: Nov 13, 2014: [09.00 - 11.00]

Solution precursor plasma spraying: Opening vistas for exciting research and niche applications

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The solution precursor plasma spray (SPSS) technique has been the subject of considerable research interest in recent times in acknowledgment of the fact that this thermal spray variant offers significant advantages in terms of eliminating expensive powder-feedstock, permitting better control over coating chemistry and yielding interesting features like vertical cracks, nano-sized pore structure, fine splats etc. Since its advent, the SPPS technique has been mostly investigated for deposition of yttria stabilized zirconia (YSZ) coatings for thermal barrier applications and, to a limited extent, for depositing other

ceramic coatings such as TiO_2 and Al_2O_3. Studies in the authors' laboratory have explored the possibility of utilizing the SPPS process for developing various functional coatings like $ZnFe_2O_4$, LSM, metal-doped ZnO etc. Illustrative examples of the large variety coatings that can be realized by adopting SPPS route will be presented, along with some recent results that highlight the significant role. The presentation also covers an innovative approach developed to deposit functional coatings with unique microstructures through simultaneous injection of powder and solution feedstock. The above technique offers remedy to the inherent low productivity of solution based process as well as bears promise to yield more durable coatings than conventional powder based techniques.

Contributory papers

Session 1: Nov 12, 2014: [13.30 - 15.30]

Characterization of sticky scale of HR steel containing high silicon in compact strip production (CSP) process

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CSP is an advanced manufacturing technology which produces thin hot strip using a short production line. The thin slab (55-65 mm) is first descaled, sheared to the desired length at 1000-1025ºC and then transported to the tunnel furnace set typically between 1100-1150ºC where it is soaked for 10-20 minutes. The thin slab is then descaled at high pressure before entering into 4 hi six strand tandem mill. The repeated customer complaint as well as internal diversion of sticky scale especially on the bottom surface and presence of Si on SEM EDS analysis at the defect region necessitated the root cause investigation of its formation. In tunnel furnace as well during hot rolling, some adherent oxides, known as scales, is formed on the strip surface. On silicon bearing steels, Fe_2SiO_4 (fayalite) layer is formed in the scale and causes eutectic transformation between FeO (wustite) during reheating / tunnel furnace at 1100 to 1200ºC. The adhesion of Fe_2SiO_4 on steel at high temperature is greater than FeO, therefore the $FeSiO_4$ might strongly adhere to the substrate steel and is not detached by the descaling/ roughing process. Scanning Electron Microscopy, EDS analysis and electron X-ray Diffraction techniques (XRD) have been used to characterize the sticky scale. The physical properties of oxides at high temperature and its influence on surface quality after rolling are discussed in silicon containing steels for better understanding of behavior of modern steels during hot rolling. Based on the analysis, inter-strand descaler has been introduced between the first and second strand to counter this surface problem.

Heat treatment of aluminum foil

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Aluminum and its alloys are used extensively for a wide range of applications in the aerospace, automotive, architectural, electronic and packaging industries. The versatility of aluminum in these fields is due to its specific properties such as low density, high ductility, good barrier properties, and strength when

it is alloyed. Aluminum sheet and foil are widely used products of the aluminum alloys and they are manufactured by casting and rolling process in the rolling mill. While cold rolling is performed grains get elongated and set up internal stresses and strain. These changes create resistance to further deformation. The cold-rolled sheet or foil is said to become "work-hardened." As the work hardening increases, it takes more power to roll the sheet thinner. Beyond a certain degree of hardness, the metal may crack if it is rolled again. This imposes practical limitation on the amount of cold rolled thickness reduction that can be achieved in an uninterrupted series of passes. To remove work hardening and to give require temper, heat treatment is carried out. One of the steps in the manufacturing of aluminum foil is annealing which is used to impart ductility to the foil and to remove lubricants from the surface of the foil and is done in the annealing furnace at temperature around 200 ^{0}C to 550 ^{0}C depending on the gauge thickness and softness requirement. In case of light gauge, aluminum foil is rolled in two layers. During this process the inner matte side and shiny outer side are created. In order to insure that the two sides are not glued together, they are moistened with the separating agent that must be diffused before using the foil and it is done by heating for some time.

Effect of thermal and thermo-mechanical processing parameters on the microstructure and properties of the API steel

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The properties of API steels can be remarkably improved through the proper selection of a suitable alloy composition and an appropriate thermo-mechanical processing (TMP) route. Present investigation aims to study the effect of thermal and thermo-mechanical processing simulation on the evolution of microstructures and properties of the API X60 grade steel at different cooling rates. The experiment consists of heating the specimens to austenizing temperature, isothermal holding at these high temperatures and then cooling at various cooling rates. It has been observed that the final microstructures and hardness values are greatly influenced by the cooling rates as microstructural constituents change and average ferrite grain size decreases with increase in cooling rate. In another set of experiment, specimens have been hot deformed in austenizing temperature with strains of 0.5 with a constant strain rate of 1 s^{-1}. It has been observed that faster cooling rate in free cooled deformed specimen has resulted in finer grain ferrite in comparison to the specimen cooled linearly at a rate of 1^{0}C/s.During experimental campaigns, 70% warm rolled specimens were annealed at two different temperatures of 750 and 850˚C, respectively with varying annealing time. The specimens were allowed to free cool after annealing for microstructure and properties evaluation. It was observed that the volume fraction of recrystallisation increases with increase in annealing hold time for warm rolled specimens annealed at 750˚C. It was also observed that annealing temperature of 850˚C is high enough for the occurrence of recrystallisation and ferrite grain size increases with increase in annealing hold time. It is also observed that hardness values are also influenced by annealing temperature and time.

Microstructure and mechanical properties of Cr-Mo-V based low alloy steel after tempering and isothermal ageing

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Cr-Mo-V based low alloy steels are widely used in thermal power plants because of their ability to withstand the elevated temperature and high pressure under continuous loading. With ferrite-pearlite and ferrite-bainite microstructures, these steels find their applications in boiler tubes, pressure vessels and steam turbines. Being cost effective is one of the important factors which draw attention of many industrialists instead on highly alloyed steels with austenitic matrix. In this project the microstructure and mechanical properties degradation of such a low alloy steel have been analyzed. Investigated material was in service for 25 years. During its service life, it had been exposed to high temperature and pressure resulting in properties to undergo changes. To figure out these evident changes, sample was solutionized and tempered for different ageing times under laboratory conditions. Changes in the morphology of the steel microstructure such as pearlite decomposition, carbide precipitation and spheroidisation etc. can be observed. Due to its coherent relation with microstructure, mechanical properties are also found to have undergone variations. In order to investigate the behavior of this steel after its exposition to different environments, hardness and tensile tests were conducted. Microstructural evolution of the sample was observed using SEM. TEM analysis was done to figure out the nature of carbides formed during experimentation. Experiments reveal the improvement in mechanical properties of the steel in the initial hours of its service due to the phenomenon of secondary precipitation. But, after reaching its peak condition (over-ageing) deterioration in the properties started to be noticed.

Study on inconsistency in mechanical properties in wheel hub forgings of military aircraft

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Foundry and Forge Division of Hindustan Aeronautics Limited, Bangalore manufactures forgings, castings, seamless rolled rings and metalloceramic brake pads for the various Indian military programmes. To name one such product, the division manufactures different types of Hub forgings for a number of internal projects fitted on the military helicopters or aircrafts. Wheel hubs used in helicopters and aircraft are made up of Al-Cu-Mg-Si-Mn (L-77) alloy which is heat treated to achieve desired high strength. The hub forgings are complex in geometry with ribs for reinforcement to withstand dynamic and impact load conditions. The criticality in geometry necessitated the qualification of forgings through checking mechanical properties at different directions in accordance with the design requirement. During the production of hub forgings, inconsistency in mechanical properties is observed despite achieving favourable grain orientation. Either of tensile strength, proof stress or ductility or in combinations, the properties achieved is low and no reproducible results are obtained. The forgings followed the forging contour. So the forging parameters is not a suspect. Therefore the investigation was focussed on heat treatment parameters. The forgings have to be subjected to T6 temper condition followed by cold water quench as per the material specification. In controlled experiments, the forgings are subjected to different ageing temperatures and soaked to different durations. Subsequently the quenchant temperature is also varied. The properties are evaluated by the comparison of hardness, tensile test and the micro examination. In the optimized heat treatment cycle, hot water quenchant is introduced and ageing cycle is altered with reference to material specification. Thus the repeatable mechanical and metallurgical properties are obtained and the process is incorporated in the traveller. The same process is extended to other hub forgings.

Experimental investigations on heat treatment of cold work tool steels: grade (D2)

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The present experimental investigations deal with the improvement of mechanical properties of cold work tool steels through different heat treatment processes. An attempt was made to get optimal combination of hardness and toughness through changes of microstructure by heat treatment. The toughness of D2 tool steel increases with tempering temperature. The hardness of D2 tool steel decreases with increase of tempering temperature. At lower tempering temperature range (160-200°C), the effect of tempering temperature on hardness is very small but as the tempering temperature increases hardness becomes prominent. The hardness of D2 tool steel increases with austenitizing temperature and the lowest and highest hardness were obtained at 970°C and 1000°C respectively. Results were compared with standard conventional test results. It is observed that different heat treatment processes significantly improves the mechanical properties like hardness, toughness and microstructure of cold work tool steel.

Effect of beta solution treatment on machinability of titanium alloy Ti-6Al-4V

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Titanium alloys are one of the most difficult materials to machine because of their low conductivity, which leads to high cutting temperature and its chemical reactivity with tool materials. A beta solution treatment process (BSTA) produces widmanstättten α-β colony microstructure and it is mainly carried out to improve the fracture toughness of Ti6Al4V. This work is carried out to study the effect of beta solution heat treatment on machinability of Ti6Al4V. The experiments are planned using Taguchi design of experiment approach and using L8 orthogonal array, where cutting speed, feed and depth of cut are varied on 2 levels. The machinability is measured in terms of cutting forces, surface finish of the machined workpiece, tool wear, etc. The optimum cutting condition is determined using Taguchi linear model analysis. The chip mechanism study is carried out to establish a direct relationship between machinability and beta solution heat treatment.

Cryogenic treatment of hot work die steels

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Hot work die steels such as AISI H13, are one of the important materials in industrial field due to their wide range of properties and applications. The main concern with respect to these materials is the conditions under which these materials work such as high temperature, application of loads with intermittency, etc.

which deteriorate these materials thereby reducing the tool life. The cryogenic treatment can be used to enhance the performance of these materials. The AISI H13 samples were hardened and tempered by conventional way. The other samples of AISI H13 were subjected to cryogenic treatment after conventional heat treatment with subsequent soft tempering at the end of the process. Different soaking times were used during the cryogenic treatment. Wear of the die materials is one of the major concerns during their actual application. Hence, the wear test was performed for samples with conventional heat treatment and the cryogenic treatment. The results were compared which indicated improved performance for the cryogenically treated samples as compared to the conventionally treated samples, which is as a result of conversion of retained austenite into the martensite, precipitation of carbides, etc. The wear test also assisted in optimizing the soaking time during cryogenic treatment as 16h at which the material showed minimum wear rate as compared to the others. The SEM micrographs also indicate the presence of carbides with different morphologies justifying the reason for improved wear resistance of the materials after the cryogenic treatment.

Development of a system for optimum thermal regime selection and control at tandem annealing line furnace of silicon steel mill, Rourkela steel plant through application of expert system

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An intriguing problem faced in Decarburization-Annealing Furnace of Silicon Steel Mill (SSM) is selection and control of optimum zone temperatures in commensuration with coil steel grade and varying line speed. This is basically due to unavailability of exact mathematical co-relations, which can define the process behavior. However, such cases are most suitable application areas of Expert System, which can take decision as would have been taken by a process expert or group of experts. A close loop Expert System based heating control system has been developed for Decarburization-Annealing Furnace of Tandem Annealing Line at Silicon Steel Mill of Rourkela Steel Plant jointly by RDCIS and RSP. The mill produces electrical grade steel used for various electrical and electromagnetic applications. One of the key factors responsible for achieving the desired electromagnetic and mechanical quality of steel is selection of optimum thermal regimes or temperature set points for the furnace zones and its accurate control. The concepts of Expert System have been used for selection of most appropriate set temperatures of different furnace zones based on steel grade, line speed and other steel coil parameters. The selection is made by Expert System knowledge base, which has been created based on years of experience and expertise of shop floor operation engineers, researchers and quality control agencies. The knowledge base works in tandem with inference rules, prepared following plant standard operating principles, to select optimum zone set temperatures. To make it truly a close loop control, Expert System downloads the selected set temperatures to a PLC for actual heating control of furnace zones. PLC controls the furnace temperature as per set points given by Expert System, by generating appropriate commands to zone electrical heaters. PLC then communicates the actual process variables like actual zone temperature, line speed etc. back to Expert System for supervision and data archiving. The system has rendered significant improvement in plant performance with respect to improvement in product quality and reduction in energy consumption. It also exemplifies an Industrial demonstration of concepts of Artificial Intelligence and Expert System for enhancing the efficiency of a plant.

Microstructure and mechanical property evaluation of heat treated AISI 5160 steel

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AISI 5160 is a shallow hardenable steel grade with near eutectoid composition i.e. 0.6 wt% C & 1 wt% Cr, used for applications such as heavy duty wheels, leaf springs etc. The steel has high wear resistance due to high pearlite content. It was decided to examine microstructural development in the heat treatment of thicker section to understand the behavior of the steel. It was decided to correlate the hardenability of the steel in 140 mm diameter bar in the hardened and in tempered condition. The steel was subjected to normalizing prior to hardening. The steel showed that in normalized, hardened and tempered condition the steel shows pearlitic structure in the matrix. The effect of the mass of the steel was correlated with the development of hardness distribution in the bar. It was found that the martensite phase prevailed only for 1mm thickness beyond which there was coarsening of pearlitic structure in the matrix. The microstructures were examined in optical and electron microscopy. It is found that fineness of the pearlitic structure increases on water quenching. The various microstructures that could be developed along with microstructural development would be presented. The influence of heat treatment on the steel properties has been studied. The pearlitic structures of the steel enables deployment of the steel for wear resistant application where harder pearlitic and alloy carbide phases are desirable.

Session 2: Nov 12, 2014: [16.00 - 18.00]

Modeling and optimization of shaft distortion during carburizing and quenching operation

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Heat treatment is known to improve the mechanical properties of engineering components. However, the side-effect of such a process is that the part gets distorted. This has significant economic implications due to further machining or correction operations. A lot of effort had been given, from both academics and industry, to investigate heat treating distortion phenomenon. The outcomes of such efforts have shown that distortion should be treated as a system attribute and the components and the manufacturing routes should be taken into account during investigation. In this present work, a systems approach of distortion reduction has been undertaken for a shaft undergoing carburizing process. We have used an integrated finite element analysis (FEA)-based model to predict distortion of the component. Investigation was carried out on the effect of raw materials and carburizing process parameters on distortion of a shaft. Bending of the shaft, change in length and change in diameter are the various types of distortion considered in this study. We conclude that to control different types of distortion, parameters and variables are to be controlled differently. We need to prioritize which particular type of distortion is problematic and control the parameters, within specification limits.

Effect of heat treatment on the properties of AISI H13 steel after duplex plasma treatment

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The duplex surface engineering involves the sequential application of two (or more) surface technologies to produce a surface with combined properties not attainable through any individual surface technology process. The duplex process consists of surface plasma nitriding, followed by a hard PVD coating to achieve improved wear, fatigue, corrosion resistance properties and higher load-carrying capability of steel substrates. In this present study, an attempt has been made to investigate the surface properties after plasma nitriding with three different time durations such as 6hrs, 12hrs, and 24hrs, followed by PVD coatings on as-received and heat treated conditions and the role of initial surface condition on duplex treatment. After duplex treatment, samples were characterized by XRD and SEM. Surface roughness was evaluated by surface profilometer, microhardness by Vickers microhardness tester and wear resistance properties by tribometer. The surface roughness and the hardness values are higher for heat treated conditions as compared to the as-received conditions for the above three different time durations. It has been observed that the initial surface condition and thickness of white compound layer formed during plasma nitriding, play important roles in governing the final properties of the coating material. The wear resistance is also found to be maximum in heat treated condition for duplex treated samples compared to the as-received condition.

Effect of austenitization temperature on transformation of austenitic phase in P91 steel

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P91 is a high temperature ferritic/martensitic steel with 9wt% Cr, 1wt% Mo, 0.1wt% C, 0.22wt%V and 0.07wt%Nb used in ultra supercritical power plants and advanced nuclear reactors. This steel transforms into austenite in the heat affected zone (HAZ) during welding. However, the HAZ region experiences a range of temperature between Ac_1 (austenite start) and Tm (melting temperature) for a very small time duration. Therefore, austenitization of this steel takes place under the conditions, which are vastly different from that in case of normalizing of this steel. Effect of the austenitization temperature on the transformation behaviour of austenite in P91 steel was studied using dilatometry. Dilatometry was performed using cylindrical pins (diameter: 4 mm, length: 15 mm) of P91 steel in normalized and tempered condition. The specimen were heated to the austenitization temperatures and then cooled to room temperature a rate of 10 ^{o}C/min, without any soak at the austenitization temperature. Four different austenitization temperatures: 850 ^{o}C, 950 ^{o}C, 1050 ^{o}C and 1150 ^{o}C, were used in this study. The specimen austenitized at 950 ^{o}C and above transformed into martensite, below the Ms temperature of 380 ^{o}C, during cooling. However, the specimen austenitized at 850 ^{o}C transformed into ferrite plus carbide in below 780 ^{o}C (780 – 720 ^{o}C range). This shows that low austenitization temperature and time can substantially affect the hardenability and therefore, transformation behaviour of the austenitic phase of

this steel. This finding is important because the outer HAZ region of the welded joints in this steel experiences very low austenitization temperature and the microstructure in this region is responsible for premature failure of the weld joints by Type IV cracking. The results of the dilatometry experiments and the resulting microstructures have been presented and discussed in this paper.

Study on mechanical behaviour of Al alloy AA 2024 in differently processed and heat treated conditions

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The high strength Al alloy AA2024 is extensively used as structural materials in aerospace and aircraft industry. The AA2x24 series alloys are strengthened by microstructure evolution during ageing, which is influenced by the amount/nature of cold work imparted to the materials after solution treatment, generating heterogeneous sites for subsequent precipitation process. It has been observed that AA2024 alloy after solution treatment respond differently to cold working process (stretching/ compression/ no cold work) which considerably affects the mechanical properties of the alloy. To understand the underlying mechanisms, an attempt has been made to study the effect of cold working (stretching/ compression) on the mechanical properties and the microstructure of alloys having different copper values (4.0 wt% and 4.5 wt%). Effect of these variables has also been studied with other heat treatment tempers. Samples are characterized using optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and electron back scattered diffraction (EBSD) techniques. Also an attempt has been made to correlate the achieved mechanical properties with the nature, size and distribution of precipitates.

Structure and property evaluation of heat treated 20CrMoVTiB410 for high temperature bolting application

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20CrMoVTiB410 (0.2%C-1%Cr-1%Mo-0.25%V-0.1%Ti-B) is a low alloy bainitic steel used in high temperature bolting application of power plants. Although the material is explored for its high temperature applications, there is lack of understanding on the room temperature behaviour of the steel. This may enable exploitation of the material for other applications. Hence, a study was initiated in which the room temperature microstructure and mechanical properties was examined. The steel was studied in quenched and tempered condition at various tempering temperatures. The steel in the as quenched condition shows 100 % bainite with good yield strength (935 MPa) and impact toughness (35 J). Tempering at various conditions showed that the steel has a tendency to acquire secondary hardening at the temperature range 600 to 650°C due to the presence of high Mo and V contents. Tempering above 680°C showed excellent impact toughness (230 J) with very good retention of strength (850 MPa) due to the presence of alloy carbides in tempered bainitic ferrite. Due to the formation of bainite in as-quenched condition, the steel does not undergo 350°C embrittlement. SEM studies were carried out to find out the different precipitates formed during tempering.

Simulation of wear condition of sugarcane cutters and its performance evaluation

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Mechanical harvester is used to cut the sugarcane grown in soil containing about 20% silica sand. During cutting, cutter blades regularly come in contact with the soil which increases abrasive wear of the cutter blade.This project reports the wear rate factor between Pin on Disc (POD) setup and Simulated Wear Test Rig Fixture (SWTRF) which was designed and fabricated in the laboratory. For experimentation, En45 material was used for cutter blade (SWTRF) and Pin (POD). This material was subjected to conventional heat treatment (HT) and cryogenic treatment (HTC). A set of four cutter blades was rotated in SWTRF at varying rotations (300, 400, 600 and 750 rpm) for varying periods (8, 16, 24 and 32 h). Similar speed and time period was maintained constant in POD wear test. The results showed that HTC has exhibited more wear resistance than HT in both wear conditions. An operating wear mechanism prevailed in dry sliding and abrasive wear environment was discussed.

Tribological behaviour of an effect of boron and carbon addition solution treated and solution treated plus aged condition Ti-10V-2Fe-3Al.

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A detailed study tribological property correlation was carried out in beta titanium alloy (Ti-10V-2Fe-3Al) with and without 0.1boron and 0.1carbon additions. The alloys were prepared by non-consumable vacuum are melting followed.by hot forging. A pin on disc apparatus is used for the testing according to ASTM standard (G99). Appling four difference load (20 and 50N), five difference track diameters from (50 and 110mm) and five difference Sliding velocity (0.5 and 1.50m/s) at contents distance 1km travel. Addition of with and without boron and carbon resulted in the precipitation of TiBC respectively. And these phases acted as reinforcements. Evaluation of tribology properties in solution treated and solution treated plus aged condition showed strengthening (weight loss) in the boron and carbon containing alloy with respect to the base. Microstructural characterization was carried out using optical microscope-worn surfaces pins a scanning electron microscope (SEM), Energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) analyses confirmed the tendency of Ti alloys to transfer material to their counterfaces and suggested possible tribochemical reactions between the steel alloys disks and Ti alloy sliders.

Effects of heat treatment on the stability of microstructures in as-cast 23-8-N nitronic steel

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Recently, nitrogen bearing austenitic steels has been gaining much more attention by the fact that these materials provide a unique combination of mechanical, chemical and physical properties because of the beneficial effect of nitrogen [3-7]. The as-cast nitronic steel possesses austenite phase with massive precipitates of carbides. The presence of amount and dispersion of carbides adversely affect the mechanical properties like ductility, impact energy, and resistance of steel to fatigue, creep, wear, and erosion. In the present investigation, effect of heat treatment on microstructure of as-cast 23-8-N nitronic steel was studied. The temperature range was decided judiciously as rise in temperature leads to more and more dissolution of carbides. As-cast 23-8-N nitronic steel was heat-treated in the temperature range of 1060℃ to 1240℃ for 90min, 120min and 150min respectively followed by water-quenching. It was observed that some phase transformation occurred during thermal exposure, through precipitation reaction $\gamma \rightarrow \gamma' + M_{23}C_6$. Hence, it is clear that quite number of newly nucleated grains is formed along the grain boundaries and further growth of nucleating grains as raising the temperature and time. After the complete heat treatment, dissolution of chromium carbide occurs rapidly and chromium diffused into austenite. XRD analyses were also carried out on both as-cast and heat-treated samples. Finally, a systematic study is presented followed by the effect of heat treatment on the stability of structure and distribution of carbides in 23-8-N nitronic steel.

Study of surface hardness behaviour of heat treated Ni-Cr-Mo alloys

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Low carbon steel is easily available, cheap and having good material properties that are acceptable for many applications. Hardening and tempering process are used as a major heat treatment method. The purpose of heat treatment of low carbon steel is to improve the ductility, toughness, hardness and tensile strength. Hardening and tempering process of metals offer enormous advantages to the manufacturing industry because the heat treatment results can reveal optimum combination of mechanical properties. In this research, Ni-Cr-Mo alloys have been chosen for this purpose of investigating the characteristics of hardness and tensile strength of the low carbon alloy steel specimen. The steel samples were heat treated in electric furnaces (for carburizing, casehardening and tempering processes) at different temperature levels and soaking to the particular time and then cooled in quenching media. The specimens were heat treated at 900°C for 1hr and the specimen then undergone to tempering process for different temperature level such as 250°C, 350°C, 450°C and 550°C for different tempering time (60min, 75 min and 90 min). The hardness of the treated and untreated samples were determined using standard operating procedures. All heat treatment components were checked for Rockwell hardness and tensile strength. The experimental results revealed that the mechanical properties of selective alloys were significantly changed by tempering treatment. By increasing the tempering time and temperature, hardness value decreases gradually, whereas ductility increases. The objective of this research is to find the effect of hardness and tensile behavior of heat treated 20 Ni55 Cr50 Mo20 alloy steel under the processes of different tempering temperature and time.

To study the effect of annealing on hardness and impurity distribution of high purity niobium

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Hydrogen degassing is an essential treatment during the fabrication of the superconducting cavities of high purity niobium. This treatment is given to reduce H content in bulk niobium which affects the performance of SCRF cavities. Any high temperature process would be accompanied by changes in mechanical properties. The aim of this study was to study the effect of various annealing temperatures and the change of vacuum level on the hardness and impurity distribution of this high purity niobium. It was found that the H of the bulk niobium decreased with improving vacuum level and increasing temperature. The hardness of atmospheric annealed samples increased with increasing temperature because of increase in diffusion of impurities like of C and O with increase in temperature. Annealing at a vacuum level of 1e-5 mbar did not show any change in the hardness of the samples at any temperature. However, still better vacuum level led to anomalous increase in hardness with increase in temperature. This was later attributed to the diffusion of C that might be responsible in the formation of NbC.

Cooling peformance of select vegetable oils and mineral-vegetable oil blend quench media

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Heat treatment of metals is an important operation in manufacturing of engineering components. Heat treatment changes the microstructure of alloys and thus affects mechanical properties of alloys .Quench hardening is a process where an alloy is heated to solutionizing temperature and held for a definite period, and then rapidly cooled into a quenching medium. In the present work, the performance of vegetable oil and mineral-vegetable oil blend quench media was assessed. The vegetable oils used in this work were olive oil, canola oil and rice bran oil. The mineral-vegetable oil blends were prepared mechanically mixing 10 and 20 vol.% of rice bran and canola oil in mineral oil. Inconel probes of 12.5mm diameter and 60mm height, instrumented with thermocouples were used to characterize the quenchants. The probes were heated to 850°C and quenched in oil quench media. The cooling curves analysis and inverse modeling technique was used to study wetting kinematics and estimate spatially dependent metal-quenchant interfacial heat flux respectively. It was found that vegetable oils exhibited very short vapour blanket stage compared to mineral oil and blends. Also, faster wetting kinematics was observed in blends which result in uniform heat transfer compared to that of mineral oil. Thus steel parts quenched in vegetable oils and blends would lead to better hardness distribution compared to those quenched in mineral oil.

Tempcore process in Visakhapatnam steel plant

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Tempcore process, developed for economic production of quality reinforcement bars, was patented in Belgium during the early 1970's. In the Tempcore process, the tempering of a previously quenched surface layer of a steel bar takes place under the action of heat supplied by the core, which takes place as it emerges from the last finishing stand of the mill. It undergoes following three stages (a) In the first, fast cooling step, the bar leaving the last stand passes through a water cooling stretch and the surface of the bar is quenched by forcing the water with a high pressure directly on the bar. This result in a microstructure, comprising martensite phase at the surface up to a certain depth, whereas the core remains austenitic phase, with a mixture of martensite and austenite phase in-between the two. (b) In the second stage, the bar leaves the cooling stretch as its surface is exposed to ambient air, a temperature gradient occurs in the cross section causing the heat to flow from the centre to the surface. The core of the bar reheats the quenched surface layer by conduction, the reheating surface layer results in self tempering of the martensite formed during the first stage, which ensures adequate ductility in the expense of hardness with a high yield strength. In this stage, the untransformed austenite in the surface layer transforms to bainitic phase and the core remains austenitic phase. (c) In the third stage, the bar lying on the cooling bed cools from the tempering temperature, isothermal transformation of the austenitic phase takes place transforms either to a ferrite and pearlite or to a pearlite, ferrite and bainite. In this work, the modification of the mill to achieve steel with YS greater than 500 MPa without reducing the miss speed will be discussed.

Effect of steel hardenability on distortion using navy C ring technique

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Distortion is a major issue plaguing the heat treatment industry. The issue of distortion occurs due to volume changes associated with thermal expansion and phase transformation. The volume changes when it is constrained leads to residual stresses that manifests as distortion. Any thermal treatment on a metal involves volume changes and if proper design of component or thermal treatment process is not carried out, distortion would manifest. Hardening process in steel involves martensite formation with about 4% volume change that imparts highest form of distortion. Hence, it is necessary to understand the phenomenon that takes place during the thermal processing. This study explores the study of heat treatment distortion in hardening of as-hot rolled steel grade AISI 4140 at two different hardenabilities. The study is carried out using a standard Navy-C ring test sample, where there is a continuously varying cross-section. The process occurring in the actual thermal treatment process was modeled using the heat treatment module of the finite element based software FORGE. The experimental data obtained could be matched with the simulation data. It is observed that the distortion is decreased in steel with higher hardenability. The software could predict the progress of distortion due to phase transformation and simulation results are in good correlation within 20% with experimental values for gap opening and outside diameter distortion. Thus, it is possible to use the software for heat treatment analysis. The

microstructure examination at various location of the sample showed formation of fine martensite at thinner location and coarse martensite with bainite in thicker zone.

The effect of section thickness on properties of retrogressed AA 7010 alloy forging

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Over the years, high strength aluminium alloys belonging to 7xxx series have been widely used as aircraft structural material because of their high strength to weight ratio and excellent mechanical properties. But these alloys in the T6 temper condition are susceptible to stress corrosion cracking (SCC). This could be overcome by using the alloy in overaged condition (T74) which reduces its strength by about 10-15% compared to T6 condition. These shortcomings of combined strength and corrosion resistance could be overcome by multi-stage heat treatment known as retrogression and re-ageing. In the present study, the properties of retrogressed and re-aged AA 7010 Al-alloy forgings are studied for two different section thicknesses. Samples having two different section thicknesses of forged AA7010 Al-Alloy are initially subjected to T6 temper condition. This is followed by subjecting the T6 treated samples to retrogression at 200^0C for different durations. These are subsequently re-aged in accordance with T6 temper condition. The sample thus retrogressed and re-aged is evaluated for different properties like hardness, electrical conductivity and tensile tests. The results of two section thicknesses are compared. The loss of strength or hardness and improvement in conductivity (for better SCC resistance) for both thicknesses are compared. The inference on the SCC resistance of the forgings is drawn based on the comparison of properties of different thicknesses with the peak-aged (T6) and over aged temper (T74) conditions.

Effect of heat treatment and surface condition on inter granular corrosion of alloy 800

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The Alloy 800 has been selected as a potential candidate material for Steam Generator (SG) tubings for PHWRs. However, widespread degradation of SG tubes occurred and many tubes were blocked during service. Inter Granular Corrosion (IGC) being one of the main degradation mechanisms in case of SG tubes, a study was conducted to analyze the effect on the corrosion rate of Alloy 800 at different process conditions involving solution annealing temperatures, sensitization and surface conditions. To understand the effect of solution annealing, cold worked samples were annealed at temperatures ranging from 950°C to 1050°C and IGC test was carried out after sensitization at 675°C/1hour. The effect of time and temperature on the sensitization behavior of solution annealed samples was studied by varying sensitization time from 15min to 150min and temperature ranging from 640°C to 700°C. The effect of processing conditions resulting in varying surface condition such as OD belt grinding and Glass Bead Peening (GBP) was also analysed. When solution annealing temperature was varied from 950°C to 1050°C, an increase in corrosion resistance was observed. For lower sensitization temperatures i.e. up to 650°C, the corrosion rate was not affected much by sensitization time. At higher temperatures, sensitization was initiated at lower time and corrosion rate was strongly affected by variation in sensitization time. The

results were correlated with Electrodynamic Polarization Reactivation (EPR) study where the degree of sensitization was evaluated. As the usual annealing of the cold worked tubes is carried out in cracked ammonia atmosphere, which promotes nitrogen pick up, the samples without surface grinding operation when tested for IGC after sensitization at 650°C/15m have shown higher corrosion rate. GBP alone could not minimize the corrosion rate as it removes very less amount of material. The combined operation of OD grinding and GBP reduced the corrosion rate drastically.

Effect of thermal cycling on structure and mechanical properties of a martensitic stainless steel

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Chromium – nickel martensitic stainless steel with micro additions of niobium / vanadium find extensive applications as structural components in power and aerospace, in view of their attractive strength – toughness combination. Such steels are often subjected to high temperature solution treatment (t~ 1150°C) in order to achieve strengthening from fine MC type carbide precipitation during subsequent aging treatment. However, the first set of solution treatment and ageing needs to be supplemented by suitable thermal cycling treatments in order to refine the martensitic sub-structure, with the objective of achieving very high toughness at ultra high strength levels which is one of the essential requisites for critical structural applications. In the present study a martensitic stainless steel with nominal composition 0.09C-16Cr-4Ni-0.1Nb steel was subjected to 6 stage treatment comprising of three set of solution annealing and ageing cycles at suitable temperatures. Detailed microstructure and property characterization was carried out after each set of thermal treatments. Although the first set of thermal treatment led to grain coarsening (60 μ), subsequent thermal cycling resulted in extensive structural refinement (15 μ). The high level of tensile ductility (>70% RA &> 25% E) and Charpy impact toughness (>200J/cm^2) coupled with high strength of over 1200 MPa speaks in favour of structural refinement achieved during the heat treatment. The present paper will highlight the microstructure and properties obtained during different stages of heat treatment schedule.

Session 3: Nov 13, 2014: [09.00 - 11.00]

Laser surface hardening of ductile iron and austempered ductile iron

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Ductile irons are widely used in various industries, particularly for automobile applications like brake cylinders, camshafts, connecting rods, gears, pistons and yokes. Traditionally surface heat treatments like induction hardening and in recently electron beam and laser hardening are being used to improve wear resistance. However, laser surface hardening has advantages such as low distortion due to high power density, process flexibility, accuracy, self quenching and limited grain growth. In this work, laser surface hardening of ferritic- pearlitic ductile iron (FPDI) and austempered ductile iron (ADI) grades was carried out. Hardening was performed with a 400W continuous wave fiber laser with the objective to investigate effect of single pass and multiple pass scans on the microstructure and case depth. The process

parameters such as power density, scanning speed, scan length and beam overlap were optimized. The effect of local tempering in multiple pass laser surface hardening on hardness profiles was also investigated. The surface hardened specimens were characterized by metallography, hardness traverse, pin on disc wear testing, XRD analysis and residual stress analysis. Transformation hardening resulted into shallow depth and limited rise in case hardness, whereas, melting cases were deeper and harder. The melted cases of FPDI iron showed mainly martensite, cementite and retained austenite with depth of 0.1 to 0.4 mm with hardness between 750 and 900 HV. Similar microstructure was found in the melted case of ADI iron with slightly higher amount of retained austenite. The molten case depth of ADI was between 0.3 and 0.7 mm with hardness between 850 and 1000 HV. The cases were induced with compressive residual stresses in both types of irons. Overall wear resistance was improved due to molten case.

Mechanical and corrosion behaviour of nanocomposite thin film coating on weathering steel

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Weathering steels are widely used for structural purpose due to their superior anticorrosion performance in atmosphere and low maintenance cost. However, the steel undergoes significant degradation in humid and sultry environments, which in turn adversely affects its fretting wear resistance. Thin films of pure Ni and Ni-TiN nano-composite were deposited over the weathering steel substrate by reactive magnetron co-sputtering of high purity (99.99%) Ti and Ni targets used as RF and DC sources, respectively in Ar+N_2 atmosphere. For this purpose, sputtering was carried out at Ar: N_2 ratio of 1:2, negative substrate bias voltages of –60 V and at ambient substrate temperature. GIXRD study have shown <111> as the preferred orientation for Ni in Ni thin film coating and <111> and <200> for Ni and TiN respectively in case of Ni-TiN nano-composite thin film. The microstructures of the thin films were examined using field emission scanning electron microscope (FESEM) while chemical composition was determined by energy dispersive spectroscopy (EDS). Nano-indentation studies have shown hardness of the Ni-TiN nanocomposite film more than that of pure Ni thin film. From nano-scratch test, it was observed that, the coefficient of friction is found to be lower for the Ni-TiN nanocomposite films than that for Ni, probably due to higher roughness and increased adhesive wear of the latter film. By addition of TiN phase to the Ni matrix, the grain size of Ni phase has found to be decreased. From the fretting wear test, it was observed that, Ni-TiN thin film has better wear resistance than pure Ni coating due to hard phase of TiN. Study of the electrochemical behaviour of the films in 3.5 g/l NaCl solution has shown a decrease in corrosion rate in the order, Ni-TiN>Ni>Steel substrate. Higher corrosion resistance of Ni-Tin film was due to formation of TiO_2 phase as observed by XPS. The average biaxial residual stress measured through XRD $Sin^2\Psi$ analysis shows that Ni film is tensile where as it is compressive in Ni-TiN film. So, we can conclude that, nc Ni-TiN thin film coating has better mechanical properties and corrosion resistance as compared to pure Ni thin film.

Pulsed laser deposition of multilayered Ti/TiB_2 films on HSS substrate

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Laser ablation technique is used widely to deposit thin epitaxial films with major applications in electronic industry. One of the major attractions of this process is almost complete ionization of the plasma which aids diffusion of ions across the surface of the substrate resulting in a homogeneous film. In this research Ti/TiB_2 multilayered film has been deposited on HSS substrate by PLD. TiB_2 is the hardest metallic material which has a hexagonal crystal structure with a melting point of 3225 $^{\circ}$C. The depositions were performed at two different temperatures of 400 $^{\circ}$C and 600 $^{\circ}$C each for a period 80 minutes. Alternate layers of Ti and TIB_2 were deposited by changing the targets at definite intervals. 4 layered and 8 layered samples were prepared and characterized to study the phase (GIXRD), microstructure (FESEM, HRTEM), mechanical properties (Nanoindentation), roughness (Stylus Profilometer, AFM) and wear (Fretting wear) properties of the film. The thickness of the film was around 300 to 500 nm. TiB_2 with a preferred orientation of (101) was detected while Ti had a preferred orientation of (101) and (002). The microstructural analysis revealed a transparent film with drag lines formed on the surface. The TEM study showed uniform distribution of TiB_2. Nanoindentation tests on TiB_2/Ti thin films resulted in hardness as high as 46.5 GPa and reduced elastic modulus of 400 GPa. Fretting wear tests on the samples were performed at a load of 5N for the duration of 30 minutes. Wear depth for most of the samples was around 20 mm with a major contribution from WC ball used in the process. TiB_2 has high hardness and melting point making it preferable material for high temperature applications. High hardness also imparts high wear resistance and abrasion resistance to the sample which are desired in most of the materials.

Microstructures and properties of SiC_P/Al composite coatings developed using friction surfacing

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Metal matrix composite (MMC) coatings gained lot of importance in the recent times due to their superior properties. Existing fusion based techniques for depositing composite coatings lead to several problems such as dilution, segregation, interfacial reactions, poor wettability, porosity, and cracking.The current work demonstrates a novel approach to develop MMC coatings in solid-state using friction surfacing. Friction surfacing experiments were conducted using aluminum alloy AA2014-T6 rods (20 mm diameter), which contained several holes (2 mm diameter) filled with SiC powder (average particle size ~ 10 µm). After a meticulous process optimization, SiC_P/Al2014 composite coatings (2 mm thick) were successfully friction deposited on alloy 2014-T6 substrates. The coatings were characterized in detail using optical and scanning electron microscopy, shear tests (ASTM A263), micro hardness tests, and pin-on-disc wear tests (ASTM G99). Microstructural studies revealeddynamically recrystallized fine equiaxed grains and uniformly distributed SiC particles without any interfacial reaction products. Shear tests confirmed satisfactory coating-substrate bonding. The coatings showed superior hardness and dry sliding wear performance compared to uncoated alloy 2014-T6. Overall, the results show that friction surfacing is a novel technique for producing MMC coatings.

Oxidation kinetics study of Ti-Si-B-C nanocomposite hard coatings deposited by magnetron sputtering

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Nanocomposite Ti-Si-B-C hard coatings are potential materials for increasing demand of complex applications to be performed from the same material for different industrial applications. Such multiphase coatings can increase life and performance of various machine tools, engine parts, cutting tool etc to protect against wear, corrosion, and oxidation and can be tailored to very low friction coefficient coatings. Though hard coatings have been achieved the high temperature oxidation studies of nanocomposite are very few. The present investigation is focussed on the study of oxidation kinetics of multi component Ti-Si-B-C hard coating at different temperature, oxidation duration and the different thickness of the coatings. The hardness and modulus was found to be around 35 GPa and 450 GPa respectively. The oxidation studies were carried out in TGA system for different temperatures and durations for varying thicknesses of the coatings. The rate constant was found to be higher for the lower thickness and smaller durations, But after sometime it decreases and gets saturated confirming the passive layer formation on the film. The Kp values were found to be varying between $2x10^{-5}$ to $7x10^{-3}$ mg^2cm^{-4} h^{-1}. Micro structural, mechanical properties of oxidized samples were studied through SEM, AFM, and nanoindentation respectively. SEM image showed very fine grains in the range of 100-200 nm after oxidation. A SEM image showed 10 µm thick coating after oxidation for 50 h. The parabolic rates were observed which indicates the diffusion controlled mechanism of oxidation.

Studies on mechanism of whiskers formation and its suitable mitigations

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Whisker is a perfect single crystalline material. It is assumed that it may form to relieve compressive mechanical stresses exists inside material by forming projections from surface. These can cause short circuits and arcing in electrical circuits. Tin whiskers caused the failure of the Millstone Nuclear Power Plant in Connecticut was shutdown due to a "false alarm" that indicated an unsafe pressure drop in the reactor's steam system when the steam pressure was actually nominal in 2005. At frequencies above 6 GHz or in fast digital circuits, the whiskers act like antennas by affecting circuit impedance and causing reflections. There have been several reports on failures which were caused by whiskers and resulting in huge financial damage. But, yet there is no perfect strategy to explain what the actual mechanism is existed out here and their precise prevention measures. In this we tried to project the relative information by comparative analysis at variable temperature and humidity conditions and special focus on theories behind the formation of whiskers in corrosion environment.

Effect of surface mechanical attrition treatment on 6061-T6 aluminum alloy

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Surface Mechanical Attrition Treatment was conducted on Aluminum 6061-T6 alloy. Repeated impact of high velocity balls on surface causes severe plastic deformation, leading to formation of Nano-crystalline layer in the top surface which improves surface hardness. Mechanical alloying wasalso done using copper powder as embedment in the surface of aluminum alloy 6061-T6 using SMAT technique. Characterization

was conducted using Optical microscope, Scanning electron microscope, and X-ray diffraction.The results showed refinement of grain size and improvement in the hardness.

Comparative study of corrosion behavior of Al, Al-Mg and Al-Zn-Mg coating on steel sheet

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In recent years, market pressure has forced the research divisions of major steel coating companies to investigate alternative coating techniques and to develop new alloy coatings with improved performance. Aluminium films have a superior resistance to corrosion when compare to their casting counterparts which are widely used for practical applications. The Al–Mg alloys due to their low specific weight and high strength/weight ratio are attractive in a variety of technical applications such as food-equipment, chemical processing, transport and structural fields, especially where seawater exposure is involved and Al-Zn-Mg coating have good corrosion resistance, mechanical and better wear resistance property for applications on different automotive industry. In this experiment, an attempt has been made to coat a commercially pure Al, Al-5wt%Mg and Al-9wt%Zn-5wt%Mg using them as sputtering target on 4130 grade steel sheets. After deposition, samples were characterized by XRD, SEM and GD-OES. Surface properties were evaluated by OLS spectroscopy and corrosion properties by EIS and SST. Bend tests were performed to evaluate the mechanical properties. Both line and surface roughness was found diminished after coating in all cases, although Al-Zn-Mg displayed highest roughness. Although, Al-Zn-Mg coating delivered high corrosion resistance property following EIS and SST, bend test revealed poor adhesion among the lot.

Wear testing and analysis of H13 die steels

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The main concern with respect to hot work die steels, such as AISI H13, is the conditions under which these materials work such as temperature fluctuations, application of loads, etc. resulting in deterioration of these materials thereby reducing the tool life. The AISI H13 samples were austenitised at 1020°C, subsequently oil quenched and double tempered at 500°C. Along with this conventional treatment, some samples were cryogenically treated at -185°C followed by soft tempering at 100°C. Different soaking times were used during the cryogenic treatment starting from 8 hours to 32 hours. The pin on disc wear tests were performed on samples with conventional heat treatment and the cryogenic treatment with different cryosoaking times. The results indicated wear rate was reduced by 20% for the cryogenically treated samples as compared to the conventionally treated samples which is attributed to the conversion of retained austenite into the martensite, precipitation of carbides, etc. The wear analysis resulted in optimization of the soaking time during cryogenic treatment as 16 hours at which the material showed minimum wear rate. The SEM micrographs also indicated the presence of carbides with different morphologies justifying the reason for improved wear resistance of the materials after the cryogenic treatment.

Dry sliding wear behavior of particulate reinforced composite prepared via two- stage melt stirring at lower processing temperatures.

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The objective of this work was to determine the wear resistance of 6061Al-9wt%B_4C_p binary system prepared through stir casting and to analyze the parameters affecting the wear mechanism. Preheated mixture containing B_4C_p and K_2TiF_6 salt (0.1Ti ratio) were added into melt at processing temperature of 750°C in steps of two to avoid agglomeration of particulates. Distribution of B_4C_p in Al matrix was fairly uniform which was evident from SEM microphotographs. Micro-hardness of the reinforced alloy were carried out using micro Vickers hardness tester for a load of 2N with dwell time of 10sec. The composite prepared at lower temperature (750°C) consists of α-Al, B_4C and minor phases like Al_3Ti, AlB_2 and Al_3BC are confirmed by XRD studies. Dry sliding wear test were conducted on the composite for load of 10 to 60N with sliding velocity ranging from 6.67m/s to 13.34m/s at room temperature with moderate humidity conditions. Very few and small grooves with little scratches along the sliding distance were observed on SEM microphotographs of worn surface reveals that 6061Al alloy reinforced with 9wt% B_4C_p have better wear resistance in comparison with monolithic materials.

Session 4: Nov 13, 2014: [13.30 - 15.30]

Study of cyclic oxidation behavior of LTA as top coat material for TBC

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Environmental and economic issues are an increasing challenge for driving power generators to produce electricity more efficiently. This can be achieved by changing gas turbine power plants operating condition that is inlet temperature of gas. Current industrial gas turbines for power generation operate with inlet gas temperatures reaching nearly 1450°C, while aero engines use a firing temperature that is reaching to 1550°C. Coatings used for aeroengine applications undergo shorter thermal cycles at higher temperatures compared to those used in industrial gas turbines (800 to 950 °C). Industrial gas turbines, in contrast with aeroengines, are ramped slowly to their operating temperature and ideally remain there for thousands of hours. In present work the LTA that is $LaTi_2Al_9O_{19}$powder is used as top coat for thermal barrier coating (TBC) material. LTA thermal grade powder is studied with thermogravimetric analyzer, to evaluate its high temperature oxidation behavior. Different intermediate bond coat and top coat materials are used to enhance its life. LTA is one of them. $LaTi_2Al_9O_{19}$(LTA)/YSZ ceramic top coat reveal good chemical stability in molten salts of Na_2SO_4 andNaCl, and the bond coat plays a significant role in providing protection for the component against hot corrosion in the LTA/YSZ TBCs. LTA exhibits very promising potential as a novel TBC material. LTA powder is heated upto 950^{0}C and slowly cooled to room temperature to study its cyclic

oxidation behavior. No weight gain is observed at 950^{0}C. It shows that LTA is the one of the suitable top coat material to resist high temperature oxidation.

Surface modification of AISI 304 steel; the role of surface mechanical attrition treatment process parameters

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Surface of any material is the critical area of study as most of the failures originate from there. In this project the surface of AISI 304 was modified to obtain surface nanocrystalline layer. The fact that nanocrystalline materials have many crucial properties superior to regular micrometer size grains and therefore has huge attention towards severe plastic deformation (SPD). SMAT (surface mechanical attrition treatment) is one such process. In SMAT shots/balls which are mostly metallic ones are made to strike the sample surface in multiple directions to generate high strains. These large strain rates results in the generation and accumulation of dislocations, twins and related slip phenomenon resulting in grain refinement up to nanometre range at the surface (average depth of ~50 µm). This work is aimed to study the effect of various process parameters such as ball diameter, time, strain, strain rate, frequency and no of balls on the SMAT behaviour of AISI 304. High speed camera was also utilized to find the velocities of metallic balls striking the surface. The velocities were used to model the strain rate and related effects on SMAT of AISI 304.

Effect of prior thermally grown oxide layer on the thermal cycling life of EB-PVD TBC

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Ni-base superalloy components of gas turbine engines such as blades and nozzle guide vanes are applied with thermal barrier coatings (TBCs) for enhancing their high temperature performance and durability. TBCs are bi-layer coatings consisting of an inner metallic Pt-aluminide (Pt-Al) bond coat and an outer ceramic (7 wt.% yttria stabilized zirconia or 7YSZ) coating. The 7YSZ layer is deposited by electron beam physical vapour deposition (EB-PVD) method. The ceramic coating deposited by this method possesses the desired columnar structure that provides excellent strain-tolerance and thermal shock resistance. It has been reported that the presence of a thin thermally grown oxide (TGO) layer on the bond coat surface prior to YSZ deposition leads to better thermal cycling life of the TBC. In the present work, the effect of prior TGO layer on thermal cycling life of the TBC has been examined. Single crystal Ni-base superalloy AM1 has been used as the substrate. Pt-Al bond coated specimens were heated at 1050°C for 2h under various atmospheres (Table 1) for the formation of a TGO layer. Subsequently, 7YSZ was deposited on the specimens having with and without TGO by EB-PVD method. The TBC coated samples were then thermally cycled at 1100 °C in an automated furnace. It is confirmed that the cycling life of the TBC corresponding to the TGO formed under 80Ar-20O_2 atmosphere at vacuum levels of 10^{-1} and 10^{-4} was the highest. The beneficial effect of having a prior TGO on the cycling life of the TBC will be presented.

Effect of plamsa nitriding temperature on surface properties of 12Cr-10Ni martensitic stainless steel

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In satellite launch vehicle, one of the components made of 12Cr-10Ni martensitic stainless steel needs high wear resistance and high hardness of above 900 HV. To improve the wear resistance, plasma nitriding process was selected. In this present study, plasma nitriding was carried out at two different temperatures (450 and 475^{0}C) for same time (10 h). Effect of plasma nitriding parameters on the surface hardness, case depth, microstructure and phases present in the nitrided layer were investigated by microhardness measurements, X-ray diffraction (XRD), optical microscopy and scanning electron microscopy (SEM). It was observed that increase in nitriding temperature increased the surface hardness and case-depth. In addition, the presence of Fe_3N and Fe_4N phases in the nitrided layer was observed using X-ray diffraction technique.

Evolution of microstructure and its influence on transition of wear regimes in cryoprocessed AISI M35 tool steel

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Cryogenic treatment is add-on process for enhancing performance of the material and holds future assurance. The behavior of cryogenically treated tool steel needs comprehension in the context of mechanical and wear characteristics of M35, due to the thermal cyclic environment experienced during service. In this paper, Charpy impact test specimens were hardened at 1473 K, triple tempered at 673 K followed by cryotreatment at -88 K for an equal interval of period and followed by soft tempering at 373 K. These treated specimens were subjected to thermal cyclic conditions in muffle furnace at 523 K, 773 K and 1023 K and such treated specimens were impact tested. After that, the test specimens were characterized for carbide density, hardness and wear behavior. Metallography study has indicated maximum carbide density at 32 h of cryosoaking period with optimum wear resistance as demonstrated by the lowest dimensionless wear coefficient. The results revealed mild oxidative wear at lower temperature (773 K) and severe delamination wear at higher temperature (1023 K) with consequent reduction in wear rate by 50% at transition point (773 K). The combined effect of temperature and cryosoaking period was plotted to obtain 2D contour map, to establish the wear regimes by delineating dominant operative wear modes and wear mechanism.

Friction stir surface alloying of Al 1200

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Friction stir processing (FSP), developed from friction stir welding (FSW), is a new and unique thermo-mechanical processing technique wherein the severe plastic deformation by a rotating non-consumable tool is utilized for improvement of properties. FSP can be used for refinement of grain structure and homogeneous distribution of second phase particles. Aluminium and its alloys are usually used in aerospace and transportation industries because of their low density and high strength to weight ratio. Aluminium matrix composites also exhibit properties like high strength and better fatigue and wear resistance. However, these Al-based composites are known to show lower ductility resulting in restricted applications of these materials.Modification of surface properties but at the same time retaining the bulk ductility in Aluminium alloys can be an optimum solution. In this work we present the study on the potential of FSP for surface modification of Aluminium alloys by surface alloying. The possible improvement in surface properties like hardness by incorporation of metal powders like Cu in surface layers of Al 1200 during FSP, followed by T6 Heat treatment, was studied. The Cu metal powder was deposited into a groove by powder thermal spray gun. The effect of process parameters on the properties of processed material was also studied. Significant increase in surface hardness was observed in Al 1200.

Ni-10wt%SiC electrocomposite coating on harvesting blade

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Electrodeposited nickel composites are strong, tough and resistant to corrosion, erosion and wear. Its mechanical properties can be varied by changing the plating conditions, alloying with other elements and by incorporating the type and extent of ceramic particles and fibers within the electrodeposited nickel matrix. There are several reports on nickel based composite coating comprising of SiC, Al, Si_3N_4, graphite, tungsten carbide and boron carbide. But, the incorporation of SiC is used widely to enhance hardness. Low carbon steel with manganese as a major alloying element is the most popular material for fabrication of harvester blades. During harvesting these blades tend to get corroded and also get worn out quickly due to the abrasive and erosive nature of the hard particles of soil. This leads to huge problems of either getting the worn harvester blades replaced by a new set of blades resulting in uncalled increased farming investments. In recent years, surface engineering methods offer solutions to the address the above issues in farming. In the light of the above, this work focuses on nickel-silicon carbide composite electroplating of harvester blades. Nickel electroplating is commercially proven technique with it unique advantages being low temperature process and easily the most economical one. Nickel coatings offer excellent corrosion resistance even under harsh environment during farming. SiC has been used in the current work to enhance hardness of the blade. Erosive wear of the coated and uncoated harvester blade material have characterized by using air jet erosive wear test rig. The effects of standoff distance (40, 30 and 20 mm) and test duration time (5, 10, 15 min) on Vickers hardness value of both coated and uncoated harvester blade material is reported.

Surface engineering of 4330V and AISI 1020 steels: Role of surface mechanical attrition treatment (SMAT) and nitriding response

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Surface mechanical attrition treatment (SMAT) is a severe plastic deformation technique by which nanocrystalline layer can be obtained in top of the specimen surface. The nanocrystalline layer is about 50 µm in 4330 V and AISI 1020 steels. Gas-nitriding of SMATed steel was performed at lower temperature (at 450 ºC) for 6 h. Nitrided specimens were investigated by using x-ray diffraction (XRD) and microhardness measurements. It has observed that SMAT helped in enhancing the nitriding kinetics. Surface hardness after nitriding was considerably higher for the SMAted specimen as compared to the non-SMATed specimen. SMAT also helped in avoiding the formation of iron-nitride network at the edges and corners of the specimens.

Effcet of Pt-aluminide bond coat on mechanical properties of single crystal nickel-base superalloy AM1

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Pt-aluminide (Pt-Al) bond coats provide excellent high temperature oxidation protection to Ni-base superalloy components of advanced gas turbine engines such as blades and nozzle guide vanes. However, these coatings are known to cause degradation in the mechanical properties of superalloy substrate. The effect of a Pt-Al bond coat on the tensile and creep properties of single crystal superalloy AM1 has been examined in this study. The bond coat was applied by first depositing a Pt layer on the substrate. The plated specimens were given pre-aluminising diffusion treatment, pack-aluminising and post-aluminising heat treatment to form the coating. Tensile properties were evalusted in the temperature renge of RT-1100°C. Creep testing was carried out using four combinations of temperature and stress. The tested specimenswere metallographically examined in SEM. Application of the coating resulted in decrease in tensile strength (both YS and UTS) at all temperatures. The extent of lowering of strength remained in the range 8-28%. The lowering of strength can be ascribed to the lower strength of the coating. The coated alloy showed better ductility than uncoated alloy because of the surface protection to the alloy provided by the oxidation resistant bond coat. The creep properties (strain-to-fracture and time-to-rupture) of the coated alloy were somewhat inferior (10-20%) to those of the uncoated alloy. Such degradation in creep properties of the alloy can also be ascribed to the lower strength of the bondcoat and also to the various microstructural changes in and near the band coat / substrate interface during the creep test.

Wear behaviour of electroless Ni-P-Al_2O_3-ZrO_2 nano-composite coatings

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In the present work, wear characteristics of electroless Ni-P-Al_2O_3-ZrO_2 nanocomposite coatings on mild steel substrate was studied. The nanosized Al_2O_3-ZrO_2 (85:15) nanocomposites were prepared by co-precipitation and used as the reinforcement (4 g/L) for the synthesis of Ni-P-Al_2O_3-ZrO_2 nanocomposite coating. The morphology, size range and phase analysis of as-prepared Al_2O_3-ZrO_2 nanocomposites and EL coatings were characterized using X-ray diffraction (XRD), field emission scanning electron microscopy

(FESEM), and the properties of the coatings such as hardness, friction and wear (ball-on-disc) were investigated and compared with Ni-P deposits. The results showed that as-prepared Al_2O_3-ZrO_2 nanocomposites exhibit spherical shaped and size ranges around 30nm. After heat treatment (400°C, 1h), the microhardness and wear resistance of the coatings are significantly improved. The Ni-P-Al_2O_3-ZrO_2 nanocomposite coatings exhibit enhanced wear resistance over Ni–P coating.

Potential detrimental consequences of excessive PWHT on pressure vessel steel properties

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Components of Pressure Vessels, during manufacturing, undergo several heat treatments as per the customer's requirements, such as normalizing, hardening, tempering, or some combination of the same. The vessel is further subjected to several cycles of heat treatment in the course of fabrication, such as pre-heating, stress relieving and Post Weld Heat Treatment. In order to minimize the effect of these fabrication-related treatments, the job is required to be tempered at a relatively higher temperature. While high tempering temperatures do help to minimize the effect of fabrication-related heating effects, what is perhaps not realized is that excessively high tempering temperatures, in conjunction with high and long post weld treatments (such as carried out on thick-wall reactors), can greatly degrade the properties of the base metal. In this paper we have gathered data from the manufacturing of several forgings of two grades of pressure vessel steel. The cumulative time-temperature effect of various heat treatments have been made comparable using the Larson-Miller tempering Parameter. The corresponding effect on the steel has been measured via standard Tensile and Charpy V-notch tests. A study of these properties against progressively higher treatments shows a decline in properties beyond a specific level of tempering treatments.

Session 5: Nov 13, 2014: [15.30 - 17.30]

Process technology for production of APIX70 HR coils at BSL for spiral welded pipes

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To establish process technology for production of API X70 HR coils for spiral welded pipes, two trial heats were made through BOF-LF-CC route. The concast slabs were processed into 7.1 mm thick HR coils under suitably designed process variables to (a) ensure total dissolution of primary carbides during reheat practice, (b) engineer fine austenite grain size prior to entry into finishing mill through controlled roughing schedule, and (c) control ferrite grain size to less than 5 micron through low finish rolling and coiling temperatures. Properties achieved in HR coils will be presented. Microstructure of Nb-V-Cr HR coils manifested very fine ferrite-pearlite structure (ferrite grain size : ~3 μm), while acicular ferritic structure was observed in Mo bearing HR coils. Desired properties were achieved in Mo bearing steel, HR coils of

this steel were processed into 28 mm OD HSAW pipes. Specified mechanical properties like tensile (YS : 511-550 MPa, UTS : 589-646 MPa, %El : 34-38%, YS/UTS : 0.84-0.87, impact toughness in base (280-296 J) , weld (184-196 J) & HAZ (184-236 J) and 100% shear area in DWTT (at 0°C) were successfully met in pipes. Surface of the rolled pipes were found free from any defect with no incidence of edge lamination. All the pipes passed the hydraulic, ultrasonic and radiography tests.. As per trial results, Mo bearing HR coils of API X70 of BSL were found suitable for making of HSAW pipes.

A study on various heat treatment and its effect on high temperature mechanical properties of inconel alloy 740H

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Inconel alloy 740H is a candidate material for the use in Advanced Ultra Super Critical boilers at operating steam temperature of 760°C and steam pressure of 37.5 MPa. The alloy 740H is ASME code approved vide Case 2702 for manufacturing of tubes and pipes for superheaters, reheaters and headers. Nickel base superalloys containing higher volume fraction of γ′ (>40 wt.%) are reported to have yield strength anomaly with respect to temperature. However, tensile behavior of alloys containing low volume fraction of γ' is scarce. The present investigation is aimed at studying: **(i)** the effect of temperature on tensile behavior of IN740H as-received material and **(ii)** optimizing the solutionizing temperature and aging time for obtaining superior tensile properties. The presently investigated alloy contains <20 wt.% of γ′ having size of 20-40 nm as confirmed from Thermo-Calc calculation and TEM respectively. The tensile tests were conducted from room temperature to 900°C at the interval of 100°C and it is observed that the yield strength anomaly of nickel base superalloys having higher volume fraction of γ' does not exist for IN740H. From the scanning electron microscopic (SEM) studies of the fracture surface of tensile tested specimen it is observed that plastic deformation is concentrated in the prior austenite grain bodies and the final separation takes place through de-cohesion of grain boundaries at all the tested temperatures. The material was solutionized at 1120°C, 1150°C and 1200°C for 1 hour and aged at 800°C for 4 hour and tensile behavior was studied to know the effect of solutionizing temperature on tensile behavior. In another set of experiments the as received material was solutionised at 1120°C for 1 hour and aged at 800°C for 4, 16, 100, 500 and 1000 hours to correlate the coarsening behaviour of γ' and its effect on the mechanical properties.

Influence of austenitizing temperature and quenching condition on wear behaviour of dual matrix ductile iron

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An investigation was performed to investigate the effect of austenitizing temperature and quenching condition on the martensite volume fraction and abrasive wear of Ductile Iron with dual matrix structure. On the verge to investigate the effect of austenitizing temperature and quenching condition ductile iron of the composition 3.61 C wt. %, 2.1 Si wt. %, 0.2 Mn wt. % is intercritically austenitized in two phase region (α + γ) at temperatures of 785°C and 815°C. Subsequently samples were quenched into two different

quenching medium to obtain DMS. Here water and oil (at 100 ^{0}C) is used as a quenching medium. Abrasive wear test is performed as a function of applied load. Pin-on-disc type wear tester with 600 SiC emery paper is used. Wear test is performed for the total sliding distance of 6000 m with the sliding speed of 2 m/sec. Optical image and X-ray Diffraction pattern shows that with the increase in austenitizing temperature martensitic volume percentage increases. The experimental result shows that, the matrix micro-hardness increases with the increase of austenitizing temperature. Total weight loss of the lower martensite volume fraction i.e., austenitized at 785 ^{0}C sample is highest; however the weight loss in the sample is directly proportional to the applied load. The wear mechanism tends from mild wear to abrasive wear with the increase of load. The friction coefficient increases with the increase of material hardness and Matrix micro hardness near the wear scar of the wear tested samples decreases due to softening of martensite during wear test.

Assessment of heat transfer in MWCNT-water nanofluids for quench heat treatment

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Quenching is an integral process in industrial heat treatment of steels. The heat transfer at the metal/quenchant interface during cooling is controlled by the ability of a quenchant to extract heat. The assessment of cooling characteristics of a quenchant by cooling curve analyses aid in providing vital information to the heat treater in the selection of right quenchant for heat treatment. Nanofluids are engineered heat transfer fluids that are made by suspending nano sized particles in base fluids like water. Recent studies on nanofluids have shown that 0.001-5 vol.% addition of nanoparticles to water offers better heat transfer ability and improved wetting relative to the base fluids. Apart from this, water-based nanofluids are environment friendly compared to mineral oil quenchants and the cooling rate of such fluids can be altered by tailoring the concentration of nanoparticles addition. Spatially dependent transient heat flow in the radial direction was assessed for quenching of an Inconel 600 probe from 850°C in distilled water (DW) and DW based multi walled carbon nanotubes (MWCNT, 0.001wt.%) quench media. Quenching in DW based MWCNT nanofluid quenchant yielded reduced peak heat flux. The video images of quenching in MWCNT nanofluid showed more uniform movement of the wetting front along the probe.

Mathematical computation of material loss in SS 316 L (N) disc during high temperature wear and friction testing

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AISI Type 316 L (N) austenitic stainless steel is a structural material in sodium cooled fast breeder reactors because of its compatibility with liquid sodium, high temperature strength, toughness, creep and low cycle fatigue. Sliding wear experiments were carried out at various elevated temperatures at constant load (40 N) and with a sliding speed of 0.8 m/s using a high vacuum pin-on-disc test rig as per ASTM standard G99-05. Wear testing was carried out at room temperature, 300, 400 and 500°C. The weight of the pin was measured before and after the sliding wear test to quantify the loss of material during dry sliding wear. Scanning electron microscopy (SEM) was carried out in order to analyze the wear tracks at various

temperatures. The accurate weight measurement before and after wear testing in Disc was not possible by the conventional method. A geometrical equation for volume computation for a partial torus was derived. The computation was compared with the existing surface Profilometric feature analysis software. The wear scars in the discs were studied to formulate the geometrical equation. At higher temperatures adhesive wear was predominantly seen which is responsible for higher material loss and the same was supported by SEM and surface profiling results. Wear rate regimes were identified and classified with respect to the operating temperatures.

Microstructural characterization, and wear behavior of nano-boride dispersed coating on AISI 304 stainless steel by hybrid high velocity oxyfuel spraying laser surface melting

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The present study concerns detailed microstructural characterization and investigation of wear behavior of nano-boride dispersed coating developed on AISI 304 stainless steel by high velocity oxyfuel spray deposition of nickel based alloy and subsequent laser melting. There is a significant refinement and homogenization of microstructure with improvement in micro-hardness due to laser surface melting (1200 VHN as compared to 945 VHN of as-sprayed and 250 VHN of as-received substrate). The high temperature phase stability of the as-coated and laser melted surface has been studied by differential scanning calorimeter (DSC) followed by detailed phase analysis at room and elevated temperature. There is a significant improvement in wear resistance of laser melted surface as compared to as-sprayed and the as-received one due to increased hardness and reduced coefficient of friction. The mechanism of wear has been investigated in details. Corrosion resistance of the coating in a 3.56 wt.% NaCl solution is significantly improved ($4.43E^{-2}$ mm/yr as compared to $5E^{-1}$ mm/yr of as-sprayed and 1.66 mm/yr of as-received substrate) due to laser surface melting as compared to as-sprayed surface.

Elevated temperature wear of thermal sprayed Cr_3C_2-25NiCr coating

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Sliding wear at elevated temperature is a serious problem in a large number of engineering components in various industrial environments such as power generation, transport, materials processing, high temperature bearing, impeller bearing of slurry pumps operated in waste tank, etc. Cermet variety of coating such as WC-Co, Cr_xC_y-NiCr, obtained by thermal spraying is well known for their excellent tribological properties. WC-Co provides excellent performances at ambient temperature. However, in corroding environment or at elevated temperature, the synergistic effect of mechanical attack and chemical attack degrades its performance. This limits the application of this coating up to 625 K. In contrast, chromium carbide nickel chromium coatings are employed at elevated temperature up to 1173 K for its excellent oxidation and wear resistance. In view of this, the present investigation is undertaken to understand the nature of film that forms during sliding of Cr_3C_2-25(Ni20Cr) coating against Fe based material, martensitic 100Cr6 steel and Ni base dispersion strengthened alloy PM 1000 at different temperatures. Towards that purpose a series of systematic experiments on high temperature wear of

Cr_3C_2-NiCr coatings on 253 MA alloy against 100Cr6 steel and PM 1000 alloys at different temperatures are carried out. Based on the study it is noted that glazed layer are formed on the worn surfaces during elevated temperature sliding wear of thermal sprayed coatings. These layers have characteristics physical appearance, mechanical properties, chemical compositions, friction response and failure mechanisms. Wearing conditions, wearing material and mating material influence formation and characteristics of glazed layers. These glazed layers are divided into different types of layers, namely, transfer layer (TL), mechanically mixed layer (MML), reaction layer (RL) and composite layer (CL).

Field performance analysis of cryoprocess HSS drills and taps

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The experimental study investigates the performance of Cryogenically Treated Conventional Uncoated H.S.S. M2 Drills and P/M H.S.S. M3 TiN coated taps. The Fermentation Factors and their assigned values were maintained constant throughout the study (soaking temperature: -185 °C, time: 16 h, cooling rate:1 to 2.5°C/min followed by soft tempering temperature at 100 °C for 1 h). For conventional uncoated H.S.S. M2 drills the speeds and feed-rate were offset by 10% on lower side and higher side of the datum. For P/M TiN coated taps the speed and feed-rate were offset by 5 % in the steps of three on the higher side of datum. The trials of drills were taken on drilling-cum-tapping machine with swiveling head attachment and the trials of taps were taken on vertical drilling-cum-tapping machine. It has been observed that tool life of conventional uncoated drills is improved by 100% or twice with 10 % increase in speed and by 80 % or 1.6 times with 10 % increase in feed-rate. The Tool life of P/M TiN coated taps increased by 175 % or 3.44 times with 10 % increase in speed and feed-rate as compared to the datum parameters. TiN coated taps shows maximum tool life without any damages or dislodgement to the coated surface. Therefore the adherence of coating is found to be strong without any interfacial imperfection between the matrix and the coating. The increased tool life of such coated tools can be attributed to decreased surface roughness values. The improved performance of the drills can be attributed to refinement in carbides which increases wear resistance during cutting. The worn surface of conventionally treated drill shows dislodged cavities and multiple deformed layers as laminated sheets.

Effect of heat treatment on mechanical and corrosion properties of stainless steel

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Stainless Steel used in a wide range of applications including plane, mechanical equipment and railways. Stainless steel is only uses in major industry but can also be used in non-major industry such as watch manufacturing that consists of micro size of elements. Stainless is an iron alloy containing Cr content greater than 12% and other alloying elements, like Ni, Mo etc. Engineering materials, mostly steel, are heat treated under controlled sequence of heating and cooling to alter their physical and mechanical properties to meet desired engineering application. In this study, the effect of heat treatment (annealing, normalising, hardening, and tempering) on the microstructure and some selected mechanical properties of

stainless steel were studied. Solutionisation is the process of heating to 1050^{0}c temperature and air cooled to get single phase homogeneous austenite phase. The steel samples were heat treated in an electric tubular furnace at different temperature levels and holding times and then cooled in different media. The microstructure of the sample was examined using metallographic microscope. The mechanical properties (Rockwell & Brinell hardness) of the treated and untreated samples were determined using standard methods. Corrosion Studies were conducted for Heat treated and untreated samples by using Potentiostatic machine.

Microstructure evolution and incipient melting during two stage solutionizing heat treatment of cast aluminium alloys

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It is always desired that solution treatment at higher temperatures be explored in order to take advantage of faster diffusion kinetics associated with higher temperatures. However, low melting eutectic phases present in the cast microstructure may limit the maximum temperature that can be utilized. Hence, two-stage heat treatments are explored to avoid incipient melting during high temperature solution treatments. In the present investigation, the microstructure evolution and the effect of Cu-content on incipient melting during two stage solution treatment was studied in modified A354 cast aluminum alloys. The samples were subjected to single stage solutionizing heat treatments directly at 520^{0}C for 7 days and two stage solutionizing heat treatment at 490^{0}C for 4 days followed by 520^{0}C for 7 days. The long solution times were utilized to make sure the close-to-equilibrium microstructure is achieved, which in turn is going to be utilized for predictive modeling of the heat treatments of the cast alloys. The experimental results were compared with the results of Thermocalc Software simulations and were found to be in agreement in most cases. Phase Fraction Analysis was carried out by analyzing samples after each stage under scanning electron microscope. Also, Optical Microscopy was utilized to observe microstructural changes and to detect incipient melting. Differential Scanning Calorimetry analysis and hardness measurements were also performed. Incipient melting was detected during 520^{0}C single stage treatment of all the as-cast alloys. The two stage heat treatment could avoid incipient melting at 520^{0}C for the alloys containing up to 3wt% Cu.

Effect of heat treatment on corrosion of nickel base superalloy

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Inconel 690TT is a candidate material for the steam generators in nuclear power plants which operates for more than 40 years under aqueous corrosive condition. This nickel-base superalloy when thermally treated, offers better resistance to IGC and stress corrosion against Inconel 600 under aqueous corrosive environment. Precipitation of a high density of intergranular carbide by aging along the grain boundary would improve the stress corrosion cracking resistance ability. This requires critical aging treatments to be established. The aim of the present study was to determine the degree of sensitization of the alloy 690 aged at various temperatures and soaking time after solution annealed at 1070 °C/6 min. Microstructural characterization of the samples aged from 600 to 800 °C with a soaking period of 1-6 hrs was done. After

stabilizing Open Circuit Potential condition, double loop EPR tests were carried out in de-aerated solution of 0.5 (M) H_2SO_4 with varying concentration of KSCN at a scan rate of 0.0017 V/Sec at room temperature. The peak current value in the activation (Ia) and reactivation (Ir) loops were noted to calculate the Ir/Ia ratio. To elucidate the exclusive effect of Cr depletion as a measure of sensitization during reactivation of the EPR test, KSCN concentration was reduced to a value of 0.001M instead of 0.01 M. The Ir/Ia ratio was found to be 2.4% and 0.019% for the sensitized and the solution annealed sample respectively. The results were correlated with conventional IGC test results. The microstructural investigation showed that the carbide morphology varies dramatically with the aging temperature and duration. The number density of the carbides along the grain boundary increases with the soaking time which is beneficial during operation.

Study of different mechanical and wear behaviors of different carburized mild steel

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The heat treatment and carburization has been acknowledged by some means of improving the various properties of metals and alloys. In the present investigation the mechanical and wear behaviors of mild steel carburized at different temperature range of 900 and 1000°C are studied and it is found that the simple heat treatment greatly improves the hardness and wear resistance of the mild steel. The aim is to examine the effect of these different carburization temperatures and conditions on the mechanical and wear properties of the carburized mild steel. For above purpose firstly the mild steel are carburized under the different temperature range as started above and then it is tempered at 200°C for half an hour after this the carburized and tempered mild steel are subjected for different kind of test such as abrasive wear test, hardness test, carburization test and the toughness test. The results of these experiment show that the process of carburization greatly improves the mechanical and wear properties like hardness, case depth and wear resistance and these properties increases with increase in the carburization temperature but apart from this the toughness property decreases and it is further decreases with increase in carburization temperature.

INDUSTRIAL REALIZATION OF ICME: CHALLENGES AND OPPORTUNITIES

IIM-TMS Symposium on IIM-TMS Symposium on Industrial Realization of ICME: Challenges and Opportunities

PREAMBLE

The carefully chosen title for this symposium is:

IIM-TMS Symposium on Industrial Realization of ICME: Challenges and Opportunities

The challenge we have before us is:

- Redefining the horizons of metallurgy/materials: Focus on automotive, aerospace, defense and energy
- It is now up to us participants to speak to the challenges and opportunities associated the realization of ICME in industry thereby redefining the horizon of metallurgy and materials AND our own commitments to bringing this about.

Session 1: Nov 12, 2014: [13.30 - 15.30]

Theme: Overview of Key Issues in Transitioning ICME to Industry

ICME Interfaces in Practice- Structural, Functional or both

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This talk will explore the importance of interfaces between the models, tools, and methodologies that are often integrated in ICME approaches. Rather than simply perceiving ICME Interfaces as 'structural' through which knowledge/data must be passed, we might also consider ICME Interfaces to be 'functional', where stimuli (questions, assumptions, less-well-known quantities) catalyze new thoughts, directions, and horizons. Such new thoughts, directions, and horizons may either help industry understand the 'risks' associated with ICME approaches and consequently amortize risk through experience or lead to materials discovery. This talk presents this concept in context of integration between the micro-, meso-, and continuum length scales with specific emphasis on properties/performance of end product.

Key Questions

1. How do we capture assumptions made as knowledge is transferred across interfaces?
2. How do we assign "risk" metrics for such assumptions, and 'amortize' risk through experience?
3. How do we identify which questions/less known quantities would, if known, have the greatest impact?

Challenges in Integrated Computational Science and Engineering of Titanium Alloys

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The highly successful applications of titanium and its alloys in safety-critical structures such as those in aircraft and aircraft engines have been largely guided by enlightened empiricism covering the practical aspects of Ti ingot melting and processing into mill products, and secondary fabrication. However a focus on cost-effectiveness and the compression of product development time by effectively integrating design with manufacturing has driven research in recent decades towards a greater predictive capability through the use of computational materials engineering tools. Nevertheless gaps in physics-based models that capture the multitude of various phenomena that govern these engineering processes continue to exist even as models evolve. We have attempted to capture in this presentation several aspects of this evolving effort towards a greater computational and simulation capability supporting the processing–structure–property paradigm of this material system.

Key Questions

1. What needs to be done to get industry to buy into adopting the ICME construct?
2. What needs to be done to increase industry participation in symposia such as the one we are attending this week?
3. What is the efficacy of the ICME construct in precompetitive research for a given industry process?

The Role of Model Integration in Industrial Realization of ICME

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ICME can play a major role in industrial realization of new products or components across various domains. However, we need to overcome several challenges in order to realize the full potential of ICME at an industrial scale. First, any product development involves several steps/unit operations. For successful integration and information flow, it is important to develop rigorous and robust models with abilities to incorporate detailed information from upstream processes and to provide the required outputs to various models of downstream processes. Any weak link can severely impact the gain we envisage in terms of time, cost and feasibility of the product development. Secondly, comprehensive models of unit operations often involve multi-physics, multi-phase and multi-scale models and there are challenges associated with homogenization, robustness, validation, and compute intensive calculations. Even with high performance computing, it will be impossible to use comprehensive models of all unit operations simultaneously and this calls for the development of robust surrogate models. These are some of the important issues related to integration of models across processes and will be discussed in this talk through some examples.

Key questions:

1. How do we accelerate the development of reliable surrogate models?
2. Experiments at various length scales and high throughput experiments are extremely important for industrial realization of ICME. How do we ensure that experiments and modeling go side-by-side?

3. Process integration at an industrial scale requires various enablers and a good enabling platform is one of them. What features and capabilities are needed in the platform that will accelerate industrial realization of ICME?

Session 2: Nov 12, 2014: [16.00 - 18.00]

Theme: Industrial perspectives in transitioning ICME to industry

Using Mathematical Models for Industrial Scale Steel Processing

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Theoretical evaluation of the processes in a steel processing unit leads to faster process optimization, savings in resources and significant improvement in the inherent quality of the products made. While the traditional models used, relied on stoichiometric and basic thermodynamic analysis of the processes, the modern tools based on transport phenomena have enabled the expression of the processes virtually. The steel industries have in particular exploited the computational capabilities to integrate the basic thermo-chemical and physical data to validate the actual processes behaviour. These efforts have in the first place evolved software that can set directions for the industries to optimize the process. This presentation would focus on efforts made in exploiting some of the software for industrial scale processes and the typical results obtained. This includes assessment of fluid flow in tundish, solidification process modeling for optimizing ingot steel casting and continuous casting, deformation modeling of the hot rolling and hot forging processes, heat treatment and sheet metal forming. The experience in utilizing some of the software for industrial problems show that data quality is very important in some cases, computation in some of the calculation are too large for the system to handle, there are difficulties in capturing all aspect of industrial processes which leads to differences in accuracy in prediction. Nevertheless the process sets the direction and indeed enables to find solution to the problems.

Key Questions

1. What efforts are being made to collate, assess and standardize the quality of input data for the models for example thermo-physical properties, constants in recrystallisation equations, Zener Holloman parameters, grain growth parameters, CCT diagrams, etc ?
2. What efforts are being made to integrate cost economics and incorporating bench marks for production processes towards enabling industry to work towards optimizing the raw material and energy?
3. Are there efforts to standardize protocols that may be needed to exploit results from different software to integrated with the ICME platform?

Modified Embedded Atom Method Potential for Ni-Al-Cr-Ta System

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Nickel base superalloys are used in the harsh environment of gas turbine engines because of their excellent high temperature properties. The alloy properties at different length scales are important in the

context of multi-scale materials modeling. Simulations at the atomistic scale using techniques such as molecular dynamics and Monte Carlo are often limited to simple binary systems and a few ternary systems because of the unavailability of interatomic potentials for higher order systems. The engineering alloys, however, are very complex containing many atomic species, and there is a need to generate interatomic potentials for multicomponent systems. The Modified Embedded Atom Method (MEAM) potential is a successfully used semi-empirical potential that describes interatomic interactions in a multi-body formulation including the angular dependency of the electron density.

The present work is an outcome of the interactions at the Indo-US ICME workshop held in Pune in December 2013. In this work, our efforts on the development of a preliminary form of MEAM potential for the Ni-Al-Cr-Ta system will be described. Properties (such as lattice constant, bulk modulus and formation energy) of binary compounds such as Ni3Ta, Ni3Cr, Al3Ta, Al3Cr (all in the L12 structure) and CrTa (B1 structure) were calculated using first-principles based density functional theory calculations employing Quantum Espresso software. Heats of solution for embedding Cr and Ta in Ni3Al were also calculated and the above properties have been fitted into the MEAM model to generate the MEAM interatomic potential parameters. The generated potential has been validated by performing molecular dynamics simulations using LAMMPS software. The potential will be refined further by incorporating other properties into the fitting procedure.

Key Questions

1. What role that multiscale microstructural modeling can play in the engineering design of Nickel base superalloys?
2. Where do atomistic simulations stand with respect to this design?
3. What are the challenges in bridging across length-scales?

Industrial Perspectives on Near-term Applications of ICME Framework

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Value creation in an industrial scenario happens at the intersection or junction point of multiple technical competencies. ICME framework provides unique opportunities to create value in the materials and manufacturing sectors, at the intersection of design (geometry), materials, manufacturing and product-performance competencies within a computational environment. The top two unique values expected out of ICME are accelerated product development and overall cost reduction. Theoretically, it is possible to start from the alloy design and continue to the value chain of product design, development, manufacturing and recycling within this framework. However, focusing on the selection of available alloys, reduction of design factors of safety, and multiple considerations at the design stage provides significant value creation opportunities through the near term applications of the ICME framework. Alloy design has near term feasibility for special applications such as coatings, powder metallurgy or additive manufacturing. In near-term much of the benefits can be derived by boot-strapping the available product design and analysis tools, manufacturing simulation tools, and materials design and processing tools. The present work would discuss the key industrial challenges and demonstrate some of these near-term industrial deployment possibilities which can be realized through the ICME framework.

Key Questions

1. What are the right near-term opportunities for industrial deployment of ICME?

2. What are the methodologies for near-term industrial deployment of ICME?
3. What are the key challenges which would enhance the near-term usage of ICME?

Session 3: Nov 13, 2014: [09.00 - 11.00]

Theme: Computational Materials Engineering

Phase Field Modeling of Microstructural Instabilities

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Within the ICME framework, phase field models occupy a niche between finer-scale or atomistic models (Kinetic Monte Carlo, Molecular Dynamics) and coarser-scale models of transport phenomena (CFD) and deformation (FEM). This mesoscale niche is one where details of microstructural changes can be studied in depth, and consequently, the changes in microstructure-dependent properties can be extracted for further use in the coarser-scale models and techniques. Therefore, phase field models must have the capability to capture, both qualitatively and quantitatively, any material instabilities during processes such as heat treatment. In this talk, we will explore several examples of such instabilities such as spinodal decomposition of an initially homogeneous alloy, thermal grooving of grain boundaries, morphological changes in stressed solid films, abnormal grain growth, dendrite formation, lamellar growth.

The complexity of plastic deformation in polycrystalline materials continues to be a challenge for phase field modeling of microstructural changes during mechanical processing. As the field develops, we can expect these models to offer a viable way of studying instabilities in plastic deformation and crack propagation in materials with complex microstructures, and become a true bridge between atomistic / microscopic models and the macroscopic models in this domain as well.

The Role of Phase-Field Models in ICME

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Integrated computational materials engineering involves the "integration" of a number of material models at different length and time-scales. The goal of the approach is to enable the engineering of a material given the property specifications, through a physical coupling/exchange of information between the different models. As the span of length and time-scales among these methods is over several magnitudes, it is unlikely that a single simulation/modeling method can be constructed which can encompass the physical processes at all the length- and time scales. Hence, the only plausible option is the multi-scale approach wherein information from several models at the different scales enable us to derive the process->structure->property correlations, which is critical to deriving the engineering material of choice.

One of the principal methods relevant at the meso-scopic scale is "phase-field modeling" that has emerged as one of the principal techniques in the study of pattern formation in materials science. In this talk, firstly I will briefly describe the basis of the phase-field formulations existing in literature, and the principal

ingredients required for modeling a given phase-transformation. Additionally, I will briefly touch upon the integration of thermodynamic databases in phase-field simulations which is in the scope of ICME. Consequently, I will describe the principal length and time-scales that are accessible to the phase-field method and the questions related to micro-structure formation that can be addressed through the technique.

Thereafter, I will present an overview of some of the applications of the phase-field method, such as solidification of binary and ternary alloys, solid-state reactions, Ostwald ripening as well as phase transformations involving electrochemical reactions, and mechanical processes such as brittle fracture.

Finally, I will place the phase-field method in the bigger context of multi-scale simulations, wherein I will list the parameters that can be transferred from lower length scales, and the information that can be transferred to higher length scales. I will end the talk stating some of the open questions with regards to modeling and the computational challenges that are present.

Key Questions

1. How do we characterize micro-structure? This is essential in the eventual formulation of structure-property correlations which can be utilized for transfer of information between scales.
2. How do we systematize to construct the solution to the "inverse" problem? Normally, given a micro-structure one can determine the property, but most often it is the reverse question of which structure delivers the property of choice that is relevant to ICME.
3. How do we begin the process of standardization of the various information exchanges between modeling techniques?
4. Incorporation of experimental data is critical to the success of ICME, since this allows the formulation firstly of the right physical questions, and therefore, is vital to the construction of the various models. How can this be systematically done? This also leads to the question of the design of controlled experiments.

Development of Mechanism-Based and Microstructure-Sensitive ICME Modeling Tools

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The key to predicting, and therefore optimizing, properties of materials is the knowledge of the state of microstructure. In the past decade, unprecedented methods to quantify, use and explore grain- and precipitate-scale microstructures have been developed. However, most modeling approaches to microstructure-property relationship utilize highly simplistic descriptors of microstructures (such as average particle size and volume fraction) that are empirically correlated to the properties (e.g., cutting vs. looping). Such approaches are utterly inadequate for addressing the design needs. ICME will remain empirical data driven with limited predicting power and payoffs without the development of next generation modeling tools that incorporate specific transformation and deformation mechanisms operating in specific alloy systems under a given set of processing parameters, microstructure states and service conditions.

In this presentation, we focus on what one could do at the mesoscale to address this difficult issue. In particular, using Ni-base superalloys as an example we demonstrate how to integrate mesoscale modeling with experimental characterization to bridge ab initio calculations and crystal plasticity (CP) simulations to

(a) identify deformation mechanisms and quantify activation pathways and (b) provide "deformation mechanism maps" as function of alloy composition, processing, microstructure and loading condition for microstructure-based CP-FEM models and fast-acting models.

Key Questions

1. Can we actually establish microstructure- and mechanism-based ICME tools today that account for alloy- and location-specific deformation modes operating under different conditions?
2. If so, what kind of collective efforts between modelers and experimentalists as well as among modelers themselves are needed? What are the infrastructure needed?
3. Will the codes, methods and people be too costly for industrial implementation? How to strike a balance between empirical data driven heuristic ICME approach and physics based predictive ICME approach in today's industrial practice? Could we make hybrids of the two?

Session 4: Nov 13, 2014: [13.30 - 15.30]

Theme: Platforms and Infrastructure

ICME in the Cloud: Online Simulations for Research and Education via nanoHUB

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The integration of data from physics-based simulations and experiments within a decision-making framework has the potential to revolutionize the discovery, optimization and certification of materials. Transforming this vision into a reality requires the rapid transition of cutting-edge research codes to researchers who can use them in design and optimization and to instructors training next generations of engineers and scientists. nanoHUB's cyber-infrastructure empowers simulation tool developers to make their codes universally accessible and useful via cloud computing and empowers users who can run hundreds of tools using a web-browser or iPad, free of charge, and without the need to download or install any software nor to provide compute cycles.

Over 300,000 users from around the world use nanoHUB resources every year and 13,000+ of them perform online simulations. I will illustrate how lowering the barriers to powerful simulations is enabling a wave of innovation in materials education and research. Simulations traditionally restricted to expert users are now enabling undergraduate students develop a more intuitive understanding of how materials look and work. In the area of research cloud computing is putting powerful simulation and analysis tools in the hands of researchers and developers who can use them in discovery and design efforts.

Key Questions

1. How do we integrate databases, simulations and experiments into an active "information engine"?
2. How can industry benefit from open cyber-infrastructure such as nanoHUB?
3. How to quantify confidence in simulation results so that can be used together with experiments for decision making?

Towards Plug&Play in ICME – The Need for Standards and Interface Specifications

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ICME by its nature draws on the combination and the simultaneous or consecutive use of a variety of software tools. By now the major developments in the area of ICME have successfully been driven essentially by academic and industrial users of simulation software. In spite of ICME currently emerging as a new and powerful discipline, coupling of different software tools is still in its infancy and represents an issue consuming significant effort in terms of time and workforce if a coupling is realized at all.

The presentation will introduce ICMEg – the Integrated Computational Materials Engineering expert group. This coordination activity of the European Commission aims at developing a global open standard for information exchange between the heterogeneous varieties of numerous simulation tools. The benefits of such a global and open standard for the different communities and the approach being taken by ICMEg towards its realization will be presented.

The expertise of a single institution cannot cover all aspects to be considered when elaborating suitable standard formulations for the heterogeneous variety of models and tools. Thus a collective and global effort of different communities is mandatory. For this purpose a forum will be established and maintained on the ICMEg website www.icmeg.eu.

Key questions:

1. How do we define an ICME language for information exchange between simulation software?
2. Who will take part in this large work of standardization?
3. How do we involve the end users of a Plug-and-Play ICME Platform already at an early stage?

A Model Driven Computational Platform for Integrated Design of Materials and Products

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Synopsis: We discuss the model driven engineering principles that underlie the architecture of a computational platform we are building for integrated design of materials, products and manufacturing processes. The platform combines modelling and simulation with knowledge guided decision making and data driven reasoning for efficient, systematic exploration of design space. When fully operational, the platform is expected to host solutions for a large number of foundational engineering design problems. In this context, one of the key requirements of the platform is its extensibility, i.e. the ease with which one can build new solutions on the platform. Building a new solution has several components to it: building ontologies of required material systems, product configurations and processes; building work-flows; building knowledge engineering blocks and so on. Model driven engineering helps us automate many of these steps. A meta modelling approach provides the foundation for flexible ontology engineering. A process modelling framework provides the means for automating work-flows. A knowledge modelling

framework provides the foundation for flexible, context specific knowledge engineering. We give an overview of these approaches and discuss the underlying principles.

Key Questions - Three key questions, in your opinion, that need to be addressed in fostering the realization of ICME in industry thereby redefining horizons.

Session 5: Nov 13, 2014: [15.30 - 17.30]

Theme: Standards and Workflows

Increasing Accessibility: Automated Workflows and ICME

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Synopsis: A *workflow* is a sequence of computational tasks in which information flows from one task to subsequent tasks via a graph topology, with decision steps at certain graph nodes. The importance of workflows are that they can be used to automate certain computational tasks, such as data analysis. The same types of information flow occurs between tasks in an ICME process, in which the information consists of data, models and simulations.

As of now, creating workflows requires experts, including experimentalists, modelers, designers, data managers and so on. Many companies, especially smaller ones, may not be able to afford the infrastructure needed for successful application of ICME. In this talk I will argue that to extend the impact of ICME, efforts should be focused on creating standardized workflows for common ICME processes. I will discuss what creating such workflows will require and what are the potential roadblocks.

Key Questions

1. What can we do to make ICME processes more accessible to all companies?
2. What form should ICME workflows take and what information would be needed?
3. What kind of education and resources would be needed to implement ICME workflows in industry?

ICME platforms for Design Collaboration – Thinking beyond Software Integration

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Synopsis: ICME for integrated realization of engineered materials and products involves collaboration across multiple teams within an organization, and often collaboration among multiple organizations. Such collaborations necessitate the development of platforms that can manage workflows and information flowing across organizations. In this presentation, the key issues in collaborative workflow management will be discussed. For example, the bandwidth of information exchange may also be restricted due to network limitations. Further, collaborating entities may be unwilling to share all details about their designs. Such issues go beyond the standards for information exchange between software tools.

Due to the privacy and intellectual property reasons, the information flow across organizations may be severely restricted. Hence, there is a need for techniques for efficient information hiding for collaborative workflows. Potential strategies include using game-theoretic models (e.g., exchanging rational reaction sets) and response surface equations to hide the details of models from the collaborators. The collaborative nature of the workflows induces additional requirements on the management of design workflows. There is a potential for inconsistencies in information generated by collaborating entities. These inconsistencies can be in requirements, assumptions about the system, levels of fidelity of models, and the manner in which resources are allocated. Hence, ensuring consistency across the parts of workflows developed by different collaborative entities is essential.

Key Questions

1. What are the challenges in facilitating multi-organizational collaborations in ICME for integrated realization of engineered materials and products?
2. What are the essential features of computational platforms to support design collaborations in ICME?
3. What are the strategies for addressing some of the challenges related to collaborative workflow management?

Integrating the ICME toolset in the emerging landscape of computing infrastructure

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The challenges in bringing ICME to the industry practice are multifaceted and depend on the context of the application. In my talk, only the computational tools are considered. The toolset should not only be customizable as the industry requires and extendible as the researchers desire but also integrable for an increased adoption of ICME. In the recent past, the landscape of storage and computing has been undergoing significant changes. A layer of abstraction that aids in portability is emerging. High performance computing at affordable cost is now being offered by a heterogeneous set of processors. Large amount of storage and bandwidth at low cost are changing the way data is being handled. I take a case study of casting simulation and refer to the automotive toolset of the ICME document from TMS to illustrate the gap in the development of computational infrastructure and the technical software. A large number of researchers must come on board to contribute to the integration of the toolset and to mature it for adoption by the industry. This requires removal of barriers in the acquisition and use of the toolset by the developers. The recent advances taking place in application virtualization and streaming can potentially address the challenges faced by research community in this direction. The role of documenting and standardizing workflow will also be discussed in this context. These changes could potentially change the way the education sector adopts ICME.

Key Questions

1. What are the characteristics of a fresh graduate to be able to contribute to the research community of ICME?
2. What needs to be done in India to get an ecosystem like ICMEg project of EU going here?
3. Who should be nucleating the standardization of workflow for simulations in ICME?

Session 6: Nov 14, 2014: [08.30 - 10.30]

Theme: Design and Data Sciences

ICME for the Integrated Realization of Engineered Materials, Components and Associated Manufacturing Processes (ICME-IREMCAMP)

Farrokh Mistree,
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Synopsis

Let Us Speculate

Imagine that all the models needed for ICME exist, the data and computational resources to execute them is available.

The Question

What is needed to facilitate rapid and robust concept exploration when the models are incomplete and possibly inaccurate?

Key Questions

- Single Decision, Single Model
  - How accurate should the information (models) be?
  - How do we trade off accuracy versus cost?
- Multiple Decisions and/or Models
  - How should the design process network be structured?
  - Which models should be coupled?
  - Which decisions should be coupled?
  - How should the decisions be sequenced?
- Multiple Decisions across Organizational Boundaries
  - How should issues of privacy and security be addressed?
  - How should we facilitate collaboration between individuals and across corporations?
- What functionalities are needed for the integrated collaborative creation, modular reuse, and execution of decision workflows in distributed engineering settings?
- In addition to standard interfaces between software tools what mechanisms / protocols are needed to ensure execution by different collaborative entities?

Integrated Realization of Engineered Materials, Components and Associated Manufacturing Processes (ICME-IREMCAMP)

Janet K. Allen.
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Synopsis: One of the major difficulties in ICME based integrated design of materials and products is the ability to manage uncertainty in the simulation-based design process, The problem of managing uncertainty is significant because all models and experiments are uncertain and uncertainty may get amplified as the models are linked into chains/networks. Designers can tolerate uncertainty as long as it

does not significantly impact the design outcomes. Hence, the need to develop a method to manage uncertainty. One such method is robust design. The principal question that needs to be addressed is:

How can we manage various types of uncertainty in a distributed, collaborative platform such as PREMAP?

Key Questions

1. In a simulation-based design process what types of uncertainties need to be quantified and taken into account in a simulation-based design process?
2. Robust design Types I, II, III, and IV
3. What surrogate models should be used to reduce the cost of computation?
4. How can we reuse surrogate models in robust design?
5. How do we know whether a result is valid or useful?

Data Sciences Applications for Industrialization of ICME

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Synopsis: While the primary research focus of the ICME community has been towards developing better models for computational materials engineering, it has also been realized that data sciences would play a major role to bridge the gap between the development of physics based models and experimental observations for acceleration of adoption of ICME in the industry. Besides this, physics based simulations are computationally expensive and use of data science models leveraging smaller set of physics based simulations for exploration of design space would be of immense utility to the industry. Finally, the industry would be more interested in addressing the question of "what needs to be done to achieve the target material properties/ product performance?" and this requires solutions to inverse problems across a chain of processes. I will discuss our research in the area of data sciences for developing statistical and machine learning based models for prediction of material properties/performance from experimental as well simulation data and also discuss our work in the area of inverse inferencing. In addition, I would also discuss some progress our team made in the area of text mining over materials publications. Finally, I conclude my discussion with the challenges and possible ways forward in the application of data sciences for industrial application of ICME.

Key Questions

1. How do we leverage a combination of experimental data and physics based simulations together for better predictive models using data sciences based modeling?
2. How well can we make inverse inferencing while the properties/performance of the material involves a long chain of steps with large number of controllable variables?
3. Can we use text mining methods towards accelerating the development of ontological models required for standardization for ICME needs?

Contributory papers

Session 1: Nov 12, 2014: [13.30 - 15.30]

An inductive method for the exploration of the solution space of ladle, tundish and caster

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In order to meet stringent requirements emanating from environmental norms, safety norms and stiff competition, auto manufacturers are demanding improved performance and reduced defect levels from steel makers. The defects are dependent on the design of unit operations, and the processing conditions in the ladle, tundish, and the caster. In order to improve performance and reduce defect levels, steel plants need to systematically design the entire process considering multiple unit operations simultaneously. However, the solution to this problem is very complex; especially when multiple unit operations are involved. This complexity is further augmented in the presence of uncertainty in the parameters and models used.In this paper, a computational method to facilitate controlling the input to the ladle and the desired output from the caster (and vice versa) is presented. The decisions associated with ladle, tundish and slab continuous casting unit operations are modeled using the multi-objective decision construct of compromise Decision Support Problem (cDSP). Within the cDSPs, the required properties and tolerable defect levels for the continuously cast slab and design set points for ladle, tundish and continuous casting are specified. Each cDSP integrates the behavior of the standalone unit operations using physics-based reduced order models that are valid for process parameter windows. The cDSPs are computationally instantiated for the three unit operations. An inductive approach is adopted for jointly exploring the solution spaces of the three unit operations. Within the proposed inductive approach, the properties and defect level requirement flows backward from continuous caster to tundish to ladle, and the satisficing set points flow in the forward direction from the ladle to tundish to the continuous caster. In this paper, the design set points obtained using this approach for ladle, tundish and continuous casting of slab are presented. The primary advantage of the proposed approach is that it enables rapid exploration of the entire steel slab production process.

Context aware information retrieval from materials publications

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Relations between material compositions, manufacturing processes and material properties are of significant interest to materials scientists and engineers. A large amount of information of this nature is present in publications especially in the form of experimental measurements, simulation outcomes, etc. In a typical problem solving context requiring information not available in standard databases, an engineer has to first go through a large collection of publications to filter the right set of documents, containing information relevant to the context. Having filtered the publications, an engineer has to then go through each of them to extract the relevant pieces of information. Our goal is to help automate some of these steps. In this paper, we present a system that provides information search and extraction based on material compositions where the composition is represented as a set of elements and their weight percentages. The repository of material publications is first pre-processed to extract compositions. The extracted compositions as well as the publication content are then indexed for use by the search system. To specify search criteria on element values, the system provides a domain specific query language. It supports “and”, “or” and “not” operators using which complex search queries can be formulated. The accuracy of our system critically depends on the accuracy of the information extraction component. To address this, we have developed a rule based mechanism for composition extraction. The experiments are conducted on a small library of publications on steel on which searches such as “get the list of publications having carbon composition between 0.2 and 0.3” are performed. Experiments show that the extraction component achieves a precision of 90% and a recall of 85%. We compare these results with results from a keyword based search approach.

Invited talks

Session 1: Nov 12, 2014: [13.30 - 15.30]

Challenges in Dissimilar Metal Welding

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A wide variety of materials are available to today's engineers from which they can manufacture increasingly complex products. Each particular material has its own distinct properties which makes it attractive for usage in the application under consideration. It is seldom the case that any material will satisfy all the requirements for an entire product. This results in the need to join dissimilar materials across industry sectors. The current environment of increased customer demand for products of enhanced performance, increased quality and reduced cost drives designers to devise individual components from different materials and then join them together to make multi material finished articles. A wide range of joining techniques can potentially be applied to dissimilar welding applications, with the choice of approach depending on the combination of materials to be welded i.e. metallurgical aspects and the geometry of the components/joints involved i.e. design aspects.This paper presents few case studies involving dissimilar metal welding in heavy equipment fabrication & the associated metallurgical issues in producing the right weld joint for the intended application.

Microstructure and Residual stress of Interpulse v/s TIG welded Ni based superalloys

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Nickel based superalloys used in gas turbine hot gas path components undergo repair and refurbishment, as part of regular maintenance. These alloys often have limited weldability, owing to the highly alloyed nature of these superalloys, especially related to their Al/Ti ratio and precipitation kinetics. In some cases, liquation cracking, solidification and strain age cracking, can make these alloys practically unweldable, and make them difficult to use after repair and refurbishment. Sometimes elevated temperature welding, a somewhat cumbersome process is employed, to weld repair these superalloys. In recent times, Interpulse welding is a low heat input derivative of the GTAW (Gas Tungsten Arc Welding) or TIG (Tungsten Inert Gas) process, has been employed for welding these difficult to weld alloys. Here the width of the arc is constricted by using advanced power electronics and by pulsing the arc 20,000 times a second in the weld pool. This results in a concentrated arc with minimised heat input, resulting in a lower bead width and HAZ. In this study, the weldability of serviced exposed Ni based superalloy, GTD111 has been studied using Interpulse welding vs the regular TIG welding technique, at ambient temperature. Three different filler wires have been tried, GTD111, Haynes 230 and Haynes 282, and the feasibility of Interpulse welding demonstrated via detailed microstructural characterization and residual stress measurements.

Session 2: Nov 12, 2014: [16.00 - 18.00]

Probing tool longevity in friction stir welding through process modeling

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The tool pin in friction stir welding is structurally weaker than the shoulder and undergoes severe stresses at high temperature especially during welding of stronger and thicker alloys and at higher welding speed. The simultaneous rotational and linear motions of the tool during welding are responsible for combined bending and shear stresses. An estimation of the maximum resultant shear stress on the tool pin is needed to avoid premature failure and ensure greater longevity of the friction stir welding tool. As the tool pin remains immersed into the plasticized workpiece materials, an experimental evaluation of the stresses on the tool pin is difficult in real-time. It will be shown as a part of this work that the stresses on the tool pin can be estimated following a numerical process model to compute the temperature field and mechanics based relations. The estimated values of the maximum resultant shear stresses on the tool pin for several welding conditions can be used further to develop process maps for tool longevity that can help appropriate selection of welding conditions avoiding premature tool failure. Such process models can also help in design of friction stir welding tools to achieve better weld joint quality.

Role of Welding Processes in the Nuclear Fuel Fabrication at NFC

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Complicated and large components fabricated with different materials and joining techniques are used in wide range of engineering industries. The ability to construct and operate such plants depends on the application of welding technology in a major way. Among all engineering processes, welding process plays an important and significant role in the fabrication, erection and commissioning of different engineering plants and their machinery. Automated and sophisticated welding methods ensure near zero failure probabilities, resulting in reduced cost and fabrication time. In the recent past, interest has been revived in the solid phase welding due to its unique advantages, such as amenability to automation, welding of dissimilar metals, absence of filler material and lower heat input. The quality requirement of these welding processes is very stringent and has to meet all the product requirements. Several improvements have been carried out over the years in the manufacturing of fuel assemblies, which helped in improving the fuel performance considerably. Nuclear Fuel Complex is engaged in the manufacturing of both PHWR (Pressurized Heavy water Reactors) and BWR (Boiling Water Reactor) fuel assemblies for all operating reactors in India. At present there are 17 numbers of PHWRs and 2 numbers of BWRs. NFC mastered the fuel manufacturing technology and supplying the required fuel to all operating power reactors since its inception. Zirconium alloy is the main structural material used in the PHWR and BWRs because of its low absorption cross-section for thermal neutrons and also due to various other nuclear, mechanical, metallurgical and chemical properties at elevated temperature. The fuel assemblies (referred to as “bundles”) are composed of a number of individual cylindrical elements joined together.

Session 3: Nov 13, 2014: [09.00 - 11.00]

Electron beam welding technology in defence applications

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The current scenario in defence and aerospace applications demand metal joining processes which can effectively perform welding of exotic and reactive materials with precise control of heat input. These applications require processes that can weld high strength materials producing high joint efficiencies. Electron beam welding (EBW) is one such process widely used in defence and aerospace industries, due to its inherent advantages like lower & precise control of heat input, minimal distortion and higher joint strengths. The present talk covers various aspects of electron beam welding process, the special characteristics of electron beam, the beam formation methodology, the art of key hole welding, the classification of EBW systems in terms of vacuum, voltage, gun position, and various EB joint configurations. The important parameters to be controlled during welding and their influence in joint penetration are discussed in detail. The talk includes diverse factors to be considered in getting quality weld joint different acceptance standards used in this process. The special characteristics of the process are effectively exploited to produce good quality EB weld joints in different defence applications. Different critical jobs carried out in DRDL using two different types of welding systems are explained in detail involving different materials.

Contributory papers

Session 1: Nov 12, 2014: [13.30 - 15.30]

Dissimilar joining of Al-2014 and Ti alloy by cold metal transfer

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Al-Ti joints are of interest in several applications in aerospace industry. Recently, there has been increased interest in joining Ti and Al alloys. In this study, dissimilar joining of Al-2014 and a near αTi-alloy was done using cold metal transfer (CMT) welding process. CMT process is a recent invention and is a variation of conventional GMAW welding technique. CMT offers advantage of low heat input reducing the amount of intermetallic compounds formed in dissimilar joints. A 1.2 mm diameter commercially available Al-Si-Mn filler wire was used. Different lap joint orientations with Ti alloy and Al alloy alternatively on top was used for welding. CMT process leads to welding of the bead with Al plate while brazing occurs with the Ti-plate. The intermetallic layer which was formed in brazing part of Ti has been studied by optical microscopy and scanning electron microscopy. Hardness along the fusion zone to base material has been studied. The effect of the orientation on the mechanical properties of the lap joint has been studied.

Gas tungsten arc welding of ZrB_2 based ultra high temperature ceramic composites

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The zirconium diboride (ZrB_2) based ultra high temperature ceramic (UHTC) composites are attractive for aerospace applications: thermal protection systems, leading edges, and propulsion components for hypersonic flight vehicles. ZrB_2 composites are generally fabricated by hot-pressing or pressure less sintering. Complex shapes or large size components of ZrB_2composites can be fabricated by assembling small pieces via joining/welding. Gas Tungsten Arc Welding (GTAW) without and with filler material has been used to join ZrB_2 - 20 vol. % SiC (ZS), and ZrB_2 - 20 vol. % SiC - 8 vol. % B_4C - 7 vol. % YAG (ZSBY) composites to themselves. Without filler material the fusion welding has lead to cracking and non-uniform joining. With suitable filler material containing (ZrB_2-SiC-B_4C-YAG) both (ZS) and (ZSBY) could be successfully welded. Severe cracking and oxidation was observed during GTAW welding of the (ZS) composite. The (ZSBY) composite exhibited excellent thermal shock resistance and oxidation resistance. The weld interfaces for both the composites were very clean and coherent. The Vickers micro hardness across the welding was found to increase from 12.53 GPa to 17.83 GPa and 17.18 GPa to 24.93 GPa for (ZSBY) and (ZS) composites respectively. The shear strength of (ZSBY) joint was as high as 100 MPa.

Fusion and microstructural characteristics of laser- MIG hybrid welded maraging steel

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Laser arc hybrid welding is a relatively new process which effectively combines the advantages of arc welding and laser welding. Maraging steels are ultra-high strength steels combined with good fracture toughness and are widely used in aerospace industry such as for rocket motor casings having 8 mm nominal wall thickness. Welding of such thicknesses with conventional arc welding requires 3 to 5 passes which means repeated heating of the material, aggravating the weldability issues. Using laser arc hybrid welding, steel sections up to 12 mm thickness have been successfully welded in a single pass at a welding speed of 1 m/minute using a 3.5 kW CO_2 laser in combination with a MIG arc. In order to evaluate the applicability and possible practical metallurgical advantages, laser arc hybrid weldability of 10 mm thick maraging steel plate of 250 grade was investigated. Initially bead-on-plate experiments were conducted to understand the effect of various parameters such as laser power, MIG wire feed rate, shielding gases by keeping welding speed constant. Based on the fusion behaviour observed, a set of parameters were chosen to carry out butt welding. Butt welded specimens could be successfully made in single pass. Metallography, microhardness survey across the weld and mechanical testing were carried out. Fusion behaviour vis-à-vis welding process parameters and mechanical behaviour vis-à-vis microstructure observed will be presented. To summarise, it was found that laser hybrid welding of maraging steel is not only feasible but has certain practical and metallurgical advantages.

A-TIG welding of austenitic stainless steel

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Activated fluxes that increase weld penetration as compared to conventional GTAW process were first reported in 1960's by the Paton Welding Institute (PWI), Ukraine. The use of these fluxes has distinct advantages when compared with the conventional GTAW process. Apart from the readily apparent advantage of productivity improvement, using these fluxes results in less heat input which leads to reduced distortion in components being welded. In the present work the influence of an in-house developed activating flux on the mechanical and microstructural properties of austenitic stainless steel welds is described.

Weldability of ADI using SMAW and TIG

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Austempered Ductile Iron (ADI) is an ausferritc microstructure, which consists of bainitic ferrite and high carbon austenite. The microstructure is the resultant of two step treatment process. The samples are austinitized at predetermined temperature and time followed by a designed process of austempering. The different sets of heat treatment schedules results in different grades of ADI. These grades in combination produce high strength and ductility, fatigue strength and wear resistance etc. Even after being researched so heavily and studied so deeply, this material is unable to replace convincingly the material for their welding applications. This paper describes the weldability of ADI. Two types of welding are performed on the sample: Tungsten Inert Gas (TIG) welding and Shielded Metal Arc welding (SMAW). The heat affected zone (HAZ) and the partially melted zone (PMZ) of the respective welded samples are analyzed and comparisons are drawn for microstructure, tensile strength and hardness profile. Comparisons between the data recorded and the data from non-welded ADI are drawn. The control parameters, thickness of the electrode, type of the electrode, time of welding, welding speed and the results from the case studies have been included in the present study. Predictions are made on the basis of the above mentioned tests to determine a process best suited for welding ADI.

Influence of shielding gas mixtures on microstructural and mechanical properties of gas metal arc welded modified ferritic stainless steel

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The present study describes in detail the effect of shielding gas mixtures on the bead geometry, microstructure and mechanical properties of gas metal arc welded modified ferritic stainless steel (SSP 409M) sheets (as received) of 4 mm thickness. The welded joints were prepared under spray (S) mode of metal transfer using 308L austenitic filler wire of 1.2 mm diameter and four different shielding gas mixtures i.e. pure Ar, Ar+5%CO_2, Ar+10%CO_2 and Ar+20% CO_2. The welded joints are evaluated by means of microstructural changes, hardness, tensile strength and toughness. The dependence of weld metal microstructure on shielding gas mixtures has been determined by bead geometry, Cr_{eq}/Ni_{eq} ratio, M_s, M_{es}, optical microscopy (OM), transmission electron microscopy (TEM) and electron probe microanalyzer (EPMA). It was observed that the variation in shielding gas mixtureeffectively manipulates the solid-state phase transformation and precipitation behavior of the welded joints. Variations in microstructure ultimately affect the mechanical properties of the weld metal as well as high temperature HAZ (HTHAZ). The present study concluded that up to 10% CO2 with Ar can be commercially utilized in the shielding gas mixture for preparing a welded joint of 409M with 308L filler wire without deteriorating microstructural and mechanical properties.

Temperature profiles in welding maraging steels

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Maraging steel are the frontrunners of the ultra-high strength steel family with superior properties like high strength and toughness derived due to martensitic transformation and subsequent ageing. In manufacturing of maraging steel equipment, welding is a process which is found to be frequently used. The reason is that maraging steels are readily weldable over a wide range of thickness and very high weld efficiencies (90-95%) can be achieved. To predict the post weld properties and microstructures, the knowledge of temperature distribution is essential. In order to predict the temperature, modelling of a GTAW process on Ni-250 maraging steel was performed using multi-physics software, COMSOL. The heat source was modelled as a Gaussian heat flux distribution on the weld surface. 3D models were made in COMSOL with moving heat source. The results of the FEM model were compared with the welding experiments that were conducted on the 8 mm and 2 mm thick Ni-250 maraging steel. Thermocouples were fixed at specific locations on the top surface and the temperature profiles at different locations were obtained using a data acquisition system. Post weld analysis includes microstructure of the weld pool and heat affected zone with a hardness survey across the weld.

Study of dissimilar metal welding of stainless steels and their property assessment

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Dissimilar metal welding is very much popular in many industries like desalination, petrochemical, marine and nuclear plants, but the welding of dissimilar metal is very difficult than similar because of physical, chemical and thermal changes. Therefore, it is required to improve the properties of these welds. In this work the main focus is on microstructural development and mechanical properties assessment of

dissimilar weld of type 2205 and 316L. The butt joint was carried out by shielded metal arc welding with different heat input range (low, medium and high). Weldment characterization was investigated by electron microscopy, X-ray diffraction and energy dispersive spectrometer. Mechanical properties were evaluated with the help of tensile and microhardness test. Intergranular corrosion assessment was carried out by using electrochemical test.

Development of stellite surface coating on H13 tool steel using cold metal transfer (CMT) welding process

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AISI H13 is one of the extensively used hot working tool steels for making hot forging dies owing to its good mechanical and thermal properties. Aggressive loading cycles and high temperature exposure cause failure of these dies in various ways. Among the various modes of failure of hot forging dies about 70% consist of abrasive and adhesive wear. Therefore, it is a general practice in the industry to protect critical parts of hot forging dies against wear by surface coating techniques. Cobalt based alloys bearing the trade name Stellite are well known for their high temperature wear and corrosion resistance and are widely used as a surface coating material. Weld overlay techniques result in good quality coatings which are metallurgically bonded to the substrate. Selection of welding process depends on amount of dilution of coating by the substrate material. Dilution of coating is proportional to the heat input of the welding process. Cold metal transfer (CMT) is a recently developed variation of the gas metal arc welding (GMAW) process. In CMT, the material transfer and electrical characteristics are controlled independently resulting in low heat input. The lower average value of welding current helps in realizing the lower heat input of the process. In this work, CMT process has been used for weld surfacing of stellite 21 on H13 tool steel. The CMT process parameters are optimised to get minimum dilution and maximum hardness of the coating. Microstructure and hardness of the coating are studied. The wear resistance of the coating is studied by using pin-on-disc wear testing method.

The influence of double pulse on bead profile and microstructure in GMAW of low carbon steel

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Pulsed GMAW (P-GMAW) has been recognized as a new and efficient technique for quality welding. The double-pulsed GMAW (DP-GMAW) technique is a variation of the pulsed GMAW technique, in which the pulsing current aimed to metal transfer control is overlapped by a thermal pulsation, which in turn means pool control. Despite some advantages claimed by this reasonably new technique, one could expect optimum quality with the selection of suitable process parameters. Thus, in this work a bead on plate welding on a low carbon steel plate at different parameter combinations is presented. The bead profile and microstructure of the weld metal is thoroughly studied. Microhardness, inclusion formation and volume fraction of the acicular ferrite is calculated for correlating the process parameters with the desired weld quality. The results indicate that the DP-GMAW technique maintains the capability of enhancing the

penetration and the microstructural property of the weld and its advantages can be better exploited in industrial applications.

Welding process and procedure for extra narrow gap dissimilar welding of thick section of austenitic stainless steel to high strength low alloy steel

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Dissimilar metal welds between austenitic stainless steel (γ-SS) and, high strength low-alloy (HSLA) steel are widely used in high-temperature applications in power stations and petrochemical plants. In this study the weld isotherm for dissimilar weld joint of austenitic stainless steel and high strength low alloy steel has been discussed for production of multi-pass single seam per layer narrow gap pulse current gas metal arc (GMA) weld joint of thick dissimilar plates free from any lack of fusion. For production of a sound weld joint, the role of pulse current gas metal arc welding (P-GMAW) has been also discussed. The weld isotherm of weld pool is very useful to decide the pulse parameter to produce the sound weld joint. The width and length of the weld pool isotherm indicate the proper selection of pulse parameter without lack of fusion to the both side of groove wall, especially in dissimilar weld joint. The model is experimentally verified by preparing weld joints of austenitic stainless steel and high-strength low-alloy (HSLA) steel using different combinations of pulse parameters.

Session 2: Nov 12, 2014: [16.00 - 18.00]

Stress corrosion cracking behaviour of fusion and solid state maraging steel (MDN-250) weldments

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While most of the friction stir welding (FSW) efforts to date have involved joining of Al alloys, there is considerable interest in extending the technology to other materials, including steels, since FSW appears to offer several advantages over arc welding processes. Maraging steels, a class of low carbon high alloy ultra high strength steel, find its application in aerospace, military and other critical applications due to its unique combination of performance characteristic- high strength, high toughness and good formability. Higher the strength more is the material susceptible to stress corrosion cracking (SCC). The present investigation is aimed at understand the SCC behaviour of fusion (gas tungsten arc) and solid state (friction stir) maraging steel (MDN-250) weldments. SCC test was carried out through slow strain rate test method at a strain rate of $4x10^{-6}$ mm/s. Base material and weldments were tested in as-weld and post weld aged conditions. The environment selected for testing was air and 3.5% NaCl solution. From this investigation it is found that friction stir weldments are more resistant to SCC in comparison to base metal and gas tungsten arc weldments. Higher SCC resistance of friction stir weldments is mainly due to the formation of very fine equiaxed microstructure in the stir zone and absence of reverted austenite. Low SCC resistance of

gas tungsten arc weldments compared to friction stir weldments may be attributed to coarse grain structure, segregation of alloying elements and presence of reverted austenite in the martensitic matrix of the fusion zone.

Comparative study of solid state welding and fusion welding for age hardenable AA 7075 aluminium alloy

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High strength age hardenable 7xxx series aluminum alloy such as AA 7075 is commonly found in several key components of aircraft and automobiles. These alloys are difficult to join by conventional fusion welding techniques. Hence realizing a weld joint in such alloys without impairing the mechanical properties is a difficult task. The 7xxx alloys are among the Al-Zn-Mg-Cu versions provide the highest strengths of all aluminum alloys. AA 7075-T6 aluminium alloy plates with thickness of 3mm were butt welded using solid state welding and metal inert gas welding (MIG). The joints were compared in terms of electrical conductivity (% IACS), a physical property of aluminium alloy used to define relationship of changes in alloy composition and metallurgy. The same joints were also assessed for hardness and micro structural properties. The results show that the solid state FSW joints have higher electrical conductivity and hardness than MIG joints. The width of heat affected zone of solid state FSW is narrower than the MIG fusion welds indicating better mechanical properties. The correlation between electrical conductivity and the hardness in different weld regions was also investigated.

Effect of carbon nanofillers addition on shear strength of adhesive bonded joints

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Adhesive joints of Aluminum alloy 6061-T6 were prepared by using an epoxy resin as the adhesive. In order to enhance the lap shear strength of epoxy adhesive joint, carbon nanofillers such as multi-walled carbon nanotubes (MWCNTs), graphene nanoplatelets (GNPs) and carbon nanohorns (CNHs) were dispersed in the epoxy matrix. Surface treatment of AA6061-T6 substrate was done by grit blasting using aluminum oxide grits. Surface roughness measurements showed higher surface roughness for grit blasted substrate. Contact angle measurements showed higher surface energy for surface treated substrates which provides good wetting and better adhesion. The nanofillers of different amounts 0.2, 0.5, 1 and 2 wt.% were dispersed using Brabender blending machine. The steady state viscosity investigations showed increased viscosity and shear thinning behavior for MWCNTs/epoxy, GNPs/epoxy, CNHs/epoxy

suspensions with increasing filler concentrations at different shear rates. The adhesive lap joints were tested using the ASTM standard test D1002. It was found that the lap shear strength of the adhesive bonded joints increased with increase of MWCNTs, GNPs and CNHscontent up to 1 wt.%, 0.5 wt.% and 0.5 wt.%, respectively. The results also indicated that increasing in the amount of the carbon nanofillers beyond a certain weight fraction of the adhesive reduced the strength of the joint. Fractured surfaces observed using a scanning electron microscope (SEM) showed that the nanofillers were embedded in the epoxy matrix. It was observed that the dispersion of the carbon nanofillers was poor at higher content. Of the three nanofillers, CNTs are found to be most effective at higher loading while GNPs were better at lower loading of the reinforcement.

Friction welding of dissimilar materials between semisolid processed A356 and 6061 aluminium alloy

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Joining of aluminium alloy component with conventional fusion welding creates voids, hot cracking, distortion in shape, evolution of dendritic microstructure which ultimately would lead to inferior mechanical properties of the weld region. On the other hand, with friction welding which is a solid state joining process, all defects associated with melting and solidification in a typical fusion weld can be avoided. This work investigates the weldability of semisolid processed A356 and 6061 aluminium alloy with friction welding. The study is carried out experimentally as well as numerically. In order to numerically model the friction welding process, temperature dependent physical properties and mechanical properties as well as viscoplastic constitutive equations of semisolid processed A356 and 6061 aluminium alloy were incorporated in the FEM model. The modelling is based on coupled thermomechanical approach fields. Thermomechanical modelling helps in deciding the suitable range of process parameters. These parameters play a significant role in temperature variation, stress distribution as well as distortion during the welding process which ultimately has an impact on weld quality. Moreover, the detailed microstructural analysis and mechanical testing of the welded samples were performed. The weld strength was found stronger than the parent materials. Microstructure study shows that friction welding of dissimilar materials helps in achieving the fine microstructure at the interface which has an impact on improving the mechanical properties.

Characterization of a CFRP butt-joint between the pipes of dissimilar materials and experimental studies on CFRP butt joint under leak pressure test

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A butt-joint was formed between two pipes of dissimilar materials, steel and aluminum by winding a wetted roving of carbon fiber with epoxy at ±45° angle. On the curing of the epoxy, a tight carbon fiber

reinforced polymer (CFRP) sleeve was formed, joining the ends of the pipes. The CFRP butt-joint was tested for two kinds of loads: tensile and bending. Based on the performance, the specimens were categorized into two groups, thin and thick CFRP sleeved specimens. In the tensile testing, the thin sleeved specimen failed through the breakage of the CFRP sleeve at the joint plane because the axial stress developed in CFRP sleeve exceeded the ultimate strength of CFRP. However, the thick sleeved specimens resisted the axial load in the sleeve and the weaker adherend, the aluminum pipe, slipped out of the CFRP sleeve. In the flexural testing, the thin CFRP sleeved specimens also failed by failure of the CFRP sleeve at the joint plane while the specimens of thick CFRP sleeve failed by the formation of a plastic hinge near the edge of the CFRP sleeve. Experimentally obtained results were explained with numerical analysis using ANSYS by determining axial, radial, and shear stresses under tensile and flexural loading conditions. A pressure oil leak test was performed on a CFRP butt-joint formed between two adherends of aluminum pipe using a dead weight pressure gauge tester to determine the pressure at which CFRP failed when the number of CFRP passes was varied. Specimens with lesser number of passes, the oil leaked through the CFRP sleeve near the joint plane. As the number of passes increased, the oil pressure expanded the CFRP sleeve and oil leaked through the sides. With the further increase in number of passes, no leak was detected till the aluminum adhere and start yielding.

Microstructure and mechanical properties of AZ91D magnesium alloy sheets jointed by friction stir welding

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The present work was carried out to understand the microstructural development during friction stir welding process of Mg based alloys, and to evaluate the mechanical properties of the welds. AZ91D Mg alloy plates having 3mm thick are FSWed under a constant rotational speed of 720 rpm with varying welding speeds ranging from 25 to 75 mm/min. Welding defects are analyzed by visual inspection and radiography tests and defects are observed at welding speeds of 25 mm/min and 75 mm/min. Good quality weld is obtained at traveling speed of 50 mm/min. The microstructure of the weld zones were analyzed using scanning electron microscope and optical microscope. The original dendrite grain structure is completely disappeared and became fine equiaxed grains in stir zone (SZ). The microstructure of each zone shows different features depending on its thermal and mechanical conditions. In the stir zone hardness increased slightly because of the fine recrystallized grain structure. The tensile strength of about 105 MPa obtained under welding speed of 50 mm/min.

Friction stir welding of high strength low alloy steel: A feasibility study

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Friction-stir welding (FSW) has been a well established solid-state joining technology for joining of aluminum (Al) alloys and other soft metals. However, this process possesses significant challenges for metals with high softening temperature such as steel and titanium (Ti) primarily because of non-availability of super abrasive and refractory tool materials capable of withstanding severe welding conditions. Recent development of tool materials with properties that can withstand the high torque and high temperatures (approximately 900°C) have facilitated joining steels with FSW. In the present work, three types of refractory-based alloys tool materials are utilized i.e., 90% tungsten (W) and molybdenum (Mo), 95% tungsten (W) and molybdenum (Mo) and 99% tungsten with 1% La_2O_3. Experimental trials revealed that the tools made from 99% tungsten with 1% La_2O_3 tools were able to withstand the high torque and heat generated. Severe tool shoulder deformation and mushrooming and tool pin breakage are common mode of failure observed for tools fabricated with 90% W and 95% W. The tool shoulder and pin profile was optimized and joint fabricated using a tool with the shoulder diameter of 25 mm having pin length 6.5 mm yielded defect free joint with improved mechanical properties (better than the base metal) without compromising the toughness. The microstructure characterization was carried out using optical and SEM-EBSD technique for the stir and thermo mechanical affected zone (TMAZ), shows the presence of ferrite lath dispersed in bainitic matrix in the stir zone. However, the TMAZ indicate microstructure consisting of acicular ferrite and lath martensite, which was reflected by the higher hardness value in the advancing side (356 Hv) and lower hardness value in the retreating side (273 Hv). With the availability of tungsten based refractory W-1%La_2O_3 plate size of 500 mm and 7 mm thickness of HSLA steel was welded. Therefore present study indicated that FSW of high softening metals and alloys can be welded with the W based tool materials, however, the tool life and higher thickness weld joint will be a key challenge for effective utilization.

Tool geometry effect on the thermal condition and material flow of FSW of 2219 aluminum alloys

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The tool geometry plays the most significant part in the material flow, stirring and thermal condition of friction stir welding (FSW). Appropriate combination of tool parameters such as shoulder surface type and probe profile together with welding variables like vertical load, weld traverse speed and tool rotational speed are required to obtain sound friction stir welds. The present investigation deals with the effect on tool shoulder geometry for friction stir welding on the weld temperature distribution and material flow. The temperature on the top surface of the weld joint was recorded using thermocouples. The geometrical features of the tools were incorporated in 3D computational fluid dynamics (CFD) model and weld parameters such as rotational speed and welding transverse speed were also utilized. Temperature dependent material properties were used in the CFD model. The numerically predicted temperature distribution and stirring pattern were compared with experimental values. The observed close proximity between numerically predicted and experimental values proves the suitability of the CFD model.

Influence of tool tilt angle on material flow behaviour in friction stir welding of AA2219

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Heat treatable aluminium alloy AA2219 is widely used for aerospace applications due to its high strength, good damage tolerance and weldability. Preferred welding process for AA2219 aluminium alloy are gas tungsten and gas metal arc welding processes due to their comparatively easier applicability and better economy. Though AA2219 alloy has better weldability compared to other grade of age hardenable aluminium alloys, it also suffers from poor welded joint strength due to melting and resolidification. Friction stir welding (FSW) is an emerging solid state welding process and hence free from any solidification related defects. Important process variables in FSW are tool rotational speed, feed rate and tool tilt angle. Most of the published work reported the influence of process parameter such as tool rotational speed or feed rate on material flow behaviour and its effect on mechanical properties of aluminium alloy welds. However tool tilt angle also plays a major role in material flow during FSW, controlling the size and location of defects. A study was undertaken to understand the role of tool tilt angle on temperature, forces and torque on friction stir welds of AA 2219. The variation in tool tilt angle resulted in variation of temperature and forging action on weld. An optimum value of tool tilt angle gives defect free welds, during which X-force and Z- force are balanced with viscosity and strain rate sufficient for material to flow and fill any voids or defects. A defect free weld, with 1 to 2 degrees tool tilt angle, depicts proper stirring of the material as well as forging action on plasticised metal of the welds.

Effect of rotation speed on microstructure and mechanical properties of friction stir welded carbon steel

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In the present investigation, friction stir welding was carried out on carbon steel under rotational speed of about 800-1400 rpm at fixed traversing speed of tool by keeping all other welding parameters constant. Study of microstructure, mechanical properties and determination of microhardness across the weld joint and evaluation of residual stress were performed. The peak temperature was in the range 1036-1087 ^{o}C, for a given tool rotational speed, indicating formation of single phase austenite at weld nugget (NW) during joining and the cooling rate was 4-7.5 ^{o}C/sec. The microstructure of the NW consisted of αferrite + bainite/ Widmanstatten ferrite of varying relative quantity depending on welding parameters. However, area fraction of bainite/ Widmanstatten ferrite in WN and pearlite in HAZ reduced with increasing rotational speed of tool. HAZ was influenced by thermal effect without any mechanical deformation. This region was also experienced phase transformation, contained predominantly ferrite + pearlite structure and exhibited increment in ferrite grain size with respect to base alloy after welding. Tensile strength of the joint initially increased, reached maximum and then decreased with continuous increase in tool rotational rate. Failure during tensile testing occurred predominantly from HAZ / HAZ-WN interface. Heat input rate, peak temperature, cooling rate and volumetric mass flow rate from advancing side to

retreating were calculated using empirical relations. Thermomechanical data of welding showed that increase in tool rotation rate increased the heat input and peak temperature at WN. Strain, strain rate and Zener-Hollomon parameter were also determined at WN by hot deformation dynamic recrystallization (DRX) equations. Empirical relations were obtained for DRX grain size and mean misorientation angle with Zener-Hollomon parameter at weld nugget by regression analysis. The volume of material within extrusion zone, width of extrusion zone and Zenner Hollomon parameter decreased while strain and strain rate increased with incremental rotation rate.

Session 3: Nov 13, 2014: [09.00 - 11.00]

Novel W-based metallic glass with high hardness and wear resistance

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Metallic glasses are an exotic class of materials that can show attractive properties like high hardness/strength and wear resistance. This work presents the development of the hardest and the most wear-resistant known metallic material – a new W-Ni-B metallic glass. Refractory metallic films of $W_{33}Ni_{32}B_{35}$ (at.%) have been deposited on stainless steel and Si substrates by dc magnetron sputtering. The alloy films are glassy, have a high crystallization temperature of 873 ^{o}C and show an extreme hardness of 24 GPa. Nanotribological testing reveals that this MG shows wear resistance approaching that of standard tribological materials like TiN as well as advanced nano-composite films based on Ti-Si-B-C. To the author's knowledge, this constitutes the first such demonstration for any metallic material. The alloy also shows a favourable combination of high hardness with low Young's modulus (hence high resilience), potentially opening up applications in certain micro-electromechanical (MEMS) devices.

Study of laser and electron beam welding of Ni-1Zr-0.1C and TZM alloy

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Nb-1Zr-0.1C and TZM (0.5 Ti-0.08 Zr-0.04 C) alloys are attractive structural materials for high temperature applications. The welding of these alloys is a challenging task due their high melting point and reactive nature. The high energy density welding processes like laser and electron beam (EB) welding capable of producing deep penetration welds are more suitable for welding of these alloys. The autogeneous laser (Nd:YAG) and EB welds in bead-on-plate (BOP) and butt joint configuration were produced on sheets of Nb-1Zr-0.1C and TZM alloy by systematically varying the process parameters. The weld zone, HAZ and base material of the Nb-1Zr-0.1C alloy and TZM weld joints were subjected to optical and electron microscopic examination. In the Nb-1Zr-0.1C alloy welds, the depth of penetration and the hardness values of the weld region were found increasing with the increase in the linear heat input in both the techniques. In comparison to the EB welds the laser weld zone and its HAZ showed significant increase in the hardness values due to oxygen pick-up during welding suggesting need for improvements in the shielding conditions. The finite element (FE) simulation software package SYSWELD was employed to predict the

thermal and stress field produced during the EB welding of 3.2 mm thick Nb-alloy plate joined in butt joint configuration. The weld region of the TZM alloy welds showed equiaxed grains without any cracks in weld zone whereas base metal shows elongated grain structure. The study of micro-hardness profile across the weld cross section showed decrease in the hardness values in the weld region as compared to the base material hardness.

Effect of beam oscillation on electron beam welding of AISI-316l joints

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Austenitic stainless steel (SS) AISI 316L shows superior mechanical properties at elevated temperature, oxidation resistance and better pitting corrosion resistance. It is widely used in exhaust manifolds, furnace parts, heat exchangers, jet engine parts, nuclear industries etc., where welding is often used as a fabrication tool. Conventional fusion welding is susceptible to hot cracking, and micro-fissuring in the fusion zone (FZ) and heat affected zone (HAZ). However, in electron beam welding process, because of its high energy density, precisely controllable heating position with a definite minuscule radius, low total heat input etc. many of the problems encountered in fusion welding can be controlled or eliminated. Even though earlier researchers have investigated welding of 316 SS by EBW, in none of such studies effect of beam oscillation has been investigated. The present work is focused on analyzing the mechanical and metallurgical properties 316L SS welds made with and without beam oscillation. The beam oscillation has an advantage of churning liquid action in the weld pool that restricts segregation of elements, lower residual stress, more uniform properties in the weld pool. This is in contrast to weld pool created without beam oscillation where directed liquid flow due to marangoni convection brings in directional properties. In such study circular beam oscillation pattern has been used and the radius and frequency of beam have been varied. The joints have been characterized through optical and scanning electron microscopy. The mechanical properties have been evaluated using tensile, impact, micro-hardness and bend test in accordance with the ASTM standards. It has been observed for certain parameters of beam oscillation the mechanical properties of the weldment can be enhanced significantly. The grain size and the amount of delta ferrite in the fusion zone are also found to be influenced by beam oscillation, which will be discussed in the presentation.

Electron beam welding studies on titanium alloys for aerospace applications

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In the present day scenario the use of titanium alloys has increased significantly due to its advantageous properties and characteristics. The alloys are classified based on the addition of alloying elements and the phase stability at room temperature. The components are being realized through welding route. In this paper we discuss electron beam welding of titanium alloys (alpha, alpha plus beta and beta alloys) which has advantages over other conventional welding techniques. A detailed characterisation was carried out

on EB welded plates of Ti5Al2.5Sn-ELI, Ti6Al4V-ELI, Ti5.4Al3Mo1V and Ti15V3Al3Sn3Cr alloys. The tensile properties of the weld joints in annealed conditions were evaluated with their microstructural characterization and a correlation has been established. The weld properties for all the alloys are observed to be at par with the parent metal.

Assessment of solder joint reliability of Sn-3.8Ag-0.7Cu alloys on copper substrates as a function of reflow time

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Reliability of solder joint depends on the intermetallic compounds (IMCs) that are formed at the interface of solder alloy and substrate during soldering. The wetting kinetics, microstructure and shear strength of Sn-3.8Ag-0.7Cu lead-free solder alloy on Cu substrate were investigated as a function of reflow time. There were four reflow times selected for the study viz., 10s, 100s, 500s, and 1000s. The reflow temperature was maintained at 270 °C. The wetting tests were carried out using a dynamic contact angle analyzer. Contact angle decreased with the increase in the reflow time. IMC thickness increased initially up to 100s and later decreased for higher reflow times in Sn-3.8Ag-0.7Cu/Cu system. The bond shear test was performed for the evaluation of solder joint integrity. The samples reflowed for 10s showed maximum shear strength.

Wettability and bond strength of Sn-3.5 Ag lead-free solder alloy reflowed on copper substrate

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The effect of solder reflow time on wettability, shear strength and interfacial microstructural characteristics of Sn-3.5 % Ag solder balls on copper substrates was investigated. Sn-3.5% Ag solder balls of about 0.08g weight were prepared using a soldering station. Cylindrical copper stubs (Φ12 mm x 8 mm) were mirror polished to obtain a surface roughness (R_a) of 0.044 µm. Solder balls were reflowed on copper substrates inside an environmental chamber for 10s, 100s, 500s and 1000s. The reflow temperature was set at 250 ˚C. The wettability of the solder alloy was determined using a dynamic contact angle analyzer. It was observed that the equilibrium contact angle was nearly the same for reflow times up to 500s. The contact angle decreased when the solder was reflowed for 1000s. Microstructural analysis of the soldered samples revealed the evolution of intermetallic compounds (IMCs). Scanning electron microscopy showed that the morphology of IMCs changed from continuous scallop shaped to long needles and finally, to coarse needles. Energy dispersive spectroscopy (EDS) analysis was performed to determine the constituent elements, their percentage and distribution at the solder/substrate interface. A layer of Cu_6Sn_5 intermetallic compound was observed at the interface at all reflow times. A decrease in the IMC layer thickness was observed for samples reflowed for 100s. With further increase in reflow time, the IMC layer showed an increasing trend. The samples that were reflowed for 100s showed the maximum average shear strength. Although the samples reflowed for 1000s samples showed good wettability, they exhibited lower shear strength.

Brazing of titanium to stainless steel

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Joining of titanium alloys to stainless steel is required in several applications in nuclear spent fuel re-processing units and satellite cooling systems. It has been a challenge to achieve high strengths due to formation of brittle intermetallics. Brazing is one of the most popular methods for joining dissimilar alloys. The dissimilar lap joint of titanium alloy to stainless steel plate was produced with a vacuum brazing furnace using two different Cu-base and Ag-base inter-layer filler alloys at different temperatures, soaking time and lap widths in order to investigate the effects of such brazing parameters on the joint strength and structure. X-ray diffraction analysis is used to investigate the presence of different inter-metallics formed in the interlayer region. The higher the brazing temperature is, the more intensive reactions occur between the filler metal and base metal and consequently thicker intermetallic layer may be formed at the interface. Both the interfacial microstructures and mechanical properties of brazed joints were investigated to evaluate the joint quality. It was found that the shear strength of brazed joints strongly depend on the thickness of the brittle intermetallic compounds.

Reciprocating sliding wear behavior of laser-clad layer of Ni-Mo-Cr-Si alloy deposited on stainless steel substrate by laser cladding

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In this study the dry sliding wear behavior of Ni base hardfacing alloy (Ni-Mo-Cr-Si) deposited on stainless steel SS316L substrate by laser cladding method has been carried out. The Ni-Mo-Cr-Si alloy powder was deposited on stainless steel 316L substrate using Nd: YAG laser (laser power: 800 W, powder feed rate: 8.3 gm/min, scanning speed: 400 mm/min, spot size: 1.8 mm). The microstructure of the clad layer was examined by optical and scanning electron microscopy. The phase analysis was performed by X-ray diffraction. Dry sliding wear behavior of the laser clad layer was evaluated against AISI 52100 chromium steel (~850VHN) in comparison to the substrate SS316L using ball on plate reciprocating wear tester at room temperature and without lubrication. The wear resistance was evaluated as a function of load and sliding speed for a constant sliding amplitude and sliding distance. The wear mechanisms of the wear track were studied using SEM-EDS. The appropriate selection of parameters resulted in defect free clad layer with little dilution. Various solidified zones were observed in the laser clad layer, i.e., planar crystallization near the substrate/clad interface, followed by cellular and dendritic morphology towards the surface of the clad layer. The clad layers were characterized by the presence of a hard molybdenum-rich hexagonal close-packed (hcp) intermetallic Laves phase dispersed in a relatively softer face-centered cubic (fcc) gamma solid solution or a fine lamellar eutectic phase mixture of an intermetallic Laves phase and gamma solid solution. Laser clad layer of Ni-Mo-Cr-Si on SS316L exhibited much higher hardness (~700 VHN) than that of substrate SS316L (~200 VHN). The laser clad layer exhibited higher wear resistance as compared to SS316L substrate while sliding against AISI 52100 chromium steel in all the test conditions.

Mechanical properties and microstructural characterization of XH67MBTЮ Ni-based superalloy welds

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In satellite launch vehicle, XH67MBTЮ Ni-based superalloy is widely used in the form of welded components. Both Gas Tungsten Arc Welding (GTAW) and Electron Beam Welding (EBW) processes are commonly used for manufacture of these parts. In this study, 3 mm thick sheets were welded by GTAW and EBW. The welds were characterised in as-welded and weld+PWHT conditions. Tensile properties, hardness survey across the weld, microstructural characterization and fractrographic analysis were done in GTA and EB welds in two different conditions. EB welds shows higher strength and ductility than GTA welds and PWHT significantly increased the strength with decrease in ductility.

Optimization of laser welding parameters to improve the quality of welds in hot rolled coils

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In high productivity cold rolling mills (CRM) hot rolled (HR) coils are laser welded to facilitate continuous processing of coils. The welding parameters such as laser power, pulse frequency and weld speed greatly affect the mechanical properties of the welds. Improper adjustment of the weld parameters commensurate with sheet thickness may lead to weld failures compelling to adopt open gap rolling. The process of open gap rolling is time consuming and affects the productivity of the mill. In order to reduce/eliminate the open gap rolling the weld quality of the HR coils need to be improved by optimizing the laser welding parameters. In the present study section wise frequency of open gap rolling were investigated and the result showed that the highest frequency of open gap rolling were in the lower thickness coils (2-2.8 mm) and the type of defects were identified to be incomplete fusion, cluster of porosity/voids and large difference in hardness between the weld zone and parent metal. Experimental trials were conducted by varying the laser power keeping the welding speed constant and then varying the welding speed keeping the laser power constant. The results are verified by the use of micro Vicker's hardness measurement, optical and scanning electron microscopy study. The incomplete fusion and cluster of porosities which were produced due to low heat input and faster cooling revealed by relatively higher micro hardness for the pearlitic lamella, were eliminated by increasing the heat input by increasing the laser power. The result showed that with the combination of 80% laser power and 45% welding speed the weld defects are completely eliminated and recommended for the operation in the plant. With the plant implementation the open gap rolling could be reduced from around 45% to below 10% resulted in significant improvement of productivity in mills.

Microstructure evolution in brazing joints of 0.07C – 16Cr – 6Ni steel and silver obtained with a Ag-Cu-Sn filler metal

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Joining of stainless steel parts to pure silver is encountered in various parts of the cryogenic and semi-cryogenic engines for space applications. In this study, the brazing process for joining of an Austenitic-Martensitic (A-M) class stainless steel with pure silver is explored. The low melting Ag-Cu-Sn based alloy was used as brazing filler metal. The effect of brazing conditions on the base material is evaluated with simulated brazing cycles. The microstructure evolution across the brazed joint will also be discussed in terms of formation of diffusion assisted zone (DAZ), intermetallic formation and heat affected zone (HAZ).

Improving properties of meta-stable beta titanium (Ti15V3Al3Sn3Cr) alloy electron beam weld joints

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Titanium alloys play a major role in launch vehicle programme due to its high specific strength and good corrosion resistance. In the present paper we discuss regarding the electron beam welding (EBW) studies of beta titanium alloy (Ti15V3Al3Sn3Cr). The beta alloys are having low weld efficiency when compared to alpha and alpha plus beta alloys. Ti15V3Al3Sn3Cr alloy is a metastable beta titanium alloy contains single phase beta at room temperature upon annealing. An attempt was made to increase the weld efficiency for this alloy by varying various parameters during heat treatment. The EBW on 7 mm thick plate was evaluated for tensile property and microstructural characterisation on specimens heat treated with various soaking time (20 to 60 minutes), welded and further aged (482 ^{0}C/16hrs/AC or 538 ^{0}C/8hrs/AC). Increasing the solution treatment time period prior to welding has improved the ductility of the aged specimens with no compromise in the strength. Also, the location of failure was shifted from the weld to parent metal location. This may be due to lower volume fraction of alpha precipitates resulting from reduced nucleation sites with increasing soaking time in the weldment.

Characterization of laser welded T-91 steel weldments

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The microstructural evolution of laser welded modified 9Cr–1Mo (T-91) steel weldments are analyzed and discussed in this paper. Due to the short heat impulse and the large heat input in laser, the microstructural change in the heat affected zone is very small. End cap butt welding of T-91 carried out by continuous Nd:YAG laser. A series of T-91 tubes and end plug were welded and investigated in a two-fold approach: (1) process optimization: the laser processing parameters were optimized to obtain welds with minimum defects, and (2) material characterization: weld microstructures were evaluated. The microstructure and

the phases present in the weld zone were characterized using optical and scanning electron microscopy, X-ray diffraction, and also the micro hardness measurement. A particular attention was made to study the correlation between surface structure and mechanical behavior. The heat energy that has been delivered during welding operation was calculated using the relationship $[\frac{P}{Vt}]$ where P is the wattage, V is the linear velocity or the welding speed and t is the wall thickness of the fuel tube. This expression represents the total heat input per unit area inside the sample irrespective of the watt and the exposure time of laser radiation.

MATERIALS FOR AEROSPACE SECTOR

Invited talks

Session 1: Nov 14, 2014: [08.30 - 10.30]

Engineering Challenges in Aerospace Industries: Indigenization of Technologies and Success Stories

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Aerospace is an unexplored domain for Indian scientist, technologist, engineers and other interested partners. There is a lot of learning and catch up from developed countries who have explored fully the three verticals namely Defence, Space and Aviation. We are about 10 -15 years behind them on all fronts including manufacturing. However, through ISRO and DRDO efforts are being taken to understand various requirements and convert them into user friendly equipment. Godrej Aerospace is one of the participating company in this above domain along with ISRO and DRDO and over three decades we have tried to learn light alloys like aluminum and titanium which play an important role considering their high specific strength. These alloys have peculiar characteristics and give very little freedom for engineers to work. Our attempt in this presentation would be to compare and highlight difference between light alloys and commercially dominated alloys with examples and the process we have mastered. Titanium alloys especially Ti6Al-4V is very widely used in aerospace owing to its high specific strength. We at Godrej Aerospace have developed vital expertise in machining, welding, surface treatment, heat treatment, forming and brazing of these alloys for control surface of missiles and booster assembly. The talk will also elaborate technical details of the work carried out by us for one of the Indo-Russian Missile.

Session 2: Nov 15, 2014: [11.00 - 13.15]

Titanium alloys for aircraft structural applications

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High strength, low density, and excellent corrosion resistance are the main attributes that make titanium alloys attractive for a variety of applications including aerospace. Titanium alloys are used both in aircraft structures (high strength in combination with low density) as well in aero-engines (high strength, low density, and good creep resistance up to about 550°C). The aircraft structure uses all the three classes of conventional titanium alloys. The hydraulic tubing in the aircraft uses the “half alloy”, while Ti-6Al-4V fits in wherever an of the shelf solution for titanium alloy usage is required. Ti-10V-2Fe-3Al is still used in various aircraft structures besides landing gears. Ti-5Al-5Mo-5V-3Cr, a variation of the Russian alloy, VT 22 is under active consideration for structures with higher section size thickness which is beyond Ti-10V-2Fe-3Al. Beta 21 S, being a beta titanium alloy is cold rollable but also has good oxidation and creep resistance upto medium temperature range and is used for various applications. In the aeroengine, several titanium

alloys are used. Leading the way is Ti-6Al-4V in the medium temperature capability upto about 350°C. Then there are conventional high temperature near titanium alloys, like Ti-6Al-2Sn-4Zr-2Mo and Ti-5.8Al-4Sn-3.5Zr-0.7Nb-0.5Mo (IMI 834). Moreover, now there is renewed interest in -titanium aluminides with its usage in the GE Genx commercial engine which has led to a substantial weight saving. Most of the titanium alloys are amenable to normal thermomechanical proceeding routes like forging and rolling, except the aluminides. The industrial complication arises because for processing titanium alloys a better temperature and strain rate control is required most of the times and microstructure and micro-texture plays a greater role in some of the mechanical properties. The usage of these alloys will be discussed and their developmental issues will be flagged.

Contributory papers

Session 1: Nov 14, 2014: [08.30 - 10.30]

Mechanical properties and microstructural characterization of ultralight Mg-4Li alloy for aerospace applications

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Microstructure and mechanical properties of binary Mg-4Li alloy were investigated upon hot rolling at 300 °C to a maximum reduction of 60 %. Hot rolling resulted in development of appreciable fractions of twins in the microstructure suggesting deformation twinning could be an important strain accommodating mechanism during rolling. Along with the twins, fine recrystallized grains were also observed in the microstructure and grain size obtained upon rolling was 17 μm. Annealing at 350 °C for 60 min subsequent to rolling resulted in twin-free polygonal shaped grain microstructure with a grain size of 46 μm. Tensile tests at room temperature at 10^{-4} s^{-1} resulted in improved strength of 155 MPa and ductility of 9.7 % in as-rolled condition. These values were changed to 122 MPa and 17.3 %, respectively, upon subsequent annealing. Thus, the ultralight Mg-4Li alloy showed a good combination of strength and ductility at ambient temperature with a specific strength of 107 kN·m/kg and 84 kN·m/kg in as-rolled and rolled+annealed conditions, respectively, which suggests the possibility of its potential use in aerospace and automobile applications.

Development of ultrafine grained Al 6063 alloys through SPD processing

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The size of the samples that can be produced through severe plastic deformation (SPD) process limits the use of Ultrafine grained materials in practical applications. Multi directional forging (MDF) is one of potential techniques to produce UFG structures in the bulk materials. Hybriding of various SPD techniques could be exploited to produce UFG material in required shape with desired grain size. In the present investigation, cryoforging was combined with cryorolling to develop UFG aluminum alloy sheets in bulk. Al 6063 alloy with initial grain size 200 µm was subjected to solid solution treatment followed by water quenching at room temperature. Before subjecting to MDF, ageing was performed at 100 °C for 4 hours and 8 hours to enhance the dislocation accumulation. This process was then followed by cryorolling up to 2.7 true strain to produce long sheets. The final product was subjected to low temperature ageing at 100 °C to ensure the beneficial effect of precipitation hardening. Microstructure and mechanical properties were evaluated through Vickers hardness testing, tensile testing and Electron back scattered diffraction technique (EBSD). The results indicate that cryoforging followed by cryorolling enhanced the microstructural homogeneity in the material. Remarkable improvement in strength (UTS- 300 MPa) and ductility (11%) was observed after ageing. The average grain size was observed to be 400 nm with higher fraction of high angle grain boundaries, which are suitable for forming applications.

Composites and their contributions in the fields of aerospace

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A Composite material is a heterogeneous material created by the synthetic assembly of two or more materials at macroscopic level,to obtain specific characteristics and properties. The individual components are separate and distinct within the finished structure. It consists of two phases; one is termed as the reinforcement phase in the form of fibers, sheets and particles; which is embedded in the phase called matrix phase. The composite have many advantages over the other metallic materials (high strength to weight ratio, highly corrosion resistant, exceptional formability and durability etc). This paper highlights the contribution of composites in the field aerospace. In aerospace sectors CFRP (Carbon Fiber Reinforcement Plastic) and sandwich structures (eg: GLARE: Glass Laminate Aluminium Reinforced Epoxy) play an important role. A study of the composite aircraft is highlighted. Some examples of composite's success in this field is also mentioned (namely BOEING787, AIRBUS A380). The Future innovations in the field of composite in these sectors to combat the challenges have been focused. The future innovations in the aerospace include the central composites and use of Carbon-nanotubes.

Optimization of aging cycle of stainless maraging steel using dilatometric and differential scanning calorimetric analysis to improve its strength

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04Х13Н5М5К9Л is a high strength stainless maraging steel coupled with excellent toughness. This alloy has been selected for making Impeller casting which is subjected to localized stresses reaching as high as 700 MPa at the tip of its vanes. The impeller rotates at 19000 RPM for 253 seconds. This alloy comprises of austenitic – martensitic dual phase at room temperature, in which martensite exhibits Body Centered Cubic (BCC) structure with moderate hardness (HRC 30) and high toughness. The required strength is achieved by precipitation of second phase particles in soft martensite matrix at suitable aging temperature. There is always a chance of reversion of martensite to austenite on heating either for extended period or at higher temperature. Thus optimization of aging temperature and time becomes highly critical keeping in mind the functional stress requirements of the component.The optimization has been done using Differential Scanning Calorimetric (DSC) analysis, Dilatometric studies and experimental iterations of heat treatment temperatures and aging time. Different combination of strength and ductility could be achieved by varying heat treatment parameters. Martensite start and finish temperatures and Austenitic reversion temperatures were established through Differential Scanning Calorimetric and Dilatometric studies. Effect of subzero temperature on properties and microstructure of this steel is also presented here. Scanning Electron Microscopy and Energy Dispersive Spectroscopy were also carried out to analyze phases in different heat treatment conditions.

Ablation resistance of polymer derived Si-Hf-C-N-O ceramics

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The ablation resistance of ceramic materials is one of the fundamental properties which need to be validated for materials to be used in aerospace applications. Ceramic foams, because of its low thermal conductivity and light weight make it an excellent thermally insulating material. But ceramic foams, when exposed to elevated temperatures are subjected to an oxidation process, which reduces the strength. Over the past two decades, immense studies have been carried out on polymer derived Si-C-N systems due to their ability to withstand temperatures in the order of 1500°C. Since hafnium based materials has a high melting point (~2000°C) and high chemical stability, high temperature oxidation can be influenced by the incorporation of hafnium to the polymer. Si-Hf-C-N-O foams were produced by thermolysis of hafnium modified polysilazane at 1000 °C for 1 h. Prior to thermolysis, the samples were cross-linked at 300 °C for 2 hours. Varying volume fraction of hafnium tetra n-butoxide to polysilazane believes to play a key role in determining the ablation resistance of the foam. High dense, Si-Hf-C-N-O was also produced by spark plasma sintering at 1400 °C in vacuum at a pressure of 30 MPa. In this study, the ablation mechanism of Si-Hf-C-N-O foams was investigated. Three different volume fractions (5, 15 and 25%) of Si-Hf-C-N-O foams were synthesized and the samples were subjected to oxy-acetylene flame at three different temperatures

(1400°C, 1500°C and 1600°C) for duration of 60 s. The mass loss of the samples was measured after the flame exposure. The oxidation products were determined using XRD and the microstructure and elemental analyses were carried out using Scanning Electron Microscopy and EDS respectively. The ablation mechanism of high dense Si–Hf-C–N-O (5% volume fraction) processed by spark plasma sintering was also studied and compared with the corresponding foam samples.

Indigenization of Al & Si – bronze for LCA and other aircrafts bearing cages

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Indigenous development of materials is of interest to defence to induct these materials for defence application. This work activities is concerned with the indigenous development of Al-Bronze 4640F (UNS 63000) and Si-Bronze 4616 (UNS C65620) aerospace grade copper base materials for defence applications. Presently both these materials are being imported for fabrication of anti-frication bearing cages required in LCA (Tejas), Kaveri and other aircraft application. These Al-Bronze and Si – Bronze have been used typically for parts requiring strength, good corrosion resistance, wear resistance at moderate temperatures. This materials usage is not limited to such applications. The above material is required for manufacturing the anti friction bearing cages of LCA and other aircrafts. Indigenization of these anti friction bearing cages with non ferrous materials requires extensive applied research on structure property correlations to achieve desired properties. Thus the objective of the proposed activity is indigenization and airworthiness certification of Al-Bronze and Si-Bronze alloys, enabling industrial scale production of aerospace quality Al – Bronze and Si – Bronze materials. The above material produced at industry level should meet the Development Test Schedule (DTS) / Aeronautical Material Specification (AMS) and qualify for airworthiness applications for fabrication of anti friction bearing cages for aircraft applications. To attain the required technical requirements the processing has been devised and optimized. Extrude tube of size OD 63mm, ID 32mm and 2 meters long has been successfully produced. This development tube is tested and characterized as per Development Test Schedule (DTS) and met the requirement as per aeronautical material specification (AMS) and airworthiness applications.

Session 2: Nov 15, 2014: [11.00 - 13.00]

Process design and control to achieve higher strength and toughness simultaneously for Ni-Cr-Mo steel (AISI 4340)

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Ni-Cr-Mo steel (AISI 4340) is very popular and widely used for many high strength application where a good combination of strength and impact properties are essential in fairly large component. This alloy steel is used in most industry sector for a variety of applications including high strength part, collets, spindle, gear and pinion shaft. Analyzed AISI 4340 steel, which is used in rig as a pinion shaft, requires high strength as well as high toughness at sub -zero degree Celsius. We analyzed this material with the help of

simulation software and found 100% cracking tendency with water. With oil and polymer it shows less cracking tendency, and standard also recommends the same. So initially we did polymer quenching but found lower strength and toughness. We did study & experiment of split quenching and showed improved result of both strength and toughness at sub -zero degree Celsius. Subsequently we modified the quenching with water, which also gives better results. With water quenching we achieved better property and productivity.

Optimization of strength and facture toughness of high strength, metastable beta titanium alloy, Ti-10V-2Fe-3Al

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Ti-10V-2Fe-3Alis a metastable high strength beta titanium alloy mainly used in structural components of aircrafts. The alloy was triple melted with iron powder as one of its constituents which resulted in higher levels of oxygen with respect to specified value. The objective of this work was to produce aerospace quality alloy at industrial scale and to optimize heat treatment cycle to achieve specified strength with fracture toughness greater than 44 MPa $m^{1/2}$ in forged 100mm. dia. bar in both longitudinal and transverse directions. The two extreme conditions of lowest and highest temperature of solution treatment (755°C & 780°C) and aging (480°C & 520°C) were compared in terms of their effect on tensile strength and fracture toughness(K_{IC}). While both yielded higher than specified strength, the K_{IC} in 755°C(ST) +480°C(Aging) (755+480)was lower(25-38 MPa√m). It improved substantially in the 780°C(ST) +520°C(Aging)(780+520) to above 60 MPa √m for both longitudinal and transverse direction. SEM and TEM were used to study the effect of Heat treatment on evolution of microstructure and fractography. While there was no significant dependence of these on orientation, the Heat treatment produced small change in the microstructure and perceptible change in the fracture surface. The improvement can be attributed mainly to low volume fraction of the primary alpha and coarser secondary alpha which are observe under transmission electron microscope for780+520 as compared with 755+480. The optical macro side profile studies of failed fracture toughness samples of 780+520 & 755+480 treatment shows deeper dimples for 780+520 which is mainly attributed to higher micro plasticity as compared to much shallower dimples of 755+480 treated specimen. The electron back scatter diffraction studies were conducted on differently orientated specimens of the alloy to understand the effect of micro-texture on fracture toughness.

Effect of grain boundary alpha on mechanical properties of Ti5.4Al3Mo1V alloy

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The paper brings out a correlation between microstructure and mechanical properties of Ti5.4Al3Mo1V (VT14 alloy) high strength alpha beta titanium alloy, an alloy widely used in aerospace programme. In general titanium alloys are processed below beta transus temperature for attaining best combination of

strength and ductility for aerospace applications. In the present study, we carried out heat treatment trials of the alloy at various temperatures: above beta transus, 980 ^{0}C and 1000 ^{0}C soaked for 45 minutes followed by different cooling rates and observed the effect of grain boundary alpha on the mechanical properties of the alloy. Observed prior beta grain boundary with discontinuous alpha phase for 980^{0}C treated samples whereas, continuous grain boundary alpha was present in the 1000 ^{0}C treated samples. Mechanical properties were evaluated for 980^{0}C beta annealed specimens with all three different cooling rates. The strength was observed to be maximum in water quenched condition (1291 - 1308MPa) and lowest in furnace cooled condition (895 - 905MPa), whereas the impact strength is highest in furnace cooled (8.03 - 8.7 Kg-m/cm^2), comparable in air cooled (7.3-8.5Kg-m/cm^2) and lowest in water quenched condition (2.5-3.1Kg-m/cm^2). Comparison of properties and microstructure is included in this paper.

Effect of orientation on recovery, recrystallization and α - β phase transformation of Ti-5553 alloy for aircraft structural applications

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Recovery, recrystallization, and α to β phase transformation behaviour for Ti-5553 alloy were investigated using dilatometric experiment and orientation image microscopy. Continuous heating of deformed samples at constant heating rates were carried out in dilatometer set up, during which different phenomena like recovery, recrystallization and α to β phase transformation occur. Expansion or contraction observed in the dilatometry curves (Relative change in length vs. Temperature) were caused by combined effect of expansion due to α to β phase transformation and contraction due to recovery and recrystallization. Dilatometer samples of different orientations were prepared keeping its longitudinal direction parallel to rolling direction (RD), normal direction (ND), and directions at angles of 30° and 60° from RD direction in the RD – ND plane. With change in sample orientation dilatometry behaviour has been found to change significantly. When sample's longitudinal direction was parallel to RD, then no contraction was observed during heating; instead a continuous expansion was seen in the temperature range of 250°C - 810°C. However, for the sample whose longitudinal direction was parallel to ND, significant contraction was observed in the temperature range of 610-915 °C in addition to expansion behaviour observed due to phase transformation. In the present paper these interesting observations would be explained in terms of texture present in the initial deformed material and its effect on phase transformation and recovery and recrystallization behaviour would be illustrated.

Microstructure evolution, phase transformation and oxidation behavior of third generation γ TiAl alloys

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Titanium aluminides have attracted considerable interest for high temperature application in aerospace and gas turbine engine due to its low density, and good high temperature strength. As a result of these properties, it has become a candidate material to replace certain Ni-based super alloy parts in gas turbine engines. In the present study, four different compositions of third generation gamma titanium aluminides have been selected for the microstructure, phase transformation and oxidation behavior studies as these have to be studied in detail to understand the full potential of these alloys. These four alloys have been

studied and compared with two standard compositions (a) Ti-48Al-2Cr-2Nb (at %) (GE) and (b) Ti-45Al-5Nb-0.2B-0.2C (TNB-V5, GKSS). These alloys which solidify through the β phase field in the beginning possess relatively fine and homogenous microstructures ($\gamma + \alpha_2$ lamellae) without strong tendency for segregation. This is unlike peritectic solidifying alloys of previous generations which have coarse grained microstructure and significant chemical inhomogeneity. Phase transformation temperatures were determined using differential scanning calorimetry (DSC). Microstructural evolution was studied after heating in the two phase $\alpha + \gamma$ and single phase α region and cooling at different rates. Equiaxed γ and $\gamma + \alpha_2$ lamellar structure was observed after heat treating in different regions and cooling at different rates. Retained β phase was observed in most of the conditions due to presence of β stabilizing elements e.g. Nb and Cr. Oxidation resistance of the alloys was studied at 900^0 C up to 200 hours and was found to improve with the increase in the Nb content.

Effect of solution treatment and aging on microstructure and tensile properties of high strength β titanium alloy, Ti-5Al-5V-5Mo-3Cr

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Ti-5Al-5V-5Mo-3Cr (Ti-5553) is a recently developed high strength β titanium alloy to be used for larger size components in aircraft structures replacing Ti-10V-2Fe-3Al. In the present study, a double VAR melted ingot was thermo-mechanically processed and its microstructure and tensile properties were determined as a function of β as well as α+β solution treatment (ST) and aging. Solution treatment in β region was carried out at 885˚C and in α+β region at 825˚C and 800˚C, the β transus temperature of the alloy being 850°C. α+β solution treatment resulted in a finer grain size in comparison to β solution treatment. Aging was carried out at four temperatures of 450˚C, 500˚C, 600˚C and 650˚C as a function of time for all solution treatment conditions. Coarsening of secondary α precipitates occurred with increase in time at a given aging temperature. The β solution treatment and aging resulted in higher strength and α+β solution treatment and aging resulted in an optimum combination of strength and ductility. Microstructures as well as fractographs of tensile tested specimens were examined as a function of aging time and temperature. Fractography revealed that the β ST & aged specimens show a mixed mode type of tensile failure, whereas α+β ST and aged specimens show a more dimpled, ductile fracture surface.

Application of non–destructive techniques on composite materials used in aerospace industries

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Composites have unusual combination of properties other than any of engineering materials. It provides almost unobstructed range for development of superior mechanical, physical, thermal properties as well as chemical properties. To get all this properties in composite materials, bonding between matrix and reinforced phases plays very important role. In this research work, studies were carried out on composite materials, such as CFRP and GFRP, to evaluate the quality of the bonding between phases by various non-

destructive testing techniques, likes ultrasonic testing, radiographic testing, thermographic testing etc., in order to detect different defects like disbond, delamination, porosity, inclusion, cracks, voids, etc. Ultrasonic and thermography techniques are used to detect delamination type defects in composite materials. Radiographic technique is used to detect volumetric flaws such as porosity, inclusion etc.

MATERIALS FOR AUTOMOTIVE INDUSTRIES

Invited talks

Session 1: Nov 13, 2014: [13:30 - 15:30]

Light is Just Right: Reducing Carbon foot print in product development

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New product development in automotive throws a bigger challenge on working with material genomics to comply with ever increasing requirements of lighter, stronger with better reliability and higher formability along with one step solution to achieve optimal shape. Multi material model with material synthesis in sync with product development and that too with an option of mass production with minimum carbon foot print and that too within a narrow tolerance band are some of the bigger challenges which need to be answered by material scientist to maintain their relevance. Finally with steeper product development cycle with limited option of proto and tight budgeting, robust computation techniques are being marveled to have real time virtualization of almost all parameters in order to productionise the optimal product with greater flexibility using innovative solutions which are the key driver for survival.

Session 2: Nov 14, 2014: [08:30 - 10:30]

Experiences of Tata Steel in the Development of High Strength Steel for Automotive Industry

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Since the 1990's regulations regarding CO2 emissions and fuel efficiency have become increasingly stringent in response to the global warming issue. The automotive industry has made strenuous efforts to reduce the weight of car bodies and improve the efficiency of engines / driving systems. Automotive companies worldwide are having three major objectives, namely Improvement in safety, Reduction in greenhouse gas (CHG) emission and reduction in cost. Steel suppliers are closely working with automotive suppliers in achieving the above objectives. Passive safety can be improved by increasing the usage of high strength steel so that the crash resistance is improved. Besides this, use of high strength steels is a major enabler in the light weighting of cars for the improvement in the fuel efficiency and reduction in CO2 emission. Thus the development and commercialization of advanced high strength steels is on priority for both the automakers as well as steel suppliers. The Indian automotive industry till recently, in absence of crash regulatory norms and non availability of high strength steels, was using steels having tensile strength upto 440 MPa. By this, auto companies could derive only a limited success with regard to safety and reduction of CO2 emissions. With the commissioning of Thin Slab Caster and stronger hot rolling mill in 2012, Tata Steel started the Journey of the development of advanced high strength steels for the automotive market. To start with, Tata Steel embarked on the development of the family of Hot rolled high strength steels with tensile strength of 590 MPa. This family has four varieties and the selection of a

particular one depends on the property attributes needed for the end product application. The present paper deals with the effects of micro alloy design, thermo mechanical rolling and ROT lamellar cooling on the micro structure, mechanical properties and material performance of the high strength hot rolled steels, having Tensile Strength of 590 MPa.

Status, challenges and opportunities in developing environmental friendly aluminum alloys for automotive application

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As the price of fuel increases continuously, it will be appropriate to reduce the consumption pattern of the same in automotive sector. This is only possible by reducing the weight of the vehicle using low density and environment friendly materials. Among the materials that are available in the market, aluminium is considered as one of the possible candidate materials. While using aluminium in this sector, both fuel consumption and greenhouse gas emission can substantially be reduced. Accordingly, attempts are made for the maximum utilization of aluminum alloys for making several components i.e. from engine parts to suspension items as well as car body panels that are required for automotive industries.70% aluminum used on today's vehicles are sourced from recycled scraps that are utilized for making the components for automobile sector.In case of recycling of aluminum scrap, it requires only 5% energy to produce the automobile componets as compared to the same produced from virgin metal. In the process, 95% energy can be saved.Aluminum consumption in automotive applications has maintained steady growth in the past 30 years and it is expected to grow further for more energy-efficient vehicles. Recycling post-consumer aluminum to build new vehicles will further reduce the manufacturing cost and emissions that lead to significantly lower the production costs. In India, the growth of automobile sector is ever-increasing. As a result, dual benefits are obtained. This is a win – win situation for the industry and our planet. In order to achieve the dual benefits i.e. protecting the environment and making the car more fuel efficient, it is desirable to use aluminum alloys extensively in automobile industry. The success of these alloys is not only based on weight reduction but also based on the maintenance cost for users and great design flexibility for manufacturers. Furthermore, as compared to other materials, aluminum alloys also offer five distinct advantages such as, safety, fuel economy, machinability, environment friendliness and driving performance in automobile sector.Presently, in India around 50 Kg aluminum is used for making different components and sheet metal parts in automotive sector, whereas the consumption pattern for the same in USA, Western Europe and South Asian Countries is more than four times. Therefore, a tremendous scope exists for automobile manufacturers in India to use aluminum in this sector. Initially, aluminum alloys were utilized in non-structural application in automotive, but presently, these alloys are also used in structural purposes. Using sheet metal forming technique, several complicated panels and load bearing components are also being formed from aluminum alloys particularly from 5XXX and 6XXX series alloys. Typical parts that are warm formed by this process are body panels, floor sheets and door frames. The major challenges for the usage of aluminum in automobile sector are the availability of alloys, manufacturing techniques and the right kind of manpower.Unfortunately, India lacks all three areas. To overcome these problems, immediate steps are required, i.e. using both public and private partnership for developing all three areas.

Session 3: Nov 15, 2014: [11:00 - 13:00]

Advances in Manufacturing of Engineering Materials

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The demand for high performance engineering materials in sectors e.g. automotive, aerospace, defence etc engine is significantly increased in recent years and it will continue in the future as well. The technological advancement in materials and design is on going to enhance the mechanical properties of the components. Manufacturing industry has taken the challenges to meet the demands. This paper covers the critical aspects of current manufacturing and future trends of engineering components required for mainly automotive and aerospace industries. The tubular components for aerospace are made of non-corrugated rigid pipes made from austenitic stainless steel, nickel, aluminium and titanium alloys. The side impact beam used in the car for crash safety is made of ultra clean steel with appropriate processing technology to achieve high impact at -40C. The manufactured pipe components must be free from contamination, which causes restricted fluid flow, blockages and excessive wear. The welding and brazing process are carried out with precision control. The bending of alloy pipes is also carried out after careful heat treatment. The special tools for bending of pipes are used to avoid galling of the surface but maintain adequate support during the manufacturing. The Non Destructive/Destructive Testing (NDT) e.g. pressure testing, X-rays, ultrasonic, 3 Point Bend, Impact testing etc. are used to check the integrity of components. The demand is shifting from austenitic stainless steel to Ni-Co-Cr-Mo-Ti based super alloys for high temperature corrosion and creep resistance properties in aerospace. Material properties are achieved by correct parameters of manipulation. The design of chemistry plays a vital role in performance of engineering components. High level of Robotics is used for close dimensional tolerance. Many components during service are subjected to high temperature and hence in future protective coatings like ceramic coating has to be developed on the tubular components.

Aluminium alloys: Current trends inautomotive applications

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Corporate Average Fuel Economy (CAFE) regulation in US has driven auto manufacturers to push technologies and advanced materials development aggressively to enhance fuel economy. The first part of the talk will relate to the current status of alternate automotive materials usage, specifically aluminium alloys in passenger vehicles. Following the introduction, a short overview of the challenges and opportunities involved in adopting aluminium alloy as a material of choice in relation to other automotive materials will be presented. Last, we will look at the some of the challenges and opportunities in aluminium alloys for outer body panels. Existing infrastructure in automotive plants and designs have been built with the familiarity of certain materials and properties. This provides unique opportunities to develop manufacturing processes that are tuned for Aluminium alloys within the constraints of existing manufacturing setup. To illustrate, couple of manufacturing processes that were modified to suit Aluminium sheet alloys, hemming and preform annealing processes, will be discussed.

Session 4: Nov 15, 2014: [14:00 - 16:00]

Advances in localization prediction in wrought aluminum for automotive closures

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Closures constitute a large fraction of the unsprung mass of a vehicle. Weight reduction in closures results is a direct improvement on all the five design drivers viz: Safety, NVH, Vehicle Dynamics (ride and handling), Durability and Environment (& fuel economy). Aluminum alloys are a potential material system for such weight reductions in automotive closures. With the advancements in continuous casting technologies, the cost disparity of aluminum alloys with steel have also reduced. However, the manufacturability (specifically formability) of aluminum alloys for A-class surfaces and large plastic deformation is still a challenge. In this reference a deformation modeling technique has been developed to predict the localization of aluminum alloy sheets under complex strain states. The model takes advantage of in-plane crystallographic anisotropy to predict the most susceptible localization orientation. This can then be used to design the closure stamping process to minimize localization. Experimental validation of the model with LDH experiments have been found to show an excellent correspondence.

Contributory papers

Session 1: Nov 13, 2014: [13:30 - 15:30]

Microstructural characterization of cold worked and hot worked twip steels by X-ray diffraction

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Twinning Induced Plasticity (TWIP) steel is a typical representative of the 2nd generation advanced high strength steels (AHSS) which exhibits a combination of high strength and excellent ductility due to the deformation twinning mechanisms. In the present work, two TWIP steel compositions Fe-23Mn-1.2Al-0.3C and Fe-18Mn-1.2Al-0.6C were analyzed for the Microstructural Evolution and its mechanical properties during both cold and hot deformations behavior. The samples were hot rolled and cold rolled with different strains till fracture. X ray line profile analysis has been used to investigate crystallite size, micro strain, twin and stacking fault probability using software package MAUD (Materials Analysis Using Diffraction) based on Rietveld Refinement and dislocation density using CMWP (Convolutional Multiple Whole Profile). The Micro strain has a general trend of increasing with the deformation whereas the crystallite size follows a general trend of being more or less same at various strains. High dislocation density of the order of 10^{15} is observed and shows an increasing trend till the maximum strain and a sharp drop at the fracture strain.

Analysis of machinability for bainitic steel 18MnCr5-3

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The Bainite structure is known for its high tensile strength and good ductility; it does not require heat treatment process after rolling or forging. This would eventually attract the Bainitic steel to become the better alternative for automotive forgings. The present work is a comprehensive study of experimental work to study the machinability of Bainitic steel at higher cutting speeds. The experiments were planned using Taguchi design of experiment approach and using L9 orthogonal array. The machinability is assessed in terms of, cutting forces, cutting temperature, chip mechanism and flank wear of the tool. Analysis of output responses; cutting forces and cutting temperature was carried out to determine the optimized cutting parameters using Taguchi linear model anaysis. In the chip mechanism study, shear angle is analyzed to establish the relationship between machinability and process conditions.

Study of recrystallization behavior and kinetics of aluminium nitride precipitation in a cold rolled low carbon extra deep drawing steel

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The deep drawability is the premier requirement of auto-body sheets. So all efforts are made to get this precious property after cold working. In the present work the recrystallization behavior and the kinetics of precipitation in deep drawing grade Al-killed low carbon steel sheets has been studied. The extra deep drawing steel was heavily cold worked to 72% of thickness reductionand the cold rolled specimens were annealed at various temperatures by isothermal holding. The hardness and microstructure studies were carried out both in the cold rolled and annealed conditions. The scanning electron microscopy (SEM) and optical microscopy were used for microstructure studies and characterization. The structural analysis was done by X-ray diffraction technique. The fraction recrystallized after different annealing time has been determined by measuring micro-hardness. Linear intercept method was employed to measure the grain size. Recrystallization behavior was modeled using Johnson-Mehl-Avrami-Kolmogorov equation.The hardness variations showed a decrease at the beginning of annealing followed by an increase trend. The increase was noted in thetemperature range 350-450°C due to precipitation effect, whereas in between 500-600°C hardness value decrease rapidly due to high recrystallization kinetics. Onset of recrystallization was noted in EDD grade after isothermal annealing at 500°C. The precipitation kinetics of AlN was indirectly studied by electrical resistivity measurements. Investigation of the results shows that the precipitation process occurs during annealing greatly affects the cold worked structure and restoration process.

Yield improvement of interstitial free steels used for skin/outer body panels

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Last five years have witnessed a significant growth in the Indian automotive market. Tata Steel aspires to become a preferred supplier in the chosen automotive Flat Products segment. This led to the development of interstitial free (IF) for skin (outer) and internal panels of cars/SUVs. Customer requirements in terms of surface quality of IF grade, particularly skin panel surface quality, are extremely demanding unlike other steel grades. This calls for necessary and sufficiency checks with respect to equipment capability and process norms throughout the IF production chain.pchhhTata Steel started production of cold rolled batch annealed IF steels for internal panel way back in 2002. The main surface defects were slivers, blisters, scale and scum. To reduce these defects, new process norms and routes were established along with addition or modification of equipment in the IF production chain. One of the casters was converted into vertical mould type and practice of m/c scarfing of slabs were started. These actions improved overall quality and yield of IF internal panel. The production of skin (outer) panel was started by the end of 2006. The surface quality norms of skin panel are even stringent which led to very low overall yield of skin panel, 22-26% from steelmaking to cold rolling till 2010.Concerted efforts were taken since 2011 to improve skin panel yield by further reducing the major causes of rejection e.g. tiny scale, special type of slivers, tong marks etc. Tiny scale was reduced to zero by new operating regime at hot and cold rolling mill. Slivers have been reduced to a very low level by modification of casting and scarfing norms. Introduction of Flow Control (FC) mould in caster and HSS rolls in Hot Rolling Mill have improved the quality further in terms of lowering slivers and scale defects. There was huge yield loss taking place at Cold Mill owing to handling of annealed coils through mechanical tongs. This has been addressed through introduction of Magnetic Tong at Batch Annealing Furnaces. As a result of these actions, the present overall yield of skin panel stands at 41%, which also has contributed towards meeting ever increasing market share and customer commitment in FY14.

Micromechanics modelling of deformation of AHSS steels

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With the increasing usage of AHSS grade steels in modern cars for vehicle light weighting and passenger safety, it has become necessary for automotive industries to deeply study and accurately predict the response of these steels to enhance their designs for better performance and light weighting. Due to their complex multiphase nature, conventional continuum based approaches of modelling the materials are not suitable to capture the physical phenomena occurring at microscopic level. With the increase of the computing power of modern day computers, Representative Volume Element (RVE) based micromechanics approaches of modelling deformation of such materials, via real or synthetically generated microstructure with appropriate boundary conditions, have become one of the promising ways of tackling this problem. The present work focusses on modelling the deformation of Dual Phase Steels using 2D and 3D microstructure based micromechanics approach in order to study the deformation pattern of the microstructures and also to extract the macroscopic flow behavior of the steel under

uniaxial tensile loading using first order homogenization technique. Chemical composition based dislocation theory model has been used to model flow curve of the individual phases. In the present framework, a number of sensitivity studies were conducted in order to characterize the prediction of present micromechanics approach and its comparison with experimental results. The studies conducted are: (i) effect of boundary conditions (ii) effect of type of analysis (iii) effect of mesh type (iv) effect of martensite distribution and (v) effect of dimensionality of RVE. OOF2, NEPER and DREAM 3D were used to create microstructure meshes and ABAQUS was used for carrying out finite element analysis. Based on the various studies conducted it can be concluded that the micromechanics based methodology is a promising approach which can be used to predict the deformation and flow behavior of the of advanced multiphase steels and thus help in designing better microstructures.

Evaluation of steel for inclusions in steel using murakami method and co-relation to fatigue properties

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Inclusions play a major role in influencing the fatigue behaviour of steel components such as bearings, shafts, pins, gears etc. While there are various techniques of inclusion evaluation, Murakami's method of extreme value statistics is used for ultra clean steels, where the oxygen content is less than 10 ppm. The critical inclusion sizes at core and subsurface influences the fatigue failure initiation. In the present study, few ultra clean steels made in the plant has been used for the estimation of inclusion, using extreme value statistics. The typical type, morphology and shape of inclusions using SEM has been examined. The correlation of the inclusion to the fatigue properties would be examined.

Mechanical and corrosion behavior of SMATed interstitial free steel for automobile industry

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Interstitial Free (IF) steel are integral part of automobile industry that are ideal for deep drawn parts due to its high formability and non - aging behavior. In addition to excellent formability of IF steel there is a need for improving its mechanical properties, in particular the fatigue resistance. There are several processes of surface modification like shot peening, surface mechanical attrition treatment (SMAT) and laser shock peening etc. used for enhancing mechanical properties. Among these SMAT is found to be quite effective in improving high cycle fatigue resistance of structural components.The present investigation was undertaken to study the effect of SMAT on microstructure, bulk hardness, microhardness profile, residual stresses, tensile properties and corrosion behavior of the IF steel. Studies were also performed following stress relieving treatment to SMATed IF steel samples.The process of SMAT was applied on sheet samples of 4 mm thickness using USSP machine (SONATS STRESSVOYAGER®, France) for 100 s, 200 s and 300 s with 3 mm diameter 100C6 steel balls. Surface modification was studied through optical microscopy & scanning electron microscopy. Variation in microhardness profile was found upto ≈ 800 µm which was quite consistant with variation of residual stresses in depth from top of treated surface. Also their was significant increase of 125 times in the yield strength compared to as received material with

enhanced corrosion resistance of treated samples at different conditions. The modification in properties resulting from SMAT was attributed to surface nanocrytallization (SNC) and the compressive stresses induced in surface region from impingement of balls at ultrasonic frequency.

Effect of 8-13wt%Mn on the microstructural characterization of Fe-Mn-C steel

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Mn based steels have shown promise for automotive application but however very limited research was conducted in medium Mn range that are aimed at enhancing the mechanical properties through microstructural control. Till now it has been reported that both high and low Mn steel are undesirable for automotive application. Thus two different steel composition characterizedas Fe-0.62C-13.64Mn-4.24Al-2.29Si, (S-1) and Fe-0.54C-8.36Mn-5.24Al-1.72Si, (S-2) produced by melting-casting route, have further been mechanically worked and heat-treated to obtain the desired microstructure. Microstructural analysis reveals austenite as the major phase along with martensite after quenching and tempering heat treatment. The steel containing higher Mn with low stacking fault energy results in hexagonally closed packed ϵ-martensite formation where as the steel containing lower Mn with relatively higher stacking fault energy results in body centered tetragonal α-martensite formation.

Study of active and passive safety systems and design of mirror impact test rig

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Active and Passive Safety Systems are very important as per the Automotive Industry Standards (AIS). One of the major active automobile safety device is a rear view mirror and the testing of rear view mirrors is a significant part as per AIS. The existing rear view mirror impact test rig available in Automotive Research Association of India (ARAI) needs to be modified, to carry out the testing of large size mirrors used in heavy vehicles. The tests are performed on Automotive Inner Rear View Mirrors (IRVM) and Outer Rear View Mirrors (ORVM), to certify them according to AIS. The proposed work is to prepare a detailed monograph of active and passive safety systems and also to redesign the rear view mirror impact test rig as per the Automotive Industry Standards (AIS). The developed rear-view mirror impact test rig can test large size rear view mirror of maximum size of 1.5 x 0.5 metres and the accuracy of test rig is increased by four degrees.

Study on microstructural characteristics in 21-4N engine valve steel material

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21-4N engine valve steels are demanded for exhaust valves of engines where service temperature have been on the constant rise. The steel is usually supplied in the solution annealed and aged condition. The desired microstructure is having nitrogen pearlite in the matrix for engine erosion resistance against the exhaust gases of combustion. The steel is a high carbon high nitrogen austenitic stainless steel and it is of interest to examine the microstructure development and mechanical properties at various conditions of aging to understand the steel behavior. In the present study, samples were cut and solution treated at 1050°C/1 hour. These samples were aged at three different temperatures [500°, 600° 760°C] for varying time durations. The C-curves were examined. Selected samples were tested for high temperature tensile behavior. The wear and erosion resistance of an automotive engine valve is enhanced by the cellular Cr_2N precipitation, although toughness and ductility of the steel deteriorates.

Session 2: Nov 14, 2014: [08.30 - 10.30]

Effect of steel chemistry and process parameters on galvannealed coating morphology

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In automobile sector, galvannealed strips offer a better alternative over normal galvanized coils as they possess better weldability and paintability properties in comparison to pure zinc coating. Galvanneal alloy coating is formed when in a typical Hot dip galvanizing process, post zinc bath treatment, steel strip is subjected to an annealing treatment. Annealing induces diffusion between zinc and steel strip thereby transforming the pure zinc layer Eta Phase (η) into a complex alloyed Fe-Zn intermetallic coating. The intermetallic layer consists of Gamma (Γ)-1 and Gamma (Γ)-2 layers which are adjacent to steel strip and richest in iron phase. Adjacent to Gamma layers are Delta (δ) and Zeta (ζ) layers respectively. The drawing and powdering properties of galvannealed coatings are mainly dependent upon morphology of these intermetallic layers. The alloying elements like Ti, Si, Mn, C, P etc are known to affect the galvanneal alloying reaction. The present paper investigates Galvannealing morphologies over different steel substrates and investigates the underlying process-chemistry relationship for the differences observed. Conclusively, an insight is also provided to achieve desirable galvanneal properties.

Development of HSFQ steel for automotive sector at rourkela steel plant

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In India cold reducer segment is growing very fast and their quality requirements for Hot Rolled coils are more and more demanding. It has been found that customers have preference for high strength and better forming quality steel grades (popularly known as HSFQ steel) with varying range of chemistry for specific end applications. Due to its unique features such as increased strength, toughness, formability and weldability properties, these HSFQ steel is required for variety of critical applications such as long and cross members of auto chassis, pre engineered buildings (PEB) structure etc. In line with the development trend and market requirement, special quality hot rolled HSFQ grades with YS 350/450 MPa minimum, used mainly for application in auto components after cold forming were selected for development at Rourkela Steel Plant (RSP) SAIL.These Grades have been developed by optimizing processing parameters through BOF-VAR-LF-CC route. Critical issues for development of these Grades were selection of suitable alloy chemistry, clean steel making and optimized hot rolling practices followed by extensive trials both at plant and customer premises. The newly developed HSFQ hot rolled steel possess an attractive combination of strength and formability properties which are better substitute of SAILMA 350/450& BSK-46 due to its economical as well as competitive edge.The formability of the steel is adjudged by high hole expansion ratio(145 % minimum), low YS/UTS ratio (0.80-0.85) and higher elongation (25% minimum). By judicious micro alloying finer grain size (7-8 micron equivalent to ASTM no 11-12) has been achieved which in turn enhances the strength and toughness of the material and also formability. This has been possible as a result of innovative alloy design and synergistic effect of Nb and Si during controlled processing with the formation of Nb(CN) during hot rolling.

Drop forging of gear blank under different cooling conditions and its mechanical properties

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Gear is one of the most widely used and mass produced components in automobile industry. Producing gear of reduced weight and increased strength results in energy savings hence there exists a strong demand for it. Gears are the most stress prone parts of a vehicle and demands high wear resistance, high contact fatigue strength. An ideal gear should have uniform and optimum metallurgical quality, excellent heat distortion control, maximum impact strength, stiff wear resistance, optimal transmission efficiency, less noise, vibration free operation.The conventional close die forging process is characterized by better mechanical properties, high production rates and good surface quality in mass production. In this paper study has been made for manufacturing of gear blank by forging process under different cooling condition such as sand, air and oil. Mechanical properties have been studies under these different conditions. Also Wear properties of forged gear blank has been studied against En 31 steel as counter body using Pin on Disc method. It has been observed that mechanical properties are influenced by forging parameters employed and different cooling rate used. Depending on cooling rates different microstructures are obtained which are responsible for different mechanical and tribological properties.

Challenges of rolling special grade steels in hot strip mill

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Rolling special grades require precise control on microstructural evolution during each thermo mechanical event happening during rolling process. Offline microstructural modelling has been done using hot strip mill Model (HSMM) interface. Special grades rolling do not just require the shape and finishing/coiling temperature control. It requires the suitable pass design along with temperature control, which is sometimes challenging to achieve in hot strip mill. This paper summarises the typical challenges to roll the below special grades in Hot Strip Mill. Thinner gauge high tensile grades: the main limitation was observed with the rolling load/torque on the finishing stands. Thicker Gauge Line Pipe Grades (API X70/80) need low temperature finish rolling and low coiling temperature, difficult to roll and coil. Dual phase steel need precise control on cooling to control the phase transformation and relieve the internal stresses that can lead to shape variation. It requires two stages or three stages cooling on ROT, difficult to achieve with available CT control automation system. Temperatures drop on the edges of strip leading to variation in microstructure and mechanical properties along the edges, leading to shape defects.Although the paper do not describe, the technological advanced solutions available in market, to meet the technical requirement. This paper shows the ways and means to overcome the limitations by alloy design concepts and available resources.

Decarburization in spring steel

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Decarburization means reduction of carbon content at the surface layers of the steel. It occurs when the metal is heated to temperatures of 700°C or above. In full decarburization the upper layer of the steel is composed primarily of ferrite materials, while in partial Decarburization, a mixture of materials may be present. It is well known that decarburization has a bad effect on strength, fatigue strength and resilience energy. This research presents few methods to improve the product quality by means of recovering the decarburization layer. Unlike conventional methods, which usually use mechanical means, this method relies on a basic metallurgical principle, the process of diffusion and thermodynamic reaction. A carbon-rich layer is coated on the surface of the object. The object is then heat treated at conditions similar to the manufacturing process. The experiment began by coating a carbon-rich material onto decarburized raw material. The samples then reaches to austenitic temperature and subsequently either annealed or quenched. The state of the carbon restoration was then evaluated. We can also minimize the decarburization extent by using cylinder of plastic gas (i.e.hydrocabon) in pre-heating furnace at the time of mill breakdown. Another method is also the use of natural gas or coke oven gas in pre-heating furnace to make oxygen present in blast furnace gas flammable. This idea is under evaluation at Usha Martin Ltd (UASD) Jamshedpur.The decarburization process on the surface of the coil is inevitable and is accepted upto 1% of diameter of the coil but at Usha Martin Ltd is occurring beyond 1.5% of diameter. After the successful implementation of this idea, it was observed that it has reduced the Decarburization process by 0.5% and hence improved the yield.

Recrystallisation behaviour of low carbon niobium bearing microalloyed steel

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As steel is deformed, the internal energy increases through the storage of dislocations. So the dislocation density increases continuously. When the internal energy reaches a threshold value, the deformed grains may be replaced by new strain-free grains via the recrystallization mechanism .So Recrystallisation is the formation of new strain free grain on the place of strained grain. In Niobium Microalloyed steel two type of the recrystallisation occur namely Primary recrystallisation and secondary recrystallisation. Niobium Microalloyed steel when deformed at 81% then recrystallisation start at 800ºC.When the recrystallisation start decrease in the hardness take place but due to the formation of the Niobium Carbide precipitate rate of decrease of the hardness is not very high. But when the sample steel is heated above 1150°C all the Niobium carbide precipitate get dissolved due to which abruptly increase in the grain growth take place which result in the sharp decrease in the hardness.

Dross: A necessary evil in galvanising process

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Dross defect is as old as galvanising itself. Dross defect is more common than ever in continuous galvanising process. Why does dross occur so often? Why it is so difficult to eliminate it? What can we do about it? This paper attempts to answer some of these questions. It also reviews technique for defect identification and root cause analysis and discusses countermeasures. Examples and case histories are used to characterise and cure defect.

Development of high toughness corrosion resistant rail

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There are many criteria, which determine the suitability of steel for rail track applications. The primary requirement is structural integrity, which can be compromised by a variety of fatigue mechanisms, by a lack of resistance to brittle failure, by localised plasticity and by excessive wear. Historically, manufacture of rails has been carried out in eutectoid composition to achieve full pearlitic microstructure throughout the matrix leading to improved mechanical properties. Alloying elements in various proportions are added to eutectoid steels to improve the mechanical and corrosion resistant properties. In the present work, an attempt has been made to develop high toughness corrosion resistant rails having superior ductility, fracture toughness and corrosion resistance properties without compromising on other mechanical properties. It has been statistically established by Indian Railways that 72 UTS rails (C-0.40-0.60 wt%) are

less prone to sudden failure as compared to 90 UTS rails (C-0.60-0.80 wt%). However, reduction in carbon level is associated with reduction in strength. 72 UTS rails are associated with good fracture toughness and elongation properties. The present work is an endeavour to develop rail steel with strength and fracture toughness properties superior to 90 UTS rails. Eight laboratory heats with carbon ranging from 0.40 to 0.55 wt% were made with addition of various alloying elements like Cr, V, Nb, Ni, Cu in different combinations to achieve desired properties. Alloying elements were selected on the merit of their strengthening and corrosion resistance properties. Results have shown improvement in tensile, hardness, fracture toughness and corrosion resistance properties. After successful trial results at laboratory scale three plant scale heats were produced. There was significant improvement in YS, UTS, %El, fracture toughness, fatigue strength, wear resistance and corrosion resistance properties.

Review on challenges in grain refinement of interstitial-free steel

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Interstitial- free (IF) steel is widely known as affordable high-quality steel for deep-drawing applications, especially in automotive bodies. Grain refinement is a powerful tool in designing microstructures with improved properties. Two kinds of methods to refine ferrite grains are a) through severe plastic deformation b) through thermomechanical processing. The challenges this research is facing are discussed. High density of grain boundaries play significant role in the development and exhibition of novel properties. Grain boundaries are characterized by excess grain boundary energy, presence of long range elastic stresses and enhanced free volumes. They possess increased free energy density, increased width, high density of dislocations (full or partial), large residual microstrains. Quantification of structural parameters including the boundary spacing, boundary misorientation angle and distribution, and dislocation density is important in order to understand the strengthening mechanisms and to establish the correlation between the structural and mechanical properties.

Session 3: Nov 15, 2014: [11:00 - 13:00]

Advance steel solution to face the challenges of automotive industry

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Automobile industry is facing challenges of ever-increasing energy crisis and environmental problems. In addition, upgraded safety norms have forced this segment to increase the proportion of high strength steels. However, limited formability and large spring back experienced with high strength steel restricts its uses to reduce the weight of vehicle. Besides the component fabricating tools requires high rigidity and strength while using these steels.To overcome these impediments, a new concept is introduced where austenitized steel blanks are formed and die-quenched simultaneously. To be best suited for this concept, Steel Authority of India (SAIL) has introduced MnB steel, which will satisfy the requirement of lightweight high strength (>1500 MPa) complex crashworthy auto component fabrication. To extend the framework for our recently developed advanced steel grades for automotive sector, a major joint program has been

conducted between The Automotive Research Association of India (ARAI) and SAIL. Present article discusses the outcome of the study with respect to advanced mechanical properties and microstructural features of the advance steel.

Niobium micro alloyed steel solutions for automotive BIW lightweighting

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Presently, there is great emphasis on greenhouse gas reductions and improving fuel efficiency in the transportation sector, such that all car manufacturers, assemblers, component producers and material suppliers are investing significantly in lightweight materials R&D and commercialization. While globally attainment of this objective is with the rider of uncompromising passenger safety, in India, car makers for the domestic market are in the initial phase of benchmarking both fuel efficiency and passenger safety against their global peers.The body-in-white (BIW) i.e. the body structure, including all hang-on parts such as fenders, doors and lids, weighing between 300 to 500 kg depending on vehicle size, constitutes the largest share of the total vehicle weight. Most of the components used for BIW, suspension, chassis or frame are produced from flat steel products such as hot or cold rolled strips. The BIW of modern passenger cars contains up to 80% high strength steel of which the majority is covered by traditional (ferritic, ferritic-pearlitic and/or -bainitic) HSLA steels. While multiphase steels offering a good compromise of high strength and adequate elongation, have been gaining a significant share since the late 1990s, more recently, press-hardening steels with a typical strength of above 1200 MPa have been introduced for applications requiring an extra resistance against crash impact.Niobium can be considered as an essential microalloying element in almost all high strength automotive flat steels. Besides its effect on microstructural refinement, niobium is also used for scavenging carbon (applicable to IF and BH steels), precipitation hardening and transformation control in most of the single and/or multiphase AHSS. In the present article, these beneficial effects of Nb-microalloying will be highlighted charting their journey into first, second and third generation AHSS in light of the new product development opportunities for the Indian steel manufacturers.

Optimization of process parameters for excellent surface quality and formability for skin panel automotive applications

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Ultra low carbon cold rolled steel sheets processed through batch annealing route often encounter with temper colour problem. The surface defects like temper colour is dependent on the processing method. The present paper will describe development of Ultra low carbon IF batch annealed having excellent surface quality with better formability ($\bar{r}$-value) of minimum 2.0, yield strength of less than 150 MPa. To achieve the desired quality different operating parameters are optimized like hot coiling temperature, cold rolling reduction, electrolytic cleaning Speed, hot and cold spot temperature in batch annealing furnace. Stacking the coils in batch annealing furnace to avoid sticker mark. The foremost point is optimizing

annealing temperature and time to eliminate tempercolour. This steel has been successfully developed at JSW Steel Ltd and under supply and approval from various auto makers.

Development of hot forming grades: a solution to light weight vehicles

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Technological and designing advances in the automobile industry has led to a constant need for development of high strength steel with improved quality, which can drastically make vehicle lighter without compromising safety. Hot forming grades are gaining impetus and momentum to replace existing high strength steels, especially in automotive and emerging agro machinery sectors. These hot forming grades provide great advantage over other high strength steel because of its leaner chemistry and amenable hardenability. Hot forming process utilizes steel's ability to achieve high hardenability and hence provide high strength, along with good toughness and formability. In this paper development of Boron based hot forming grades is presented for automotive applications as well as agricultural machinery. The microstructure of these steel after heat treatment is a typical quench and tempered Martensite. Complex automotive parts can be manufactured by this process along with very low spring back and high part strength. The key challenge lies in correct Boron addition and its distribution as well as achieving uniform microstructure. Industrial problem encountered is segregation of Boron during casting and control of Nitrogen and other alloying elements. This poster deals with the problems encountered in industrial production of highly formable hot forming hardenable Boron grade steel for this specific application. Trials for this product is successful for making automotive part like door inner as well as agro equipment's like Harrow Disc and is found to meet all the requirements for end application.

Development of bulk ultrafine interstitial free steel by equal-channel angular pressing followed by flash annealing

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Interstitial Free (IF) Steel is extensively used in automobile sector owing to its commendable ductility and formability, but fuel efficiency demands continuous improvisation in strength with decrement in weight. Despondently, any attempt to strengthen IF Steel by bringing down the grain size to ultra-fine level, compromises its ductility, rendering it useless. Here, we propose a novel two step strategy for achieving manifold increase in strength of IF steel, without much compromise in ductility, by tailoring a bi-modal grain size distribution in the material, such that, sub-micron sized grains enhances strength, while micron sized grains sustain ductility. Rolled IF steel billets were deformed by Equal-Channel Angular Pressing (ECAP), using a die that introduces equivalent strain of 0.6 at every passage of the billet, adopting Route B_c for 5 passes. Microstructural and mechanical investigations of these samples attest significant increase in dislocation densities, resulting in increased strength, but decreased ductility. Samples are flash annealed for 5 min at five different temperatures; 650^0C, 675^oC, 700^0C, 725^0C and 750^0C and subsequently water quenched. Flash annealing at elevated temperature for short duration facilitates secondary

recrystallization, allowing a fraction of favoured grains for a controlled growth. Optical microscopy, Electron back scattered diffraction (EBSD) and Transmission Electron Microscopy (TEM) confirm the bimodal structure, with both sub-micron and micron sized grains, in different proportion at specific conditions. These samples were evaluated for mechanical properties. Detailed results will be discussed in the presentation.

Development of micro-alloyed steel for crankshaft application

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During the last decade, the auto components manufacturing sector in India has grown significantly. Such auto-component include variety of engine, transmission, suspension and drive line components such as gears, crank shafts, connecting rods , axles, spindles etc. Conventional crank shafts are either cast or forged from medium carbon or low alloy steels reheated to form austenite and then quenched and tempered to achieve the desired strength. In this process route, besides the rising cost of alloying elements ,the machining and heat treatment costs are also high(energy ,distortion /Rework, longer lead time are issues for this type of processing).In order to overcome these problems , Micro Alloyed Steels (MAS) , where small quantity of V or Nb is added to help precipitation strengthening were developed. With their favorable cost, high strength to weight ratio, good workability with out the use of costly alloying elements these steels have found acceptance in the forging industry. The MAS are able to achieve the required strength levels through precipitation hardening and grain refinement in the hot forged condition thus avoiding the need for heat treatment. Recent developmental efforts are focused on Micro-alloyed steel bars for certain critical forging applications. The quality requirements for such applications demand.Close control on chemistry within restricted range of overall spec.Freedom from nonferrous impurities.High degree of cleanliness.Favorable mechanical propertiesFreedom from internal and surface defectsNo grain coarsening at working temperature of 1200°CControl on rolling temperatureUniform cooling post rolling .This paper outlines the steel-making/rolling facilities at MUKAND LTD and the process capability to meet the mechanical/metallurgical parameters of micro-alloyed steels forgings for crankshaft application. Industrial experience and data on chemical, mechanical and metallurgical parameters of medium carbon micro alloyed steels are presented and discussed.

Develop new kind of bake hardening steels by precipitation of copper for automobile applications

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The requirement for the high strength steels for automotive industry is crucial. Apart from strength there is also a the thickness of the steel used also plays an important role, as the thickness increases the weight increases resulting in the increase in the fuel consumption as well as excess metal required. If the thickness of the steel sheet that is used in the automotive bodies is not sufficient enough, it poses a problem for denting or it reduces dent resistance of the automobile bodies. Requirement to develop a new kind of bake hardenable steels by adding 0.5 % Cu, and necessity to precipitate the copper in between 150^{0} C-180^{0} C, as the copper in the Fe-C system will precipitate as the pure elemental form. This contributes to the strengthening of bake hardenable steels. Also determined to develop the precipitation

at as low temperatures as possible so as to ease the process during the production which contributes to the fuel consumption in return.This kind of steel will also increase the dent resistance, so the thickness of the steels that are being used in the automobile bodies can be reduced. This will greatly affect the fuel consumption as well as the production cost of these steels will be less as the paint baking can do the ageing treatment and there will be no requirement for the special ageing treatment to increase the strength as the ageing temperatures as same as paint baking temperature.

Development of dual phase hot rolled steel for automotive applications

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Hot rolled dual phase steels (DP590 grade with minimum UTS-590 MPa) are suitable for multiple automotive applications, where high strength is required with formability and weldability. This paper will describe the development of microalloyed DP590 grade through hot rolling. This grade has shown superior elongation (minimum 24% at gauge length of 50mm) and low YS/UTS ratio (less than 0.65) along with minimum required UTS level of 590 MPa. The final microstructure of this steel typically contains approx 25% of hard bainite/martensite coupled with ferrite (ASTM grain size No.-12.5) .The critical consideration during rolling of this steel includes sufficient strain accumulation before phase transformation to allow faster strain induced phase transformation from austenite to ferrite. Small microalloy addition of Cr or Mo is done to improve the hardenability and resist any pearlite formation during run out table cooling. The two step cooling pattern is utilised to allow austenite to ferrite transformation in first step of cooling, than the second step of fast cooling transform remaining austenite in to the hard phase. This steel has been successfully developed at JSW Steel Ltd and under supply and approval from various auto makers.

Session 4: Nov 15, 2014: [14:00 - 16:00]

Aluminium foams: A potential material option for automotive sector

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Among metal foams, closed cell aluminium foams are the most commonly produced that provide unique combination of properties such as: ultra low density, high energy absorption under static and dynamic conditions, blast amelioration, sound absorption and flame resistance. In DMRL, a technology based on liquid metallurgy processing has been established to produce structurally sound aluminium foams of uniform density varying from 0.3 g/cm^{3}to 0.8 g/cm^3. Capability exists to produce foam bodies of different shapes, foam filled inserts and other conventional sizes up to 220 mm diameter or 175 mm^2 square sections, and 800 mm long foams. Structural uniformity and control over cell size distributions (hence density) were obtained by both experimentation and process modelling efforts.The effect of foam characteristics i.e. cell structure and density on foam properties is investigated. Extensive evaluations includeuni-axial compression tests of different densities & strain rates, dynamic impact/indentation tests, drop hammer tests on foam filled & empty tubes,shear tests on foam sandwich panels, sound energy

absorption studies and heat resistance tests. In all the tests, the response of aluminium foams is found to be superior compared to existing materials/solutions.It is recognized that closed cell aluminium foams provide new materials options to designers of aerospace, transport and other defence systems. Various applications such as bumper, crash box, and centre pillar have been reported in the automotive sector. Our efforts at DMRL in developing Al foam technology at semi-commercial scale, along with extensive characterisation and testing will be discussed with specific reference to their potential for automotive applications.

Interdiffusion studies in Ti-Nb Binary Alloys

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Titanium alloys have a wide range of applications in aerospace, automobile, medical implant & surgical instrument. Ti-Alloys are most promising lightweight materials in automobile industry because of high strength to weight ratio. Ti-alloys can offers strengths that are almost comparable to steels but have half the weight of steels. This provides a unique combination of reduced fuel consumption and improved performance in automobiles.Binary interdiffusion data as a function of composition in Ti-Nb system are essential in modelling and controlling the kinetics of many metallurgical processes like phase transformation and phase growth, in multicomponent Ti and Nb alloys. Interdiffusion in Ti-Nb system was studied by assembling single phase diffusion couples between pure Ti & pure Nb at three different temperatures 1100, 1125 & 1150ºC. All the diffusion couple were characterized by a much steeper concentration gradients in Nb-side than in Ti-side. This indicates that the diffusion is very sluggish in niobium matrix compared to that in titanium matrix. It was observed in Ti-Nb diffusion couples annealed at 1100, 1125 & 1150ºC for 48, 24 and 24 hours respectively that the interdiffusion coefficients decrease and activation energy increases with increasing niobium content.

Foaming behaviour of Al-TiB_2 composite and ALPORAS (Al-1.5 wt.% Ca) foams

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Metallic Foams are a class of cellular materials. These are of great technological interest due to their novel physical and mechanical properties. Aluminium and its alloy composites can be foamed by different processing routes. Melt route processing of foams has more advantages than other processing routes because of lower cost of raw materials involved in the melt route. In the melt route technique, melts which are thickened either by in-situ formation or externally added ceramic particles can be foamed by creating gas inside it.In the present work, Alumnium-TiB_2 and ALPORAS (Aluminium-1.5 wt. % Ca) foams were produced by melt route technique in which TiH_2 was used as blowing agent. The parameters which influenced the foaming behaviour are temperature, stirring time and holding time. The role of these parameters will be presented. In the case of Al-TiB_2 composite foams addition of magnesium shows significant improvement in foam stability Stabilisation mechanisms of these foams will be discussed and the properties of the foams will be presented in terms of their cell size distribution, cell wall thickness and microstructural features.

Deformation behavior of friction stir processed magnesium alloy

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Strategic usage of magnesium alloys in automobile and aviation sectors was absent due to their poor formability. However, ductility of Mg alloys can be improved by fine grained microstructure. Friction stir processing (FSP) has its own virtues compared to the other severe plastic deformation techniques to achieve fine grained microstructures. In the present study, Mg-8Sn-1Al-1Zn (TAZ811) magnesium alloy was cast into plate shape. Fine grained and defect free microstructures are produced by optimizing the processing conditions. Multi-track FSP experiments were carried out with a 50% overlap in each pass. Phase evolution studies and interface between multiple tracks was characterized using XRD and optical microscopy. Tensile and compressive deformation behavior of the FSPed material was studied over a wide range of temperatures using the samples extracted from the multi tack FSPed regions.

Semi-solid processing of in-situ Al-15Mg_2Si-Si composite for automotive application

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Aluminum (Al) based particulate metal matrix composites have drawn attention of material scientists and design engineers because of their light weight, low density, excellent castability, good wear resistance and low cost of production.Though there are several methods exists in practice for metal matrix composite (MMC) development, melting/casting route is one of the cost effective techniques to develop Al based MMCs. But there are wettability issues between matrix phase and reinforcing particulates in case of ex-situ composites. Present trend of composite development is directed towards in-situ nucleation and growth of reinforcing phase within the liquid melt to produce a thermodynamically stable system.The present study reports semi-solid processing route of Al-Mg_2Si composite development to obtain spheroidal morphology of primary Al and in-situ formed Mg_2Si phases. Hypereutectic composition of Al-Mg_2Si system (Al-15Mg_2Si) has been chosen here to impart sufficient strength within the developed composite by increasing percentage of reinforcing hard Mg_2Si particles, where semi-solid processing facilitates enhanced ductility within the developed composite due to globularisation of microstructural features. The normal as-cast microstructure of Al-15Mg_2Si-Si consists of dark phases of primary Mg_2Si, bright phases of Al and eutectics of Al, Mg_2Si and Si. Semisolid processing of the composite melt following cooling slope rheocasting technique transforms irregular polygonal and hopper like morphology of primary Mg_2Si into near spherical one. Simultaneous effect of heterogeneous nucleation and crystal fragmentation mechanisms are found to be responsible for generation of spheroidal microstructural features during cooling slope slurry generation process. After pouring of the superheated melt into the cooling slope, solid nucleation and successive detachment of the same under application of shear driven flow of the melt along the slope yields semi-solid composite slurry upto the slope exit with approximate solid fraction of

0.45. Further enhancement in degree of globularity/sphericity has been evidenced in case of both the primary phases i.e, primary Al and primary Mg_2Si after isothermal holding of the cooling slope processed slurry. Significant improvement in mechanical properties has been evidenced in case of cooling slope rheocast Al-15Mg_2Si-Si composite compared to its conventional liquidus cast state due to reduced solidification shrinkage and air entrapment during mould filling.

Effect of deformation on precipitation behavior of aluminium alloys

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Poor ductility in ultrafine grained materials remains an important issue for its practical applications. . It has been reported that precipitation hardenable alloys are suitable candidates to develop UFG structure with excellent strength and ductility as compared to its coarse grain counterpart. In order to achieve this, Severe plastic deformation (SPD) after soild solution treatment followed by low temperature ageing has been used to enhance the strength and ductility of UFG Al alloys in the present work. After SPD, microstructure contains high dislocation densities, which influence the quenched in vacancies by the way precipitation behavior. Deformation after solution treatment had a significant effect on the precipitation sequence. In the present investigation, low temperature differential scanning calorimeter (LTDSC) was used to study the effect of deformation on precipitation evolution of Al 6061 alloy. Before performing LTDSC, alloy was subjected to solid solution treatment followed by water quenching. Cryorolling was used to induce deformation in the material without any precipitation. At lower deformation (5%), clustering at low temperatures got suppressed and with increasing deformation beyond 5%, two distinct clusters are formed and their heat effect is remarkable. Even 5% deformation is sufficient to suppress or accelerate the β prime peak in the alloy. Beyond 15%, with increasing deformation, the precipitation behavior was observed to be similar. Cluster formation has completed after two days of natural ageing in both coarse grained solution treated and water quenched and cryorolled 96% deformed material. It was observed that with 5% deformation, activation energy is reduced for β", whereas with increasing deformation beyond 5%, activation energy is increased and maintained constant at all deformations.

Study on composite helical spring for automotive applications

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Helical spring is one of the essential parts of an automotive system that absorbs high shock and stabilizes the vehicle, during motion. Most common helical springsare made by steel, different types of alloys and composite materials. Researchers are interested in studying composite springs, due to the high strength to weight ratio they, offer for automotive applications. The aim of the present work is to present a comparative study of different types of composite materials that are used to make helical springs for automotive systems. The comparisons have been carried in purview of the deflection and strength capabilities. The finite element analysis results have been obtained using ANSYS, and show the stresses and deflections of the helical spring; maintaining same load while varying the composite materials. The

results show that more stress has been generated on the carbon fiber composite. The glass fiber composite has been more deflected. The base pattern has been modified with the influence of both carbon and glass fiber composite for the study and establishing the suitability of this type of helical spring automotive applications.

Composite-automotive body structure

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In today's world fuel-effecient and light weight vehicles is a great need. These two characteristics are closely interrelated. Reduction in vehicle weight corresponds to decrease in fuel consumption and increase in the performance. Hence weight has a direct and inverse relation with fuel consumption and performance. This paper describes the design, fabrication, and assembly approach used for the composite body structure.The present paper discusses about the Hypercar, Inc.'sRevolution concept vehicle. A comparision between car of 90's and present structure is also done .The Revolution's body structure is 57% lighter than a conventional steel body structure of the same size, providing superior crash protection, improved stiffness, and favourable thermal and acoustic properties. The integral part of the structure is an advanced-composite passenger safety cell. This paper describes the design and production method of the composite body, explains how the body is integrated with the rest of the vehicle, and analyzes the benefit of lightweighing on overall fuel-cell vehicle cost. The composite safety cell design is also discussed along with part consolidation, material selection, part design, structural analysis and part fabrication methods.. the composite car body has been highlighted with some composite success in past has been mentioned.Finally future advancement in the composite car structure has been discussed. Hence composites can prove to be a nice and effective alternative to steel bodies.

A study on the composites and their contributions in the field of automotive

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A Composite material is a heterogeneous material created by the synthetic assembly of two or more materials at macroscopic level,to obtain specific characteristics and properties. The individual components are separate and distinct within the finished structure. It consists of two phases; one is termed as the reinforcement phase in the form of fibers, sheets and particles; which is embedded in the phase called matrix phase.The composite have many advantages over the other metallic materials (high strength to weight ratio, highly corrosion resistant, exceptional formability and durability etc). This paper highlights the contribution of composites in the fields of automotive. A comparision between the car of the 90's and present structure has been highlighted. Further their application in the automotive sector is discussed. In this sector Thermosets, Thermoplastics and CFRP composites play an important role. The reasons behind their use are discussed. The composite car structure and the importance of carbon fibers in this sector has been focused. Some examples of composite's success in this field is mentioned (namely JAPANESE DCF WHEEL, AUSTRALIAN CR-9 WHEEL, LAMBORGHINI'S "SESTO ELEMENTO" 2011).The Future innovations in the field of composite in these sectors to combat the challenges have been focused. In the automotive

sector the use of advance composites (combination of carbon and glass fiber) and the advantages they would provide are discussed.

Aluminium-silicon alloys with high magnesium content

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A new class of ternary Al-Si-Mg alloys (both hypoeutectic and hypereutectic compositions) containing high amounts of Magnesium (upto 3 wt.%) was developed. The microstructure consisted of fine spheroidal Mg_2Si and eutectic Si and/or primary Si particles dispersed in α-Al matrix depending on the alloy composition. The studies in hypoeutectic Al-7Si alloy show that an optimum amount of Mg and Sr have a modification effect on eutectic Si and Mg_2Si particles respectively. While in hypereutectic Al-14Si alloy, an optimum amount of Mg suppresses the formation of primary Si particles while refining the eutectic Si. Furthermore, an optimum combination of strontium and misch metal modifies and refines the eutectic Si and Mg_2Si phase. It was observed that T6 heat treatment accounts for enhanced room temperature tensile properties in both the alloy systems owing to almost complete spheroidization of eutectic Si and Mg_2Si particles.

Influence of processing parameters on Mechanical Properties of parts and theMicrostructure studies made by for Aluminum alloys using Direct Metal Laser Sintering

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Direct Metal Laser Sintering (DMLS) process is one of the promising Additive Manufacturing (AM) techniques, which is capable to build intricate parts directly from the 3D CAD data. This technology uses layer by layer deposition technique. In several industries like Automobile, Aerospace, Bio-medical etc., which need light weight component as well as various functionalities like high strength, low density etc., within a single component. Manufacturing of parts with aluminum alloys like AlSi10Mg using DMLS process corroborates the above said areas of application along with considerable design flexibility. This paper shows the influence of process parameters on mechanical properties (like strength, hardness) and micro-structure of aluminum alloy parts which are built by DMLS process. As there is very fast heating and cooling cycle is associated with DMLS process, therefore very fine microstructure of DMLS parts can be expected. In addition, the design flexibility allows novel structures to be incorporated in the marts which would not have been feasible with conventional designs. Improvement of mechanical properties of AlSi10Mg alloy with respect to the conventional process as well as introduction of design variations leading to stronger products opens a new area of application for the material of pressure shell can be used for underwater application. In this work some studies were made with respect to design novel structures and as well as applying process parameter control for variants of shell design used in underwater vehicles/devices for various depth of application. These studies were carried out on parts made using AlSi10Mg alloy powder on the DMLS process.

MATERIALS FOR DEFENSE

Invited talks

Session 1: Nov 14, 2014: [08.30- 10.30]

Tungsten Heavy Alloys for Defence applications

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Tungsten heavy alloys (WHA) are two-phase P/M composites consisting of nearly rounded BCC tungsten grains (20-50 mm dia.) dispersed in a low melting temperature ductile FCC matrix containing metals such as W, Ni, Fe, Cu and Co. These alloys are processed by liquid phase sintering to near full density and they offer excellent combination of mechanical properties (tensile strength: 1000-1800 MPa and ductility: 10-30%), high density (16-18 g/cc), high modulus of elasticity, easy machinability, good corrosion resistance, high absorption capacity against X-ray and Gama-Ray. Therefore, the alloys are extensively used for a range of applications that include anti-tank penetrators, counter-weights, center of gravity adjusters, vibration dampeners and radiation shields. While the early generation heavy alloy were based on W-Ni-Cu, they were gradually replaced by W-Ni-Fe with W varying from 90-95%) as the latter class of alloys exhibited substantially superior mechanical properties. In the continuing quest for developing superior alloys which has been mainly driven by a constant demand for realizing penetrators with superior ballistic performance, alloying additions such as Co, Mo, Re, Mn and Ta have been incorporated in order to enhance the mechanical properties. Concomitantly, there have been improvements in the thermo-mechanical processing resulting in superior property profiles. Over a period of time (about 4 decades), there has been a significant improvement in the properties of WHA (doubling of tensile strength with same or superior ductility). Impact properties of these alloys have received considerable attention since they are a prone to considerable scatter possibly due to structural inhomogeneities introduced during P/M processing. DMRL has been pursuing tungsten heavy alloy development for a diverse range of defence applications since last four decades. Heavy Alloy Penetrator Project, Tiruchirappalli that is currently producing a variety of heavy alloy products for defence needs has been the result of joint efforts undertaken by DRDO labs and Ordinance Factory Board. A brief history of alloy development will be presented in this talk. The effect of alloy chemistry and thermo mechanical processing on microstructure and mechanical properties will be elucidated. Recent advances in these materials will be briefly covered. Finally, the application of these alloys in defence related applications will be highlighted.

High temperature protective coatings for defence applications

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A major challenge in technological development is to continue meeting the requirements of new materials for use in progressively more stringent conditions. In the material-environment configuration, the surface of a component is of vital importance in determining the optimum usefulness of the material. This has formed the basis for the development of coatings technology. The primary requirement of a protective

coating is to have qualities superior to that of the substrate in order to shield the component from an aggressive environment. The combination of the coating and the substrate is called a coated system and it includes one or more surface modification processes. The need for coatings in the field of high temperature arises when improved performance criteria cannot be met adequately despite new materials with superior physical, mechanical and metallurgical properties. Further, coatings are also necessary when operating efficiency and production economy have to be considered and an improvement sought. There are innumerable applications in defence where high temperature protective coatings are used. Two of these defence applications where protective coatings are critical for enhancing the life and performance of components, have been considered here. The operating conditions in gas turbine engines of advanced fighter aircrafts are usually severe in terms of complex mechanical loading and material degradation mechanisms involving oxidation, hot corrosion and excessive heat load. For the efficient functioning of such turbine engines, Ni-base superalloy components such as blades and nozzle guide vanes are provided with a protective coating called thermal barrier coating (TBC). Thermal barrier coatings (TBCs) not only provide insulation against heat load, but also protect the superalloy substrate from high temperature oxidation. Over past several years, DMRL has been involved in the development of TBCs for gas turbine engine applications. The various aspects of these coatings, including the recent developments made at DMRL and elsewhere in the world, will be presented.The other defence application where protective coatings are being developed at DMRL is for the components of hypersonic test demonstrator vehicle (HSTDV). The extreme speeds (> 6 mach), rarified atmosphere and high temperatures that are involved in this application call for special substrate materials (e.g. Nb-alloy, zirconium diborides and C-SiC composites) and protective coatings (e.g. alloy silicide and yttrium silicate coatings). The details of the various coatings that are being developed at DMRL for HSTDV program of DRDO will be discussed.

Contributory papers

Session 1: Nov 14, 2014: [08.30- 10.30]

Indigenous development of aluminium alloy extrusions at industrial scale for defence applications

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Aluminium alloys AA 2219 and AA 6082 extrusions of various sizes are being imported for various defence applications. DMRL took the responsibility for developing and manufacturing these aluminium alloy extrusions in the country. These materials were successfully manufactured at Ordnance Factory, Ambajhari, Nagpur meeting all the specified properties required for their applications. These extrusions have been produced in T8511 condition in sizes of 40 mm, 60 mm and 85 mm diameter for AA 2219 alloy and in T651 condition in sizes of 60 mm, 100 mm and 150 mm diameter for AA 6082 alloy, respectively. The present paper will deal with the processes involved in the development and manufacturing of these alloy extrusions.

Development of homogeneous weldable steel armour plates for Indian defence

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This work is a part of the indigenization efforts of development of special alloy steel plates for Indian defence. A program was undertaken at Essar Steel to manufacture indigenous advance high strength alloy steel plates for main battle tanks of Indian defense. In this paper the development of homogeneous weldable steel armour plates for manufacturing main battle tanks are presented. Industrial trials of one heat of 150 MT were casted from which plates in 20 to 80 mm thickness were rolled. Detailed microstructural characterization and typical industrial problems associated during production of thick plates for battle tanks have been investigated. Specific problems and issues such as microstructural banding, central segregation and inclusion control are presented, along-with discussion on ways to overcome these problems at industrial scale. The key metallurgical factors contributing to the ballistic resistance in terms of hardness, toughness and microstructural homogeneity are discussed. Special heat treatment processes to avoid segregation as well as banding in these plates are discussed. Finally, the results of the plates rolled and tested at field trials are presented.

Certification of Ti-6Al-4V ELI grade TIG weldments for fabrication of high pressure gas bottles for missile applications

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High Pressure Gas Bottles for missile applications are designed to withstand a pressure of 400 bar taking into consideration of temperature which they may encounter during their life time of approximately 12 years. These air bottles are fabricated from Titanium alloy Ti6Al4V ELI grade material. Ti6Al4V ELI grade is similar to Ti6Al4V, except that ELI grade contains reduced levels of oxygen, nitrogen, carbon and iron. ELI stands for “Extra Low Interstitials”. These low interstitials provide improved ductility and better fracture toughness for the Ti6Al4V grade material. Due to this reason this material is extensively used in the manufacture of components for aerospace, missile and cryogenic applications. Furthermore, this material has high resistance to stress corrosion cracking (SCC) and hence is the obvious choice for applications which require both high strength and corrosion resistance. In addition, it has good machinability and excellent mechanical properties, high strength, low weight ratio.The welding technology for the gas bottles is well established by TiG welding route. In this process, material will be scooped out from forged bars to make a cylinder and two hemispherical domes will be welded on either side of the cylinder. This paper summarizes the indigenous efforts towards certification of Ti6Al4V ELI grade TiG weldments for fabrication of high pressure gas bottles for missile applications.

Indigenization of AA2219 alloy in the form of unclad plates and alclad sheets & plates for defence applications

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Requirements of Alclad and unclad AA2219 rolled products for critical defence applications were being met by import till recently. These materials having unclad and Alclad plate thickness ranging from 12-30 mm in 'O' and T87 tempers and Alclad sheet thickness ranging from 2-4 mm in T83 temper have been successfully indigenized by DMRL at BALCO, Korba meeting all the desired properties. This paper discusses the technical challenges met by DMRL at various stages of processing of these materials. The microstructure and mechanical properties of the resultant materials are discussed.

Fe-base amorphous coatings for marine applications

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Fe based bulk metallic glass (BMG's) is new class of materials that exhibits outstanding properties that are often not achievable in conventional crystalline materials. Fe base BMG's are generally known for their merits of high hardness, wear resistance, superior corrosion properties and relatively low cost. In order to widen the industrial application, significant attention has been paid to BMG's coatings which have potential to be extensively used in corrosive environments. Among various sprayed techniques, high-velocity oxy-fuel (HVOF) spraying is more suitable for preparation of amorphous metallic coatings due to the high kinetic energy and relatively low spraying temperature, which favors the formation of an amorphous structure with less porosity and superior bond strength with the substrate. In the present study, Fe base amorphous is powder prepared by atomization technique. Synthesized Fe base amorphous powder is sieved into useful sizes range (≥ 15 µm and ≤ 53 µm) for HVOF coating applications. The same Fe base alloy or composition is synthesized in the form of metallic ribbons (3 mm width and 50 µm in thickness) using melt-spinning technique for comparative study. Fe base amorphous powders were coated on steel substrates to achieve 200 µm and 400 µm thick coatings. XRD analysis indicated that Fe base coatings are amorphous in nature. Microstructural characterization revealed that the Fe base amorphous coatings possess dense structure with ≤ 0.2% porosity with compact bonding with the substrate. These coatings have a high hardness value around 700 VHN. The corrosion resistance of the HVOF coated samples and metallic ribbons in 3.5% NaCl solution were studied using potentiostat. Corrosion test results show the HVOF coated samples and metallic ribbons exhibit very high corrosion resistance with high passive range comparable to cast duplex stainless steel. The studies indicated that these coating are very much suitable for marine applications.

MATERIALS FOR ENERGY SECTORS: THERMAL, SOLAR, HYDRO AND WIND

Invited talks

Session 1: Nov 14, 2014: [08.30- 10.30]

Fuel for Fuel cells: Novel Catalysts for Hydrogen generation and processing

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Fuel cells, particularly PEM fuel cells hold great promise as pollution free providers of electricity for both stationery as well as mobile applications. These fuel cells need hydrogen as fuel to produce electricity. At present, H_2 has to be produced from fossil fuels or from renewable fuels. Though, presently H_2 is produced commercially in refineries and for fertilizer production, there is no storage or distribution network. Hence, there is a need for on-site generation of H_2 using primary fuels, till such time that H_2 storage and distribution systems are developed. At present, only fossil fuels such as natural gas, LPG, gasoline and diesel qualify as primary fuels because of their easy availability and wide distribution network. Hydrogen for PEM fuel cells has to be mostly CO free (<10ppm), whereas syngas produced by reforming contains 8-10 vol% CO. Hence, it is processed in multiple stages to make it suitable for PEM fuel cells. These fuel processing steps involve many consecutive steps that finally yield CO free (< 10ppm) hydrogen. Some of the important steps are (ii) reforming (steam reforming/ autothermal reforming), (ii) water gas shift (WGS) and (iii) preferential oxidation of CO or methanation of CO. National Chemical Laboratory, Pune, has been spearheading the development of PEM fuel cells for distributed generation of power, for which onsite generation of H_2 is important. The presentation gives a brief overview of various H_2 generation and processing steps (for removal of CO) that involve novel catalysts developed at NCL, Pune.

Session 2: Nov 15, 2014: [11.00- 13.00]

Importance of Materials of Wind Mill Towers

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Materials & Metallurgy is one of the key considerations particularly for the energy sector. Wind mill is one of the large sources of the green energy since last decade, and its worldwide development is eye catching. Material of construction of the wind mill tower play key role in the development and growth of the same. This structure is responsible to carry the total load of the Turbine & Blades. Depending upon the direction of the wind this complete assembly over the Tower rotates 360 degrees. This has to bear the wind velocity of 6 meters per second & more continuously, as well as has to resist against the big storms time & again.. Majority of the locations being near to Sea areas, it has to sustain against the salty atmospheres. The Author discusses the cycle of the development of the towers, which is the main load bearing component

of the wind mill. The selection of the type of material, its grade, Chemical composition, Physical & Mechanical properties, corrosion parameters, etc determines the capacity and life of the wind mill, in addition to the overall cost considerations. The life of this is warranted for minimum period of 20 years. Initially the structure was constructed with different size of angles, likein transmission towers, which was modified to tubular towers having totally closed & lockable arrangements, .From which, again of late, these are designed having mixed structure of the angular & tubular types. The journey is interesting & the key role of Technological Developments is described. The Global competition provides encouragement for such developments.

Processing and applications of iron aluminides

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Among the intermetallics, Iron Aluminides based on Fe-40Al have been investigated as possible replacements for a wide variety of structural and functional applications due to their low density, high strength, and excellent oxidation and corrosion resistance. Several approaches were considered to process iron aluminides and enhance the strength, creep resistance and rupture life of Fe-40Al alloys while maintaining or enhancing the ductility. In this paper, we will discuss a novel manufacturing technique employed to process FeAl sheets by cold rolling of compacted sheets prepared by using water atomized FeAl powders along with a polymeric binder followed by annealing and rolling until a fully dense sheet is obtained.. In this paper, we examine the microstructural evolution of FeAl sheets processed by roll compaction and annealing. The FeAl sheets exhibit fine microstructure with a dispersion of Al_2O_3 particles throughout the matrix . It is found that the dispersoid particles pin the grain boundaries giving rise to smaller grains sizes. Interstitial carbon is seen to remain in solution, but is precipitated as fine perovskite on ageing. We also discuss the effect of microstructural state on the material hardness, grain boundaries and influence of precipitate or dispersoid particles. We look at the applications of FeAl in oxidation, carburization and sulfidation atmospheres and show that FeAl alloys are superb as compared to a wide variety of materials at high temperatures.

Contributory papers

Session 1: Nov 14, 2014: [08.30- 10.30]

Experimental studies of electroplating on copper substrate for development of solar receiver material

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Open air porous volumetric receiver based concentrating solar tower technology can be employed for process heating. The role of solar receiver is to transfer solar radiation energy received from reflectors to the fluid (air) flowing through the receiver. In order to have efficient heat transfer, the solar receiver material needs to have high thermal conductivity and reasonable resistance to oxidation at operating

temperature (500̊C in present application). It is well known that copper has good thermal properties but it has less resistance to surface oxidation. While, it is observed that brass shows reasonable resistance to oxidation at 500̊C. In present work, the experimental studies is done to evaluate feasibility of having brass coating on copper substrate so that material has good thermal properties and resistance to surface oxidation. Initially, experiments with brass coated copper substrates shows that zinc from brass surface diffuses into copper substrate and it doesn't provide stable surface layer. To overcome diffusion of zinc at higher temperature, a diffusion barrier layer Nickel is evaluated. Electroplating experiments are done to deposit a Nickel layer on copper substrate, followed by copper and zinc layers where it has layer thickness of the range 20µm. Later post flame heating of electroplated sample, it is observed that copper and zinc converted into brass. In order to study the diffussion barrier characteristic of nickel layer for zinc elements, receiver sample is heat treated where it heated to 450°C for 2 hrs. SEM-EDS analysis carried out for receiver sample to confirm the zinc diffusivity into substrate through nickel layer, the results indicated that there is almost no change in chemistry before and after heat the treatment of the sample.

High temperature low cycle fatigue behaviour of a Ni based superalloy, Haynes 282 for ultra super-critical steam turbine applications

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Recently developed Ni based superalloy Haynes 282 has gathered much research interest for applications in hot gas path components in ultra supercritical steam turbines. It is a gamma-gamma prime alloy with uni-modal size distribution of gamma prime. Variations in cooling rate (CR) from solutionizing temperature has been found to have a significant effect on gamma prime size and lattice misfit between the matrix and gamma prime precipitate in this alloy, which in turn control the high temperature low cycle fatigue (HTLCF) behaviour. HTLCF behaviour for different microstructures as a function of test temperatures (650-760 deg C), and strain amplitude (0.4-0.8%) would be presented in this paper. Comparative performance of different microstructures in different regions of strain amplitude-temperature space would be presented for this alloy. Performance of different microstructures depends on underlying deformation mechanism. Hence a deformation mechanism map as a function of temperature and strain amplitude as constructed based on TEM study would be presented.

Influence of niobium on the drop weight tear test property in linepipe steels

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In the late 1960s it was found that brittle fracture propagated through a large distance along the length of the pipeline at very high speeds while the ductile fracture propagated at much lower speeds and to a shorter distance before being arrested. Hence the presence of ductile fracture on the fracture surface of a test specimen became a criterion for determining the crack arrestability in linepipe steel, which then led to the development of drop weight tear test (DWTT). Linepipe steel composition has significantly evolved since its first application in the 1950s. Significant advancements in the steelmaking technologies such as

low interstitial control (C<0.05% and N<50ppm), impurity control such as sulphur (S<20ppm) and phosphorous (P<0.010%) to ultra-low levels, and clean steel with respect to inclusions can be attributed to the stringent requirements for linepipe steels. Also, the advent of the thermomechanical control rolling with accelerated cooling technology resulted in the production of steels with finer final microstructures of ferrite, ferrite-pearlite, acicular ferrite or dual phase microstructures like ferrite-bainite and bainite-MA (Martensite-Austenite combination) depending on the alloy design and the processing parameters chosen.Niobium is a critical element in linepipe steels for obtaining better DWTT property and plays the following significant roles during the processing of linepipe steels: 1) control of austenite grain size during reheating and roughing rolling, 2) restrictingaustenite grain growth during the hold time between roughing and finishing rolling, 3) enhancing strain accumulation in austenite during finishing rolling, and 4) promoting the non-equilibrium acicular ferrite microstructure during austenite transformation while cooling. The present article will focus on the above mentioned effects of niobium and will highlight how these influences can be exploited during the hot rolling of linepipe steels to achieve better DWTT values.

Interaction of alumina with liquid Pb83Li17 alloy

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Eutectic lead lithium (Pb83Li17) alloy is being considered a coolant, neutron multiplier and tritium breeder for International Thermonuclear Experimental Reactor (ITER) and Fusion Power Reactors (FPR). In order to reduce the magneto-hydrodynamic drag (MHD) and to prevent corrosion of structural materials due to the flow of lead lithium (Pb83Li17) alloy, alumina (Al2O3) is proposed as a candidate ceramic coating material. Interaction of liquid Pb83Li17 alloy with Al2O3 at the operating temperature of these reactors is therefore an important issue. The present paper deals with the characterization of Pb83Li17 alloy and its interaction with Al2O3 at the reactor operating temperature. The interaction was studied using EPMA, XRD and thermal analysis technique. The result indicates that alumina can interact with Pb83Li17 alloy at 550 ºC even in high purity argon atmosphere. The role of oxygen in the interaction process has also been discussed.

Diffusion properties of Nb(Al) solid solution

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In the recent past, there has been lot of interest in development of refractory alloys for hightemperature structural applications. Niobium compounds, due to their low density and highmelting point, are potential candidate materials for high temperature applications. Most of these high temperature alloys are aluminides because of their high melting point, low densityand resistant to high temperature oxidation resistance at high temperatures. The performance of these materials during operation is strongly dependent on the high temperature degradationphenomena such as oxidation, creep etc., which are in turn primarily dependent on the diffusionproperties of the materials. In the present study the diffusion characteristics in the Nb(Al) was studied using diffusion couples of pure Nband Nb-6.5 at% Al (a dilute alloy of Nb-Al) in the temperature range 1273 K - 1473K. Concentrationprofiles across the interface of the couples were determined using electron probe micro-analyser (EPMA). The interdiffusion coefficients (*D*)

in the Nb(Al) solid solution at various concentrationswere determined using Boltzmann-Matano method and were found to be in the rangebetween 1.56 X 10^{-18} to 1.04 X 10^{-16} m^2/s. It was noticed that the values of *D* increased with theincrease in Al concentration and followed a linear relation. The temperature dependence wasalso established and the activation energy (*Q*) for interdiffusion ranged between 172 to 220 kJ/mol. The value of *Q* decreased linearly with the increase in the Al concentration in the Nb(Al)solid solution.

Process optimization for obtaining quality amorphous ribbons of FINEMET alloys for ultra soft magnetic applications

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Dual phase FeSiBNbCu (FINEMET) alloys consisting of nanocrystalline Fe_3Si phase embedded in an amorphous matrix are potential candidates for replacement of conventional soft magnetic materials in energy savings applications. These alloys are generally produced by controlled heat treatment of precursor amorphous alloys in the form of continuous ribbons with good quality surface finish, which are prerequisite for the final applications. The quality amorphous ribbons are processed using a planar flow melt spinning (PFMS) technique which is a one step process to make continuous ribbons directly from the melt.DMRL has developed the technology for producing nanocrystalline soft magnetic FeSiBNbCu alloys. To produce thin, wide and continuous amorphous ribbons with good surface finish, a 1 kg capacity vacuum planar flow melt spinning equipment with unique capabilities, which are not available in other existing equipment has been established. In the present investigation, a Taguchi technique has been adopted to optimize process parameters to produce amorphous ribbons with good surface finish during planar flow melt spinning experiments. The alloy was melt spun to obtain rapidly solidified ribbons using an L16 orthogonal array varying process parameter such as wheel speed (V), nozzle-wheel gap (G), melting temperature (T) and ejection pressure (P). The images of puddles for all experiments were captured using high speed imaging system at a speed rate of 100f/s and analyzed using Image – Pro Plus software. The physical properties such as length, width, thickness, surface roughness and structures have been measured.The results are analyzed using analysis of variation (ANOVA) method.The percentage contribution of each parameter have been evaluated and correlated with the thickness, surface roughness and the structure of the ribbons.

Heat resistant steels for superheater tubes: effect of stable Cu rich precipitation

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The traditional ferritic creep resistant steels cannot be used above 600 ^{0}C temperature applications. As per recent trends, materials for super heater tubes need to maintain creep resistance strength and oxidation resistance properties beyond 600 ^{0}C and for these necessities, advanced austenitic heat resistant steels are used(most suitable for superheater/reheater tubes).All of the austenitic heat resistant steels commonly contain Nb element for inducing 'MC' phase precipitation strengthening. In order to further increase high temperature strength, COPPER [Cu] has been reported to be used in some of the austenitic steels to achieve strengthening effect with Cu rich phase precipitation.It is well established that precipitation

strengthening is the main strengthening used for these advanced austenitic heat resistant steels.[Cu] phase age hardening steel is also of considerable commercial importance due to its good strengthening effect and unique mechanical properties. The precipitation sequence, structure and composition of Cu-rich phase in ferritic matrix have been studied by different analysis technologies such as TEM, High Resolution TEM , SEM analysis, 3DAP analysis etc.The [Cu]-rich phase prcipitation behaviour during 650°C aging has been analysed using the above mentioned processes. The experimental results showed that Cu atoms can quickly concentrate in clusters at very early precipitation stage to form the fine nano-size Cu-rich "segregation areas".In order to understand Cu-rich phase precipitation strengthening mechanism, a typical Cu-added heat resistant steel 18Cr9Ni3CuNbN has been reported to be suitable for such applications of mechanical properties and various tests related to this steel have been discussed in detail in this REVIEW paper.

Session 2: Nov 15, 2014: [11.00- 13.00]

Slurry erosion studies of as-cast 23-8-N nitronic steel for hydroturbine components

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13/4 martensitic stainless steel used as a turbine material, is generally subjected to heavy-erosive wear and loss of efficiency in hydro power projects. In the present study, an attempt is made to develop an alternate material for hydro turbine applications. For this purpose 23-8-N nitronic steel were studied for slurry erosion performance. A comparative study has been carried out with presently used as- cast 13/4 and newly developed as-cast 23-8-N nitronic steels by means of slurry erosion tester. It is observed that the as-cast 23-8-N nitronic steel possesses excellent erosion resistance than 13/4 steel. SEM analysis showed that the erosion tracks developed on the as-cast 13/4 steel specimen was wider and deeper than that formed on the as-cast 23-8-N steel specimens for erosion test. This is attributed the fact that austenitic matrix of the nitronic steel possesses optimum hardness coupled with ductility, high tensile toughness, which results in higher erosion resistance.

Process technology for production of APIX70 HR coils at BSL for spiral welded pipes

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To establish process technology for production of API X70 HR coils for spiral welded pipes, two trial heats were made through BOF-LF-CC route. The concast slabs were processed into 7.1 mm thick HR coils under suitably designed process variables to (a) ensure total dissolution of primary carbides during reheat practice, (b) engineer fine austenite grain size prior to entry into finishing mill through controlled roughing

schedule, and (c) control ferrite grain size to less than 5 micron through low finish rolling and coiling temperatures. Microstructure of Nb-V-Cr HR coils manifested very fine ferrite-pearlite structure (ferrite grain size : ~3 µm), while acicular ferritic structure was observed in Mo bearing HR coils Desired properties were achieved in Mo bearing steel, HR coils of this steel were processed into 28 mm OD HSAW pipes. Specified mechanical properties like tensile (YS : 511-550 MPa, UTS : 589-646 MPa, %El : 34-38%, YS/UTS : 0.84-0.87, impact toughness in base (280-296 J) , weld (184-196 J) & HAZ (184-236 J) and 100% shear area in DWTT (at 0oC) were successfully met in pipes. Surface of the rolled pipes were found free from any defect with no incidence of edge lamination. All the pipes passed the hydraulic, ultrasonic and radiography tests.. As per trial results, Mo bearing HR coils of API X70 of BSL were found suitable for making of HSAW pipes.

Thermodynamics and kinetics of VH_2 decomposition

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Vanadium and its alloys could be potential candidate materials for on board hydrogen storage applications because of their high hydrogen storage capacity (~ 3.8 wt % better than AB_5, AB_2 & AB intermetallic). Hydrogen de-sorption from VH_2 is two steps process: (1)$VH_2(\gamma)_{(s)} \leftrightarrow ½ V_2H (\beta)_{(s)}$ and (2) $V_2H (\beta)_{(s)} \leftrightarrow 2V_{(s)} + ½ H_{2(g)}$. First step de-sorption takes place at the ambient conditions while the second step requires high temperature (470K). Thus only half of the total storage capacity is available on subsequent sorption cycle. Cyclic hydrogen storage capacity of VH_2 system could be enhanced by tailoring the thermodynamics and kinetics of desorption process. We have investigated the plateau pressure changes of 1st part of the reaction and change in thermodynamics of second part of the reaction with respect to the alloying element Ti, Zr, Nb, Mo and Cr. The details of the results will be presented.

Modified CdSe nanowires, radially grown on TiO_2 nanowires, for realization of "Rainbow" solar cell

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Achievement of low-cost-high-efficiency photovoltaics has become promisingly possible due to use of Quantum Dots (QDs) as photosensitizers due to their lucrative properties of size tunable band gap energy and composition control, high molar extinction coefficient and large intrinsic dipole moments for enhanced charge separation. The redox-couple-deposited-on-large-bandgap-semiconductor-oxides architecture of Dye Sensitized Solar Cells (DSSCs) has to be ameliorated to render it suitable for Quantum Dot Sensitized Solar Cells (QDSSCs). Recent efforts has seen introduction of recombination barrier layer between TiO_2 and QDs, protective layer between QDs and electrolyte for enhancing photovoltage, reducing dark current and corrosion/ hole mediated oxidation of QDs, increased photocurrent, grafting molecular dipoles between QDs and a nanometric protective layer, to facilitate electron injection without suffering loss in open circuit voltage (V_{oc}).Fabrication of a "Rainbow" solar cell consisting of QDs stacked inside a T_NT to absorb visible light was proposed recently, indicating that stacking QDs of different sizes shall allow maximum visible light absorbance thereby placing us in the vicinity of theoretically reachable quantum efficiencies. However, it is important to realize that this proposed increase in efficiency is not only due to broad absorption of sunlight by differential absorption from a mixture of quantum dots of

varied sizes and bandgap energies, but also relies on the fact that when QDswith varied bandgaps are very closely packed in order of higher to lower bandgap energy, a QD of higher bandgap absorbs energy corresponding only to its bandgap and transmits the remaining lower energy to be absorbed by the next QD. Thereby, energy loss due to thermalization is avoided and harvesting of the broad visible spectrum of light can be realized. However, nanotubes of wide bandgap host oxides have dimension typically 10^3 times higher compared to the size of the QDs. Thus, the proposed architecture in this form (vertical stacking) is not practical due to the disparity in the scale of the deposited QDs and the host oxide nanowire. Radial stacking is a theoretically feasible idea, but not easy to achieve experimentally. In the present work, we use Bi nanoparticles as nucleation sites for CdSe nanowires as well as segmented CdS/CdSe nanowires with photoelectrochemical properties better than CdS or CdSe alone. Also, there are many modifications which can be made to the nanowires to improve their absorption energy range, Work is currently underway.

Improvement of tensile properties in 2-1/4Cr-1Mo low alloy steel by microalloying additions: L&T SSHF experience

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SA508Gr.22 CL.3 is a very popular steel grade used extensively for forged components in the field of hydrocarbons, oil and gas. One of the major drawbacks of this grade is the reduction of tensile strength after prolonged post welding simulation heat treatment which is generally a requirement of the equipment manufacturers.Shells made with grade SA336Gr.F22CL3, having same chemistry as SA508Gr.22CL3 with a min. tempering temperature of 710°C/715°C though met the tensile strength after Quality heat treatment, tensile values were falling well below the min. specified after maximum Post Welded Heat Treatment (PWHT) at 685±5°C for 26 hours. The challenge was to make shells of grade SA508 Gr.22 CL.3 of 45 MT finished weight (160- 225 mm thick) meeting the tensile strength requirements even after the max. PWHT i.e. 685°C for 26 hours. Micro alloying of Boron, along with minor additions of V, Ti and Nb were done as per the code requirements. Shells processed with this modification met the requirements of the code with respect to tensile, impact, hot tensile values.This paper describes the experience of LTSSHF in successfully developing the grade conforming to the stringent requirements of the customer

Materials development for efficient energy storage in batteries:From consumer electronics to automobiles

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Li-ion batteries have significantly contributed to commercial success of consumer electronic devices and in the verge of dominating transportation sector. Hence, the criterions and expectation on these batteries are pushing electrochemist to improve in terms of materials, mechanism, design or the cost that are superior to the current technologies. With this in view, new concepts are being tried to meet the challenges of sustainability and to develop battery materials which over smart the conventional electrode materials used. Progress in these directions crucially depends on developing novel materials with improved properties. In this context, new compositions of layered cathode materials have been developed which exhibit higher capacity, on the other hand nanomaterials of transition metal oxides, metals, alloy

composites etc which are previously disregarded for anodes showed exorbitant high capacity as negative electrode materials. Over the past decade, significant progress and efforts have been made in developing the new generation of Li-ion battery materials. This talk focuses on the recent developments of materials aspects in designing high performance Li-ion batteries and alternate systems such as rechargeable Na-ion and Li-air batteries.

Gold nanoparticle and quantum dots modified TiO2 nanotube based hole conductor free perovskite solar cell.

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TiO2 is a wide bandgap material (3.2eV) with interesting applications in optoelectronic field due to its porous Nanostructure. TiO2 Nanotube has a vertical charge transfer mechanism as well as it has an in-built continuous layer thus decreasing the diffusion length and recombination loss thereby enhancing the collection efficiency of the electrons atTCO substrate. It has been shown through simulation that Perovskite (PbCH3I3) has an ambipolar character; therefore, it does not necessarily require a p-type hole conductor but a n-type host is essential. Therefore focus of the current work is the use of Perovskitein combination with noble metal nanoparticlemodified TiO2nanotubes which enhances electron lifetime in the matrix. Furthermore, Quantum dots have been added to this model for synergistic visible light absorption. Moreover, use of expensive hole transport material (HTM) in the perovskite based photovoltaic devices hasalso added complexity in the device design to a great amount. Reports have shown perovskite itself can act as holeconductor. Thus eliminating HTM layer can lead to cost efficiency and simplicity in design with minimal sacrifice in cell efficiency. The solar cell structure followed in this work, which is also shown, is very successful and emerging photovoltaic design namely “Hole Conductor free Perovskite Solar Cell”. Preliminary results show respectable efficiency with good stability under 1 Sun illumination with a Xenon light illuminator.

MATERIALS FOR SPACE

Invited talks

Session 1: Nov 14, 2014: [08.30 - 10.30]

Manufacturing of special steels, superalloys and Ti-alloys for aerospace applications

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A wide spectrum of engineering materials ranging from metals and alloys to highly advanced ceramics, polymers and composites find application in aerospace sector. Adequate knowledge of service requirements and detailed information regarding material capabilities are absolutely essential for the designer to make right choice of materials. The designer working in aerospace industry goes through an elaborate evaluation of various characteristics of materials, while making his choice for intended application. Among the important criteria the designer adopts for material selection are – high strength to weight ratio, functional & environmental stability as well as high degree of reliability. Over the years, Midhani has catered to the requirements of Indian Space, Aeronautical and Defence sector for many high performance materials. A wide range of special alloys – many of them being tailor made to customer's specific needs are developed and supplied. Development of massive rings and plates meeting stringent customer's specification for PSLV and GSLV hardware has been the most eventful milestone. The materials supplied prominently include Special steels, Superalloys and Titanium alloys. The most important essentials for manufacture of aerospace materials include clean melting techniques, tight control of alloy chemistry and the analytical capability to characterize the product. This paper describes the processing of aerospace materials and their salient features.

Session 2: Nov 15, 2014: [11.00 - 13.00]

Materials for Indian space program: Achievements and the road ahead

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Successes in materials development technologies are critical to the success of space missions. This essentially arises from the fact that materials are subjected to hostile conditions during space flight. Materials in typical space launch vehicles are subjected to such extreme temperatures as 20K (boiling point of liquid hydrogen in cryogenic engine) to several thousands of Kelvins (as encountered in a re-entry vehicle nose cone). Further, the demand for higher specific strength materials puts additional burden on the metallurgists to develop complex alloys with exotic tempers. Finally, the materials should be fabricated to put to use and should withstand the aggressive/corrosive propellants. This demands materials with closely controlled properties processed under most stringent controls.Realizing the importance of materials in the space technology, Indian Space Research Organization (ISRO) has developed material technologies to cater to its needs and embarked on the indigenization of a number of advanced aerospace alloys. These alloys have been successfully inducted in to the space program and are routinely used in

PSLV/GSLV missions. Colossal contributions have been made in quintessential areas of materials research in the last four decades that paved way for the successful realization technologies to put India on the global map of space faring nations. This talk focuses on the achievements in the development of materials meeting the stringent aerospace quality control norms. The material requirements to meet the futuristic demands are also presented.

Aluminium alloys produced in Indian industries for strategic applications

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There has been a consistent requirement of a variety of wrought aluminium alloys in different semi-product forms and heat treatment tempers for manufacturing components in both primary and secondary structures of aerospace and defence applications. These requirements, till date, were being met through import from multiple sources resulting in issues involving quality, time, cost overruns and impediments of country's technological growth.This talk discusses the technical challenges met by DMRL while successfully indigenizing and developing eight varieties of wrought aluminium alloys in eight different semi-product forms in seven medium to large scale aluminium industries in the country.

Very high temperature stable materials and modules for aersopace vehicles

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Development of materials for very high temperature applications is a national priority area with respect to future aerospace technologies. The feasibility of realising long duration aerospace vehicles crucially depends on the availability of materials and modules having sufficient strength and oxidation resistance at very high temperatures, coupled with resistance to thermal shocks, all within usual weight budgets of aerospace vehicles. The talk gives an overview of various materials being developed for vehicles expected to perform at temperatures in the range 700°C to about 1700°C and high speed air flow for moderate durations. Work carried out at DMRL on C_f-SiC composites, ZrB_2 based composites, metal-ceramic layered modules for thermal protection systems, niobium alloy Cb752, nickel based superalloy foams and protective coatings will be described.

Contributory papers

Session 1: Nov 14, 2014: [08.30 - 10.30]

Nanostructured single- and double-bi-layered NiTi thin film shape memory alloy

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Recently NiTi thin film shape memory alloy has attracted much more attention due to its interesting mechanical properties, excellent chemical resistance, biocompatibility, which leads to the development of exciting industrial applications. To fulfil all the demand of durability, mechanical stability, dynamical coupling, chemical and physical compatibility, nanostructure multilayered thin film is going to be a demanding objective. In this research single and double bi-layers of Ni-Ti SMA thin film were deposited with the help of DC and RF magnetron sputtering technique. Layer-wise deposition of Ni and Ti on Si(100) substrate was performed with Ni as the bottom layer and Ti as the top layer. To get the crystallinity structure in the multilayer, the as-deposited thin film was subjected to annealing treatment. Influence of various annealing temperature on the physical and mechanical properties of as-deposited thin film were analyzed. Field emission SEM, grazing incident XRD, High resolution TEM, AFM, profilometry, and nanoindentation analyses have been done to reveal different properties of the as-deposited as well as the annealed samples. It was found that, increase in annealing temperature results in gradual increase in atomic cluster coarsening with improved ad-atom mobility in both the bi-layers. Phase analyses show the development of binary and ternary silicide phases and some intermetallic compounds (NiTi, $NiTi_2$, Ni_3Ti, Ni_3Ti_4). XTEM micrographs exhibit the interdiffusion between the two layer-constituents, especially at higher temperatures, which resulted either in the amorphization or in the crystallization after annealing at temperatures above 400 °C. Surface roughness at 400 ºC provided better smoothness that of the samples annealed at other temperatures for both bi-layer cases. Nanoindentation measurements helped to estimate hardness (H), reduced modulus (E_r) and wear properties.

High temperature oxidation behavior of titanium aluminide for RLV skin panel applications

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The aerospace industry is always on the lookout for materials which are light weight but having strength. As low density is the prime importance to aircrafts and space shuttles, it economizes the fuel consumption and enables them to carry more payload rather than structural load. Super alloys are widely used as materials for rotating components such as blades and vanes but these materials have high density and therefore heavy. Intermetallics have the combination of this high strength and low density making them suitable for such applications.Alloys in the Ti-Al system are of interest for high temperature systems such as aerospace engines because of its low density and substantial high temperature strength. These alloys form an alumina rich scale which provides good oxidation resistance. These materials have good creep strength. In the present work, the oxidation behaviour of Ti-28Al-9Nb-2.5Cr-0.025B (wt.%) was studied in

oxidizing environment at temperatures 700°C, 800°C and 900°C.This study discusses the kinetics and characterization of the alloys and proposes a realistic assessment for space shuttle skin panel applications.

Understanding phase selection in sol-gel synthesis of yttrium monosilicate

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Si-based non-oxide ceramic composites which are candidate materials for re-entry vehicles and hot sections in gas turbine engines suffer from silica volatilization in water vapour environment. Environmental barrier coatings (EBCs) made of rare earth silicates are used to mitigate water vapour attack. While yttrium monosilicate is an attractive EBC material, the main challenge remains in obtaining pure yttrium monosilicate phase. In this study, yttrium monosilicate (Y_2SiO_5) composition was synthesised by the sol-gel route. The subsequent phase transformations upon calcination and thermal treatment were studied up to 1600°C. It was observed that multiple phases formed after crystallisation of the amorphous powders. X-ray diffraction (XRD) studies confirmed the presence of monosilicate along with impurity phases such as the yttrium disilicate, apatite as well as free yttria in the pyrolysed powders. However, yttrium disilicate formation can be avoided by using highly acidic solution (pH 0). The acid catalysed gel pyrolysed at 800°C was found to be X-ray amorphous. Later, the gel was pyrolysed at 900°C, and the first crystallization products were found to be yttria and monosilicate (X1 polymorph). With subsequent thermal treatment up to 1300°C, apatite (yttria lean phase) was observed besides yttria and monosilicate. After prolonged exposure at 1400°C, the major phase was observed to be yttrium monosilicate with 5 vol% of Y_2O_3 as the only other phase. A thermodynamic model was constructed using two ionic sub-lattice models for the supercooled liquid. From the Gibbs energy versus composition curve, a submerged miscibility gap was observed at temperatures relevant to calcination and crystallisation. The formation of multiple crystalline phases during yttrium monosilicate synthesis can be explained on the basis of phase separation in the amorphous phase due to the miscibility gap, prior to crystallisation.

Oxidation behaviour of 2D Nicalon/SiC composites in static dry air and combustion environment

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The silicon carbide fiber reinforced silicon carbide matrix composites are promising materials for the hot sections of advanced aviation gas turbine engines as combustion liners, vanes and jet-exhaust vanes. In such applications, the composite is exposed to moisture-laden high-velocity and high-temperature gaseous combustion products. Hence it is necessary to study the degradation of these composites at high temperature combustion environments. In the present study, two dimensional Nicalon/SiC composite with

boron nitride (BN) interface fabricated by isothermal isobaric chemical vapor infiltration process (ICVI). To prevent the fiber and matrix from oxidation, a silicon carbide seal coating is applied to the composite by chemical vapor deposition (CVD) process. The oxidation studies of the SiC_f/SiC_m composites were carried out at 1200°C, 1300°C and 1400°C for various time periods up to 100 h, in static air and in a combustion gas environment. The durability and integrity of the composite has been investigated in combustion environment by using an oxy-acetylene torch flame apparatus. The change in the specimen weight due to oxidation was plotted against the time of exposure to determine the oxidation kinetics. The phase constitution of the as-received specimens and the oxidized specimens were investigated by X-Ray diffraction (XRD) studies. The microstructure and degradation of the composite after oxidation were analyzed by scanning electron microscopy (SEM) and elemental analysis was done by energy dispersive spectroscopy (EDS). The results from above analysis show that the most severe degradation of the composite occurs in combustion environment. It was found that the degradation of the top surface, the fiber and the matrix exhibit different morphological features.

Session 2: Nov 14, 2014: [11.00 - 13.00]

Establishment of first hafnium plant in India- Issues and challenges

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Hafnium (Hf), a reactive group metal with high melting (2227°C) and boiling point (4600°C) and high density (13.3 g/cc), is found in nature closely associated with its much more abundant sister element zirconium and hence is one of the costliest metals (costing @ Rs.2.00 lakh/kg & more). Its major application is in high temperature aerospace alloys (C-103 alloy) as rocket thrusters and nozzles. It is also used as control rods in nuclear reactors because of its ability to absorb neutrons. It has very high affinity towards oxygen and is a very good getter. Other applications include electrode in plasma cutting. Hafnium dioxide is a candidate for high-K gate insulators in future generations of integrated circuits. As oxide, carbide, nitride and boride, hafnium is used in various high temperature and optical applications. Zircon is most common ore of zirconium and is available in plenty in many countries including India. Zirconium content in hafnium should be reduced below a tolerable limit for applications in high temperature alloys and as control rods in nuclear applications. For its applications, VSSC has been importing Hafnium and wanted indigenous source. A feasibility study project was sanctioned to C-MET to extract hafnium from Zr scrub raffinate solution of NFC and on successful completion of the demonstration, a pilot plant project was sanctioned. 2.5 kg hafnium sponge was prepared and demonstrated to VSSC. A Quality control team of VSSC closely monitored the trial runs and certified the quality of the product at that stage. The Extended Pilot plant project with 320 kg Hafnium sponge / annum production capacity (which is sufficient to meet VSSC annual requirement) was then sanctioned to C-MET. The plant is now commissioned with first batch of Hf sponge of required specifications already produced and further process optimization trials continued. The raw material is zirconium raffinate solution (1-4 gpl of Hf, 30-40 gpl of Zr and other impurities like Fe, Ti, Si etc.) of NFC requiring 800 - 1000 kL/ annum to be processed. The process includes solvent extraction, calcination, chlorination, Kroll reduction and separation of Mg & $MgCl_2$ from Hf sponge. Separation of Zr from Hf was a very tough challenge. Hf is also pyrophoric in nature and has very high affinity towards oxygen. Hence, the other big challenge has been safe handling at each steps of chlorination, Kroll reduction and high temp.-high vacuum Mg distillation steps. In this presentation, the issues and challenges

of establishing the plant including manpower planning, building, electrical, safety, automation, system and equipments design and procurement, establishment, commissioning of the plants and operation of the plants will be discussed.

Development of Inconel 718 spherical gas bottles for oxygen storage

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In this paper, the details of metallurgical process for the production of Inconel 718 spherical gas bottles are discussed. The process involves (i) realization of hemispherical domes by closed-die hammer forging and (ii) welding of two hemispheres to realize a spherical gas bottle. Hemispherical domes were produced by hammer forging using shaped dies in the temperature of 980-1050°C. The grain size of the domes at all locations was finer than ASTM No. 5. The typical mechanical properties of the dome in the solution treated and aged condition were ultimate tensile strength (1370 MPa), yield strength (1208 MPa) and ductility (16.5 %). All the forged domes were solution annealed at 980°C for 1 h and air cooled. The solution annealed domes were machined to a predetermined wall thickness and two domes are joined by electron beam welding to realize a spherical gas bottle. The weld parameters were optimized to minimize micro fissuring. The weld joints were subjected to dye penetrant and radiography to confirm the soundness and integrity. The gas bottles were subjected to functional tests and qualified.

Cu based promising materials showing SMA effects & their characterization

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For the manifestation of shape memory effect the formation of martensitic phase is the essential criterion. The shape memory effect is shown by the alloys due to the microstructural phase transformation between austenitic phase and martensitic phase and vice-versa with increase/decrease in temperature. In order to synthesize a shape memory alloy, the possibilities of good martensitic formation is an elemental requirement. In this paper, the effect of composition for a Cu-Al-Mn alloy with varying alloying constituents and quaternary additions on the martensitic phase formation on quenching will be discussed. Different characterization techniques have been used in order to confirm the formation of martensite such as optical microscopy, XRD and DSC. A study of this nature would help in pin pointing alloy compositions for making effective shape memory alloys in the future.

Microstructure and mechanical-thermal properties of Cu-Cr-Zr-Ti alloy

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Cu-Cr-Zr-Ti alloy is used for fabrication of the inner wall of the liquid engine thrust chamber of a launch vehicle owing to a combination of high thermal conductivity and strength. This alloy responds to aging by precipitation of excess chromium as fine coherent precipitates. Small amounts of zirconium and titanium are added to improve the intermediate temperature ductility by fixing the elemental sulfur. In the present investigation, the microstructure, mechanical and thermal properties of the indigenous Cu-Cr-Zr-Ti alloy were characterized using optical/electron microscopy, hardness, tensile testing and laser flash apparatus. In solution treated condition, four phases were observed, namely copper matrix, chromium-rich, TiS_2 and ZrS_2 particles. Upon aging at 500°C for 2 h, uniform distribution of chromium precipitates was observed throughout the matrix. Best combination of strength and ductility was obtained by solution treatment and aging: ultimate tensile strength of 330 MPa, yield strength of 212 MPa and ductility of 23%. The thermal conductivity of the alloy was found to be 280-320 W/m.K in the temperature range of 27-500°C.

Invited talks

Session 1: Nov 12, 2014: [13:30 - 15:30]

Different aspects of developing creep resistant steels

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Thermally activated movement of dislocations leads to the creep deformation in material. Creep deformation proceeds with the nucleation, growth and coalescence of creep cavities leading to failure. To increase creep strength, it is required to increase the creep deformation resistance as well as the creep cavitation resistance. Naturally, the philosophy of development of creep deformation resistant material aims at the retardation of movement of dislocations both by glide and climb. All the strengthening mechanisms like solid solution, precipitation / dispersion, reduction in stacking fault energy etc. except the grain refinement have been extensively used for enhancing creep deformation resistance of materials. Restriction of dislocation movement through the formation of dislocation substructure produced on imparting prior cold work and phase transformation etc. has been widely used to increase creep resistance of materials. However, stability of the dislocation substructure and precipitates under creep exposure is a concern for long-term creep strength and could be partially addressed through appropriate alloying. Climb of dislocation to surmount different obstacle to dislocation motion is associated with the self-diffusion of material. Crystal systems having close-pack structure and high melting point are preferred for higher creep resistance from the dislocation climb point of view. Alloying resulting in the reduction of self-diffusion retards the climb rate to enhance creep strength. All these aspects of creep strengthening mechanisms are discussed while considering the development of creep resistant ferritic and austenitic steels. Another concern in high temperature materials is the grain boundary sliding which occurs above the equicohesive temperature. Inadequate accommodation of stress at grain boundary irregularities like grain boundary triple point, ledges, precipitate etc., raised due to grain boundary sliding, leads to the intergranular creep cavitation causing premature creep failure. Therefore, it is required to increase grain boundary sliding resistance as well as to facilitate the relaxation of stresses raise due to grain boundary sliding to increase creep cavitation resistance. Several methods including serrated grain boundary, precipitate on boundary, enhancement of viscosity of the boundary on micro-segregation with highly surface segregating elements etc. has been adopted to decease grain boundary sliding. Further, over-strengthening of matrix than that of grain boundary should be avoided to facilitate the relaxation of stresses resulting from the grain boundary sliding. The creep cavitation aspect to enhance the creep rupture strength has been illustrated considering the creep behaviour of precipitation hardened austenitic steel, microalloyed with boron and cerium.

Session 2: Nov 12, 2014: [16:00 - 18:00]

Mechanical behavior of Aluminum alloys at very high strain rates and temperatures

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Aluminum alloys are increasingly used in automotive and defense industries. For both applications knowledge of their constitutive behavior over a wide range of temperatures and strain rates is required. In this presentation, methods and results from quasi-static and dynamic compression experiments will be discussed in the context of very high strain rates and high temperatures. A combination of conventional and miniaturized Kolsky experimental methods were used to achieve strain rates in the range of 1000 to 100000/s. The Kolsky apparatus was also combined with a high temperature setup to load materials upto 0.8 T_m. Experiments on aluminum alloys from the 5xxx and 2xxx series showed differences in the rate sensitivities and also, the thermal softening behavior as a function of strain rate. This behavior can be attributed to the kind of barriers to the dislocation motion. Furthermore, it was found that the existing constitutive models couldn't capture the stress vs. strain response accurately due to the dependence of deformation mechanisms on strain rate and temperature.

Session 3: Nov 13, 2014: [09:00 - 11:00]

The mechanics of damage evolution in engineering materials

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Damage represents the state of deterioration in materials that lead to failure, disintegration or fracture. It is important therefore that the evolution of damage in engineering materials be understood well so that their destruction is preventable. The manifestation of damage in materials when they are subjected to fatigue cycling or overloads may be understood from various perspectives. It may suffice, in many instances, to simply characterize the damage in terms of fractographic or microstructural features. One may comprehend and quantify it at an appropriate length scale, usually at micro- or meso- level. The progression of damage in the material under specific types of loading, e.g. under torsional, seismic or high rate of deformation, may be of interest. The role of specific microstructural constituents in promoting or preventing the accumulation of damage may be an aspect that requires elucidation. Material deformation behaviour may be modeled to represent, and predict, the development of damage in it. Some of these premises are exemplified in this talk to highlight the mechanics behind the evolution of damage in metallic materials. In order to understand the process through which damage is incorporated in a material due to the imposition of mechanical factors, it is instructive to trace the concatenation of events at the microstructural level that lead to amalgamation of defects responsible for the appearance of mesoscopic flaws, which in turn propagate to ultimately result in fracture. The recognition that the whole chain of events in a continuous process helps in comprehending why and how correlations exists between various points in this chain. A number of specific damage processes will be presented to underscore the existence

of such correlations. Modeling is a powerful tool to represent the progression of damage. While scale-hopping realistic models to portray damage from atomistic to component levels are yet far away, substantial progress has taken place to enhance multiscalarity of concepts and conclusions. However the assumptions inherent in such models and difficulties in experimentally obtaining model parameters, together with the computational prowess necessary, often make results and predictions unattainable or inconclusive and unreliable. There are of course hybrid variants that are able to capture the overall behaviour of the material without incorporating some of the finer details, and to a great extent help visualize the evolution of damage in material microstructures and predict their behaviour at an engineering scale. Instances of application of such models will be discussed. The overall application of domain of material mechanics is enormous. The importance of the mechanics of damage in materials is not insignificant, and the talk will highlight the scope and potential in this regard.

Session 3: Nov 13, 2014: [13:30 - 15:30]

A glimpse on the role of pre-strain on mechanical behaviour of materials

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Several structural components, for example naval structures, automotive components etc., are cold deformed into shape and welded together and cannot be often stress relieved due to their prior history. As a consequence, the materials enter into service in the cold deformed condition. Cold deformation is essentially a pre-straining process that alters the dislocation substructure of a material. The manner in which these structures subsequently respond to deformation, fatigue or fracture is thus of significance to the engineering community. It is well established that pre-straining in general increases strength with attendant reduction in ductility. But the mechanics of fracture or fatigue is intimately connected with dislocation dynamics; thus it is naturally expected that cold deformation or pre-strain would have profound influence on the fatigue or fracture behaviour of structural materials and would also affect their tribological characteristics or mechanical properties related to the above phenomena. This report addresses some of these issues based primarily on research results generated at the author's laboratory. At the outset the influence of pre-strain on strength and ductility of commercial sheet metals of IF steel would be discussed with an emphasis to illustrate the influence of pre-strain on ductile fracture behaviour high-lighting the fact that void size depicts a signature of pre-strain. This is followed by revelation of an interesting fact that strength of spot welds in steel sheets used for automotive applications increases with increased pre-strain. But the influence of pre-strain on tribological behaviour is unusual. Simultaneous increase in wear rate and hardness occur up to some critical value of pre-strain which is contrary to Archard's law; this has been explained considering the ease of nucleation of voids/microcracks and their subsequent propagation during sliding wear of pre-strained specimens. The fracture toughness of HSLA steels, J_C, is found to remain invariant up to 2% pre-strain, beyond which it decreases deleteriously. The nature of variation of fracture toughness with pre-strain has been explained and modeled in terms of the variation of the tensile flow properties of the steels with pre-strain and has also been examined considering stretch zone width and depth. It is found reported that fatigue limit increases with increased pre-strain due to surface compressive residual stress, fatigue threshold first increases and then decreases being controlled by both dislocation density and micro crack nucleation, fatigue crack growth rate is not considerably governed by the pre-strain, but the number of cycles for accumulation of some critical percentage of true ratcheting strain increases with the amount of pre-strain. The influence of pre-strain on

the flow characteristics and mechanical behaviour of materials can be visualized in-depth by thermal activation analyses; examples are cited from some current results generated on different materials.

Session 5: Nov 13, 2014: [15:30 - 17:30]

Control of microstructure by high temperature deformation processing

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The mechanical behavior of materials is dependent on its microstructure of which the grain size and shape is an important feature. Thermomechanical processing of materials is a common technique used for microstructural modification. A simple thermomechanical processing technique is the working of materials at high enough temperatures where non-conservative motion of defects result in either dynamic recovery or dynamic recrystallization. Of these the process of dynamic recrystallization is important as it results in the formation of new strain free grains. The manifests in the stress strain curve reaching a maximum followed by either a gradual decrease in the stress to a steady-state value or the occurrence of multiple peaks (oscillations) in stress finally leading to a steady state. Depending on the applied strain rate and temperature of hot working, the resultant microstructure is either refined or coarsened. This talk will present the microstructural evolution of various hot deformed metals and alloys carried out by our group. These will include Zr alloys (such as Zr-1Nb, Zr-2.5Nb, Zircaloy-4), cobalt (in both the wrought and sintered condition), SS304 and 316, natural uranium and U-9wt%Mo alloy, Nb, Nb-1Zr and Nb-1Zr-0.1C alloys, V and V-4Cr-4Ti alloy, and Mo-Ti-Zr alloy. In each of these alloy systems the relation between strain rate sensitivity and microstructural features correspond to either dynamic recovery and recrystallization will be presented. The microstructures (optical as well as electron back scatter diffraction maps) of regions of high strain rate sensitivity will be contrasted with regions of low to negative strain rate sensitivity. The kinetics of dynamic recrystallization based on the Avrami-type relation will also be presented for some alloy systems.

Session 6: Nov 15, 2014: [14:00 - 16:00]

Microstructure evolution and relation with flow properties of polycrystalline materials—A revisit

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Plastic deformation of crystalline materials results in several imperfections to control their flow properties, which can be designed according to material applications. Hot working gives combinations of line and interfacial imperfections simultaneously whereas the same is achieved by a combination of cold rolling and annealing. Cold working causes strengthening by the increased dislocation density but hot working concurrently results in hardening, softening or pseudo-steady state flow. Depending on tensile test condition and initial microstructure, all these changes occur to varying extent, which result in the variation

of flow stress with strain. The present work highlights a range of microstructure evolution and its effect on the nature of stress-strain curves and relevant constitutive relationship for deformation.

Contributory papers

Session 1: Nov 12, 2014: [13.30 - 15.30]

On manifestations of planar slip during low cycle fatigue of 316LN stainless steel with varying nitrogen content

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Influence of planar slip on cyclic deformation and fatigue life of 316LN stainless steel (SS) is investigated, taking into the account varying nitrogen content in 316LN SS (0.07, 0.11, 0.14, 0.14 wt. % N). The source for the origin of planar mode of deformation is found to determine the consequences of planar slip, and associated hardening/softening behavior and fatigue life. At room temperature, planar mode of deformation is evidenced in the form of closely packed coplanar accumulation of dislocation arrays. This has lead to a pronounced cyclic softening of the material which is found to increase with increasing N content. In contrast to the above, at high temperatures in the range 773-873 K, planar mode of deformation is evidenced in the form of planar slip bands and is found to cause considerable matrix hardening, particularly for strain amplitudes $\leq$ 0.6 %. Two mechanisms namely, dynamic strain aging and secondary cyclic hardening are found to offset the cyclic softening, and strongly induce the planar slip at above mentioned temperatures. Nitrogen content in 316LN SS is found to enhance the planar mode of deformation. It is important to mention that planar mode of deformation not only altered the cyclic stress response behavior from cyclic softening to cyclic hardening, but is also found to cause dual-slope in the Coffin-Manson plots at 0.11-0.14 wt. % N. Nitrogen content in 316LN SS is observed to play a dual role (detrimental and beneficial) in influencing fatigue life. An improvement in fatigue life up to 0.14 wt. % N is observed under those test conditions that promoted transgranular fatigue crack propagation (e.g.: strain rate of 3×10^{-3} s^{-1}). On the other hand, nitrogen addition induced significant reduction in fatigue life for test conditions causing intergranular cracking (LCF tests at strain rates < 3×10^{-3} s^{-1}).

Low cycle fatigue behaviour of modified 9Cr-1Mo steel at room temperature and 300°C

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Low cycle fatigue behaviour of the modified 9Cr-1Mo steel was studied in normalised and tempered condition at room temperature and 300 °C, at strain rate of 10^{-2} s^{-1} using different strain amplitudes of ±0.25%, ±0.31%, ±0.38% & ±0.5%. Cyclic Stress response at high strain amplitudes (±0.31% to ±0.5%) showed initial softening followed by hardening up to a few cycles and subsequent continuous softening till failure. On the other hand, at the lowest strain amplitude of ±0.25% there was stabilized stress response up to the initial 30 cycles, followed by mild hardening and subsequent continuous softening. The extent of softening increased with increase in strain amplitude. Coffin-Manson relationship between the plastic strain amplitude ($\Delta\varepsilon_p/2$) and number of reversals to failure ($2N_f$) was obeyed both at room temperature as well as 300 °C. However, fatigue life at 300 °C was found to be considerably lower as compared to that at room temperature over the entire range of strain amplitude. Dislocation density of the LCF tested specimens was determined using XRD and was found to be dependent on strain amplitude. The stable hysteresis loops displayed Masing behaviour at higher strain amplitudes (±0.38% to ±0.5%) but there was non Masing behaviour at lower strain amplitudes (±0.25% and ±0.31%). Inter striation spacing was found to increase with increase in strain amplitude. Inter striation spacing was observed to be higher at 300 °C than that at room temperature at identical strain amplitude. Dislocation substructure revealed formation of sub-grains which might have caused cyclic softening at all strain amplitude.

Correlation of micro-mechanisms involved during thermo-mechanical processing with the grain boundary structure of stainless steel 316L

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Stainless steel 316L is used in most of the nuclear reactors and steam generating plants as a structural material due to its excellent corrosion resistance and mechanical properties even at high temperatures. Further enhancement in the life span of these reactors will need improvement in functional and mechanical properties of its components. Such characteristic properties can be achieved by tailoring the microstructure through various available processing techniques. Grain boundary character distribution (GBCD) plays an important role in determining the characteristic properties needed. Current study involves non-iterative as well as iterative route of thermo-mechanical processing for getting the desirable GBCD. In this work we aim at correlating the mechanisms involved during each step of processing with the evolution in structure at the grain boundaries. In the non-iterative route, as received samples were given some pre-strain by the process of rolling at different temperatures. Further, pre-strained samples were annealed at high temperature for different interval of times to generate low energy structure either as a means of recovery or recrystallization. Similarly in iterative route, cycles of pre-strain and annealing at high temperature for very short times were repeated to incorporate high fraction of special boundaries. Microstructural characterization was done using optical microscopy and scanning electron microscopy (SEM). Microstructure hence obtained for various conditions was correlated with the processing parameters. Quantitative analysis involving twin density, grain size, misorientation and fraction of various special and high angle boundaries was done using electron backscattered diffraction (EBSD). Comparison and hence the suitability of single step and iterative processing was analyzed on the basis of mechanisms involved as well as their effectiveness for improving the GBCD.

Effect of interrupted annealing on recrystallisation of heavily cold-rolled austenitic stainless steel

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Austenitic stainless steel AISI 304L with excellent corrosion resistance and good heat resisting properties is widely used but because of its lower yield strength it cannot be used as a structural material. In recent years increasing emphasis is on the development of new processing routes for tailoring the mechanical properties of this material. Present work investigates the effect of interrupted annealing on Recrystallisation of heavily deformed commercial AISI 304L grade steel plates of dimensions 100mm x 30mm x 10mm solution treated at 1070°C for 1hr followed by water quenching. Multi-pass unidirectional cold rolling with uniform thickness reduction per pass was performed in two-high rolling mill to a total thickness reduction of about 90%. Cold rolled specimens of 1mm thickness were subjected to heat treatment between 775°C and 925°C in a muffle furnace. Standard metallographic techniques were employed for the preparation of specimens for optical microscopy. Microhardness measurements were made on cold rolled and after thermo-cycles by using 100gms load. Using JMAK equation process parameters for recrystallisation were optimized. Results showed retarded recrystallisation kinetics observed at 775°C even after annealing for 2mintues which was confirmed by microstructure also. However, an accelerated recrystallisation kinetics was observed at 900°C and above with an early onset of grain growth. It was found that in the isothermal annealing the grain size increases with time and dislocation density decreases with time. The grain size was larger than 500nm even at 1 min of annealing at 825°C with a large deformed area. However, in interrupted annealing at 925°C after 2 cycles of 30 s, largest grain size was about 500nm with an average grain size of 120nm and with a large fraction of unrecrystallised region in microstructure. It is felt that the strain left in the material after each thermal cycle was responsible for grain refinement.

Effect of strain rate on tensile flow behavior of interstitial free high strength steel

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Room temperature tensile tests were carried out on Interstitial Free High Strength Steel (IFHS) using strain rates ranging between 10^{-4} s^{-1} to 3 x 10^{-1} s^{-1}. The experimental result reveals that with increase in strain rate, yield strength and tensile strength increases, maintaining a power law relationship with the strain rates. Strain hardening of this steel occurs in two stages. This was established by carrying out Jaoul–Crussard (J-C) analysis. Stage-I hardening occurs by interaction of applied stress with the dislocations. During Stage-II hardening, dislocation cell structure begins to form. Well defined dislocation cell structures formed with increase in strain. With further progress of deformation, the cell size does not change. The

additional dislocations get packed in the cell walls. Thus degree of strain hardening decreases in Stage II. Strain hardening rate was observed to decreases with increase of strain rate. Strain rate sensitivity was found to decrease with increase in true strain. Fracture is the termination of the deformation process. Therefore fracture surface bears the signature of the deformation process that a material is subjected to. Ductile fracture occurs in three steps, e.g., initiation, growth and coalescence of voids. The deformation characteristics of a material dictate the nucleation of voids, their subsequent growth and the ultimate coalescence of the most mature voids at fracture. Deformation at various strain rates will not only affect the mechanical behavior of material but also influences the void nucleation and growth process. In the present study two-dimensional dimple geometry and the distribution of dimple features (i.e. diameter and density) were quantified from tensile fractographs and linked the same with variation of mechanical properties with strain rates.

Nanoindentation studies on Cu-Al-Mn shape memory alloys

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Nanoindentation is an instrumented indentation technique for determining the hardness and elastic modulus from indentation load-displacement data. Application of this method to SMAs is quite recent. This technique has been particularly used for determining strain recovery by superelastic effect. Evidence for forward and reverse stress-induced martensitic transformation in Ni-Ti SMAs has been obtained from load-displacement curves. These studies reveal that the percentage of superelastic recovery is dependent on the type of indenter used. This has been confirmed by calculating the percentage recovery using energy and depth recovery approaches. In the present investigation, the superelastic recovery of Cu-Al-Mn shape memory alloys has been studied using Hysitron TI 950 nanoindenter with a Berkovich tip.The alloys were prepared from pure Cu (99.9 wt. %), Al (99.9 wt. %) and Mn (99.8 wt. %) by ingot metallurgy. Titanium (0.5 wt. %) was used as grain-refining addition. The elements were melted in an air induction furnace under argon atmosphere. The as-cast ingot was homogenized at 900 ^{o}C for 1 h, to get rid of micro segregation, and subsequently quenched in water. The alloys were hot rolled at 900 ^{o}C to a thickness of ~1 mm. The samples were cut and then mirror polished. A number of indentations were then made on the samples with loads ranging from 50 µN to 3000 µN. A trapezoidal loading profile (10s loading, 5s holding and 10s unloading) was used. The load-displacement curves at different loads were plotted and the subsequent recovery ratios for different composition were obtained and compared. The recovery ratios were calculated based on energy approach.

Sliding wear behaviour of ultimet and stellite 6B cobalt based alloys

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Cobalt based alloys are well known to provide good wear resistance in unlubricated systems or elevated temperatures. Among them, Stellite alloys are widely used for different wear applications. Recently, Ultimet alloy has been introduced that offers good ductility, high temperature strength, and corrosion and wear resistance. Very little is known about dry sliding wear behaviour of this alloy. The present work investigates sliding wear behaviour of wrought Ultimet alloy at room and elevated temperatures and compares with the sliding wear behaviour of wrought alloy-Stellite 6B. The as-received materials were

characterised in detail to understand the microstructure and mechanical properties. The microstructure and phase analysis of the materials were done using optical microscopy, scanning electron microscopy and X-ray diffraction. Hardness, yield strength and strain hardening exponent were determined by hardness measurements and compression tests. Compression tests were done both at room and elevated temperatures. The sliding wear tests were performed in a pin-on-disc configuration in ambient atmosphere, at different normal loads of 30, 40, 50 and 60 N and sliding speed of 0.5 ms^{-1}. During the tests, coefficient of friction (COF), wear and pin temperatures were recorded. From compression tests, Stellite 6B alloy was found to have higher yield strength compared to the Ultimet alloy but the latter has a better strain hardening ability than the former both at room temperature as well as at 600 °C. When Ultimet alloy pin is slid against stainless steel disc, the pin wear was prominent in comparison to Stellite alloy pin which exhibited material transfer. However, the steady state COF for both the materials was quite high (0.4 - 0.6). A systematic analysis of the wear surface and the subsurface regions including wear debris analysis has been done to understand the wear behaviour both the alloys. Detailed results of the work will be presented.

Mechanical behavior of fine grained Nb-Ti microalloyed steel processed by controlled rolling and cooling

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A SAILMA grade low carbon (0.16%) Nb-Ti microalloyed steel has been investigated to improve mechanical properties by thermomechanical processing. The rectangular slab with a thickness of 20 mm was annealed at 1200°C for 1 h for its homogenization prior to rolling. Gleeble-3800 thermo-mechanical simulator was used to obtain dilatometric curves from which T_{nr}, 952°C, (γ-recrystallization stop temperature during deformation), Ar_3, 772°C, (temperature at which ferrite begins to form) and Ar_1, 580°C (temperature at which austenite completes its transformation into ferrite) were determined. Controlled rolling was performed using a two high rolling mill in 3 different regions i.e., in γ-recrystallization region at T_{nr}+ 50°C (during deformation), γ-nonrecrystallization region at T_{nr} -50°C, and dual phase region below Ar_3 (at 760°C). The rolling was carried out in multiple passes to obtain a constant reduction of 50% in cross-sectional area. Specimens were cooled down to room temperature subjected to 3 different cooling rates i.e., at normal air cool, force air cool and water quench. TEM analysis confirmed the formation of ultrafine precipitates by the scanning thermomechanical treatment. Electron back scattered diffraction study was performed to observe morphological changes of the microstructure after deformation, and revealed the formation of subgrain and misorientation angle of grains. It was noticed that the yield strength (YS) of the forced air-cooled and quenched specimens is significantly improved without much interfering the ductility, especially when rolled in $(\alpha + \gamma)$ phase region. The best combination of YS (825 MPa) and ductility (24%) was obtained for the quenched steels after rolling in $(\alpha + \gamma)$ phase region. The improvement in the mechanical properties with high ductility can be enlightened with the special features of microstructure such as dual size grain distribution, formation of subgrains and homogeneous distribution of ultrafine precipitates within the ferrites and pearlites.

Studying the effect of carbon on mechanical property and microstructure of TMT rebars

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Thermo-mechanical treated (TMT) rebar is suitable material for reinforcing concrete structures on accounts of similarity in thermal expansion, ability to bond well with concrete and, above all the ability to shoulder most of the tensile stress acting on the structure and also steel manufacturing industry has successfully developed a corrosion- resistant variety of rebar for the construction industry. The objective of our work is to study the effect of carbon on the microstructure & consequently on the mechanical properties of TMT rebar. We have considered two different variants of TMT, one having low carbon content(C=0.25) & the other one with high carbon content(C=0.29).We have observed the changes in the variation of the thickness, hardness & the mechanical properties of the core, transition zone & rim due to the difference in carbon content, thus we have tried to correlate property of TMT with microstructure. It is seen, that C has profound influence on all the microstructural parameters within the TMT. The following inferences can be drawn. 1. The hardness of rim, transition & core has increased in the high carbon variant. 2. Increase in core hardness is more prominent due to increase in pearlite fraction. 3. Exceptional UTS/YS ratio is observed in the higher carbon variant, which implies that YS is more dependent on rim thickness.

Session 2: Nov 12, 2014: [16.00 - 18.00]

Role of mechanical property anisotropy in the selection of materials for high strain rate applications

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The properties of materials under high strain rate applications significantly differ from those determined under static or steady state loading conditions. Hopkinson and Charpy were some of the earliest researchers to recognize this phenomenon which led to the development of machines and test procedures known presently with their names. The deformation phenomena and microstructural characteristics significantly differ in materials tested under high strain rate conditions. Therefore, understanding the high strain rate properties of materials is the key to their applications such as separation systems of launch vehicles or impact of debri on launch vehicle and satellite structures. Precipitation hardenable stainless steels are widely used in aerospace applications due to their excellent strength and corrosion resistance. In view of these advantages, Fe-17Cr-4Ni steel (17-4PH) is used for stage separation of satellite launch vehicles. This steel, in view of its composition, has delta ferrite in a martensitic matrix in the hardened and tempered condition. In wrought products delta ferrite gets oriented in the direction of thermo-mechanical processing. This paper highlights the effect of delta ferrite orientation on the impact properties of 17-4PH steel in various heat treatment conditions. It was found that the transverse impact properties are severely affected by the presence of highly oriented delta ferrite for all heat treatment conditions studied.

Deformation behavior of dual phase steels at high strain rates

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Dual phase (DP) steels are extensively used in automobiles due to their good combination of strength and ductility, and high strength to weight ratio. Quasi-static deformation behavior of DP steels has been fairly studied, wherein, for example in ferrite-martensite DP steels, soft ferrite phase contributes for ductility and martensite gives the strength. Depending upon the nature, amount and distribution of martensite, the deformation is either due to ferrite/martensite interface decohesion or cracking within martensite colonies or mixture of both. However, considering the automobile crash events, it is necessary to evaluate the deformation behavior of these alloys at high strain rate regimes, analogous to automotive crash events. Such high rate deformation on DP steels has not been clearly understood. This work presents the high rate deformation behavior of two varieties of DP steels, namely, DP600 and DP800. The steels possessed varying volume fractions of martensite (10 % for DP600 and 30 % for DP800). The steels have been deformed in tensile mode at different strain rates in the range of 0.001 to 800 s^{-1}. The variation of strength of these alloys with strain rate, and those of strain hardening behavior and strain rate sensitivity has been examined. The strength increased with strain rate, whereas the strain hardening rate decreased as the strain was increased. The strain rate sensitivity of both the steels was high in the high strain rate regime to that of quasi-static response. The fractographic examination revealed a change in void size distribution when the strain rate increases. Moreover, the deformation behavior of DP600 was mainly due to ferrite/martensite interface decohesion at all strain rates, whereas it changed from interface decohesion with martensite cracking at slow strain rates to only interface cracking at high strain rates.

Flow localization in AA5052 after severe plastic deformation at low and high strain rates using hat-shaped samples

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It is well established that ultrafine grained (ufg) Al alloys produced using severe plastic deformation technique have higher strength at ambient temperature as compared to the coarse grained alloys. However, their tendency for strain localization has not been well explored. In this study, AA5052 was subjected to severe plastic deformation using the equal channel angular pressing (ECAP) technique. The microstructure, quantified using electron backscattered diffraction, showed that ECAP refined the grain size, increased the fraction of high angle boundaries (from 0.46 to 0.75) and increased total boundary area per unit volume of AA5052. ECAP increased the flow stress from about 325 to 425 MPa, strain rate sensitivity from 0.005 to 0.012 and reduced strain hardening exponent from 0.34 to 0.25. Shear localization in the ECAP as well as unpressed AA5052 was studied using hat-shaped samples deformed at both quasi-static strain rates in a screw-driven machine and dynamic strain rates in a split-Hopkinson bar. The shear band formation in the hat-shaped sample was narrow and well defined for the ECAP condition sample as compared to the unpressed condition sample, where the shear band was broad and diffuse.

This implied an increased propensity for shear localization as compared to the unpressed condition. The microstructure within the shear band of the ECAP condition sample showed that the boundary area per unit volume decreased as compared to that away from the shear band.

Effect of laves phases on the creep rupture properties of advanced 9Cr steel (P92)

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Stress rupture tests of normalized and tempered P92 (9Cr-0.5Mo-1.8W) steel were carried in the range of 135 to 215 MPa at 650 °C. Resulting rupture times varied from 100 to 3000 h, and creep rate by 1 order of magnitude. Effect of tempering temperature in the range of 740 to 780 °C on the creep rupture life was investigated. In the high stress regime, lower tempering temperature resulted in highest rupture time due to the initial high dislocation density and fine laths. However, at lower stresses, highest rupture time was observed for highest tempering temperature. Formation of Laves phase (Fe_2Mo, Fe_2W), next to $M_{23}C_6$ carbides was responsible for increase in rupture time. Back scattered electron imaging (BSE) in scanning electron microscopy (SEM) was used to identify Laves phases, and study their distribution.

Assessment of the effect of Cu-addition on the fracture toughness of as-cast ductile irons by multiple approaches

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Ductile cast iron (DCI) finds wide applications for the production of gears, crank shafts, engine blocks, sewer pipes etc., but it is currently being considered as a candidate material for nuclear cask for the storage and shipping of radioactive materials. For suitable structural integrity as a cask material, DCI should possess high toughness in as-cast condition. This investigation aims to illustrate the effect of varying Cu on the structure-property relations of some as-cast DCIs with special emphasis on the assessment of their fracture toughness. Three DCIs have been prepared by induction melting in batches of 300 kg with varying amounts of Cu (0.07, 0.11 and 0.16 wt.%) but with ~1 wt.% Ni. This is followed by examinations of their microstructural features (amount of phases, morphology, size and count of graphite nodules), determination of hardness and tensile strength values using standard methods. Quasi-static fracture toughness of the as-cast DCIs have been determined using J-integral test using half C(T) specimens (K_{JIc}), ball indentation test (K_{ISF}), and dynamic fracture toughness (K_{Id}) tests on pre-cracked Charpy specimens, followed by fractographic examinations of the broken specimens. Following inferences can be drawn: (i) the amount of pearlite and nodule count increase with increasing amount of Cu in the DCIs resulting in enhanced hardness and strength with associated reduction in fracture toughness; these modifications has been attributed to the additions of Cu acting as a pearlite promoter and Ni as a graphitizer, (ii) both $K_{JIc}$and K_{ISF}values exhibit decreasing trend with increasing amount of pearlite in the

DCIs unlike K_{Id} and it is noted that $K_{JIc} > K_{ISF} > K_{Id}$, which has been explained, and (iii) fractographic examinations reveal that crack initiation occurs due to decohesion of the graphite nodules from the matrix, while crack propagation takes place primarily through the matrix phase constituents.

The microstructure and mechanical properties of thin wall austempered ductile iron

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In the present work, ductile iron plate castings with different section thicknesses of 2 and 3 mm were cast with an appropriate casting design to assure good mold filling, shrinkage free without chill. Short term austempering treatment process was used to obtain thin wall ADI castings. The samples of 2 and 3 mm thickness were austenitized at 910 °C for, 30 minutes followed by holding at 350°C and 400°C for 5 and 10 min for each temperature. The mechanical properties like hardness, yield strength and ultimate tensile strength decreased, if the austempering temperature was increased with austenpering time. Hardness and ultimate tensile strength are higher as in case of ausferritic matrix than the ferritic matrix structure.

Wear behaviour of titanium alloys used for automobile and structural applications

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Titanium alloys are noted for the outstanding strength to weight ratio and corrosion resistance. Because of these exceptional properties, titanium alloys have become major structural materials where weight reduction is a primary concern such as automobiles, sports, airborne applications etc. However, titanium alloys suffer from poor wear resistance. Several surface modifications are developed for improving the wear resistance of titanium alloys. The objective of the present study is to evaluate wear behaviour titanium based alloys, subjected to 'thermal oxidation'. In this work, the sliding wear behaviour of thermal oxidation (TO) treated and untreated Ti-6Al-4V alloys have been investigated using pin-on-disc wear testing machine. The results indicate that TO treatment can significantly enhance the wear resistance of Ti-6Al-4V alloy. It was found that the significantly reduced tendency to adhesive wear has contributed to the enhanced wear resistance of the TO treated Ti-6Al-4V alloy.

Microstructure and creep behaviour of two different Mg-Al-Ca alloys

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Two creep resistant magnesium alloys MRI153M and MRI230D, developed for powertrain applications, were prepared by high pressure die casting. These alloys were tested for their creep behaviour using continuous creep test at different temperatures and stress levels. For comparison the same creep tests were also carried out on the conventional magnesium alloy AZ91D. A detailed microstructural characterization of the alloys before and after creep tests were carried out. The as-cast microstructures of AZ91D alloy consists of primary Mg (α-Mg) and $Mg_{17}Al_{12}$ phase; the MRI153M alloy consists of α-Mg, $Mg_{17}Al_{12}$ and $(Mg,Al)_2Ca$ (C36) phases; the MRI230D alloy consists of $(Mg,Al)_2Ca$ (C36) phase only. The best and worst creep resistance were exhibited by the MRI230D and AZ91D alloys, respectively. It was observed that the additional precipitation of $Mg_{17}Al_{12}$ phase during creep test increased the creep rate in the AZ91D and MRI153M alloys, whereas the additional precipitation of $(Mg,Al)_2Ca$ (C36) phase during creep test decreased creep rate in the MRI230D alloy.

Session 3: Nov 13, 2014 : [09.00 – 11.00]

Experimental evaluation of fracture toughness in transition temperature regime using PCVN Charpy specimen of 20MnMoNi55 RPV steel

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Ferritic steel 20MnMoNi55 is used as reactor pressure vessel (RPV) steel in the nuclear reactor. The RPV's are subjected to irradiation, due to this material degrades resulting in upward shift of ductile to brittle temperature (DBTT). Hence it is very important to evaluate the transition temperature of the steel and fracture behavior in the transition regime of the RPV. Fracture behavior in transition regime suffers with huge scatter. This stochastic behavior is addressed utilizing weakest link theory by master curve approach. The ASTM E1921 is used to determine the reference transition temperature T_0 using the master curve technology. In this study, the fracture behavior of 20MnMoNi55 RPV is investigated on pre-crack V-notch (PCVN) Charpy geometry of 10 × 10 × 55 and 5 × 5 × 27 mm^3 in three point bend loading. The reference transition temperature obtained using conventional master curve is liable to constrain effects which are predominant in smaller specimens. The constraint loss correction is imposed on the conventional master curve results using T-stress model proposed by Wallin. The difference between reference temperature T_0 obtained from the conventional master curve and the corrected master curve is found to be justifiable.

Microstructure, mechanical and impact wear resistance of TiC reinforced austenitic manganese steel

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The aim of the present research work is to determine the mechanical and wear resistance properties of TiC reinforced austenitic manganese steel composite (AMSC). Austenitic manganese steel (AMS) is generally used in the as-solutionised condition which consists of a single phase austenite. AMS has high work hardening capacity at high impact loads but at low impact loads it shows less work hardeneing. Reinforcement of the TiC particles in the AMS and secondary processing of the AMSC may help to improve the work hardening capacity of the AMS at low impact loads. Hardness, impact toughness, elastic modulus

and impact wear resistance of the AMSC are compared with the austenitic manganese steel (AMS) in the as-solutionised and secondary processed conditions. It is observed that, incorporation of TiC particles and secondary processing help to improve the hardness, elastic modulus and impact wear resistance but deteriorates the impact toughness.

Mechanical and microstructural studies of zircaloy-2 processed by room temperature wire rolling

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In the present work, microstructural and mechanical behavior of room temperature wire rolling of Zircaloy-2 has been investigated. Zircaloy-2 was subjected to solutionising treatment at 1073 K followed by quenching in mercury. The quenched alloy of 4x4 mm cross section was wire rolled in rolling mill at room temperature to different reduction (3x3 mm, 2x2 mm, 1x1 mm) and further characterization was performed. SEM/EBSD and TEM analysis were carried out to understand the mechanisms of formation of ultrafine grains in the alloys. The mechanical strength of 1 x 1 mm wire rolled deformed zircaloy-2 was found to be increasing due to increase in dislocation density. Various extension twinning has been seen from EBSD microstructure and pole figure images of deformed alloy. Dislocation density and stored energy is calculated with the help of EBSD results. Bulk ultrafine grained zircaloy -2 has been produced after wire rolling of zircaloy-2. It has been observed that ultrafine grained zircaloy-2 exhibits improved tensile properties as compared to coarse grained zircaloy-2.

Effect of microstructure on deformation behavior of Ti6Al4V at low and warm temperatures

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Ti6Al4V is the most extensively used titanium alloy in aerospace industry. Hence there has been lot of studies carried out to understand the deformation mechanism of Ti6Al4V alloy at high temperatures. But in order to cut down the processing cost of Titanium alloys, there is a need to find out the processing window at low and warm temperature region. At low temperatures, there is a competition between the slip and twinning activity in titanium alloy but the dominant deformation activity (either slip or twin) is a function of stress state, rate of loading and the microstructure of the materials. The aim of the present study is to understand the effect of microstructure on deformation behavior of Ti6Al4V at low and warm temperature conditions. In order to achieve the aim, various microstructures are developed via heat treatment and deformation behavior is studied as a function of deformation temperature and strain rate. The correlation between the microstructure and deformation parameters is established with the help of

characterization tools like EBSD and XRD. It is anticipated that the twins produced during the deformation increases the plasticity of Ti6Al4V.

Mechanical and microstructural behaviour of IMI 834 titanium alloy severely deformed by high pressure torsion

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IMI 834 titanium alloy is a near alpha (HCP) titanium alloy used for high temperature applications with the operating temperature up to 600°C. This alloy is extensively used in gas turbine engines such as high pressure compressor and low compressor discs, having desired better dwell fatigue life and creep properties. These desired properties demands ultrafine grain microstructure with random crystallographic orientations. Alpha (HCP) titanium phase, being limited by lesser number of possible crystallographic slip systems, upon conventional deformation generates strong texture. On the other hand severe plastic deformation (SPD) imparts large plastic strain without change of sample size and can generate ultra-fine grained microstructure, in turn imparting higher material strength and may generate random texture. The present work deals with the severe plastic deformation (high pressure torsion) of IMI 834 titanium alloy at various plastic strains (several rotations) and then study of its mechanical-microstructure evolution. The hardness profile of the SPD samples will demonstrate its mechanical properties and the orientation maps with crystallographic texture and grain boundary details will provide its microstructure evolution.

Modified Hall Petch relationship of mechanical properties with structural parameters for ADI

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A combination of good strength, toughness, wear resistance and fatigue strength made Austempered Ductile Iron (ADI) suitable for many applications in fields of automobile, farm digging equipment, earth moving machines and other engineering equipments. It is prepared in two steps of heat treatment namely Austenitization at 900°C and Austempering in the range of 450 ^{o}C - 250 ^{o}C. The time and temperature of the heat treatment process decides the grades of ADI and is also responsible for the peculiar microstructure of ADI called Ausferrite, a combination of bainitic ferrite and high carbon austenite. The two microstructural parameters which affect the properties of a given grade of ADI are ferritic needle size (d_{α}) and the volume fraction of retained austenite (X_{γ}). The modified Hall-Petch Relation is used to correlate mechanical properties like Hardness, tensile strength, UTS and % elongation with these two parameters using Multi Linear Regression analyses. The coefficients in the equations obtained showed that the particle size is relatively more important for hardness. In fact, it establishes that increases in X_{γ} will serve to slightly decrease the Hv_{10}. 0.2% proof strength and UTS, is quite similar to the HV_{10} results. These findings are in good agreement with those of previous investigations for ADI developed from pure ductile iron and from those with Mn additions. On the other hand, %El, showed relative values of the coefficients describing the effect of X_{γ} and d_{α} are much closer to one another, and that the effect of the austenite volume fraction is more important than particle size of the ferrite. This is intuitively correct because

austenite is nominally more ductile than ferrite. On the other hand the decreasing % elongation with decreasing particle size is a natural consequence of increasing strength observed in all engineering alloys.

Effect of aging on the mechanical behavior of beta-titanium alloy (Ti-10V-2Fe-3Al)

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Titanium alloys are suitable candidate materials for different aerospace applications due to their high strength to weight ratio and excellent corrosion resistance. Ti-10V-2Fe-3Al is a metastable beta high strength alloy having the best combination of strength and toughness of the commercially available Titanium alloys. It is capable of attaining optimized strength and fracture toughness depending on the selection of heat treatments (solutionising and aging). One solutionising temperature, i.e., 780°C and three aging temperatures were chosen (505°C, 515°C and 525°C) for the present work. A systematic microstructural investigation of aged samples was carried out using optical microscope which revealed the microstructure having the β grains with primary α at the grain boundaries and fine secondary α within β grains. Tensile test were carried out on the samples obtained from materials subjected to all the heat treatment conditions with a strain rate of 0.03mm/min. Sample solutionised at 780°C followed by aging at 505°C exhibited higher yield strength and ultimate tensile strength compared to the other heat treatment conditions whereas the sample in the only solutionised condition at 780°C showed highest elongation and reduction in area. Micro hardness was found to be highest in the specimen, which underwent the heat treatment of solutioninsing followed by aging at 505°C.

Achieving enhanced strength in Cu-3Ag-0.5Zr alloy by cryo rolling and aging

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In the present investigation, the mechanical properties and microstructure of Cu-3Ag-0.5Zr alloy were studied as a function of cryo rolling (CR) and aging using hardness, tensile testing, and transmission electron microscopy. Room temperature tensile strength increased significantly with the degree of cryo rolling with maximum strength at 80 % cryo rolling. Aging of 80 % CR sample showed the maximum hardness at 240°C and decreased thereafter with minimum at 500°C for holding time of 1 h. 80 % CR sample aged at 240°C for 1 h showed ultimate tensile strength and yield strength of 659 MPa and 643 MPa, respectively with a ductility of 3.1 %. Increase in aging temperature (320-400°C) has led to improvement in ductility without any significant loss in strength. Optimum combination of strength and ductility: ultimate tensile strength (647 MPa), yield strength (614 MPa) and ductility (8%). The strengthening in cryo rolled and annealed sample is attributed to dislocation strengthening, grain boundary strengthening and precipitation hardening.

Study on flow behavior of plain carbon steel during hot rolling through thermo-mechanical simulation

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Uniform mechanical properties and homogenous microstructure in hot rolled steel strips are critical requirements for various complex forming operations involved in the manufacturing of automotive components. During hot rolling of plain carbon steel the microstructure evolution is largely dependent on the flow behavior of the steel under deformation which in turn is dependent on the processing parameters such as temperature and rolling rate. Hot strip rolling in a typical tandem mill is carried out at temperatures close to or above Ac_3 temperature. The deformation rates for thin strips are generally very high with strain rate in the order of 100 s^{-1}. In the present study single-hit compression tests were carried out on plain carbon steel, using Gleebe-3800 thermo-mechanical simulator, at various temperatures in the range of 800-900°C and strain rate ranging from 0.01-100 s^{-1} representing deformation during hot strip rolling. The results show an increasing trend of flow softening as the temperature of deformation decreases closer to Ac_3 temperature. At higher temperatures the flow curve shows oscillations typical of either dynamic recrystallization or flow instability. The oscillations increase as the strain rate is increased irrespective of the temperature. At higher strain rate the material exhibits negative strain rate sensitivity at all strains. Below Ac_3 temperature, where α and γ two phase region exists, negative strain rate sensitivity is observed at all strains. The microstructure of the deformed samples is correlated with the flow behavior of the material. Based on the homogeneity of the microstructure and grain size an optimum temperature regime for hot rolling of present material has been established.

Session 4: Nov 13,2014: [13:30-15:30]

Nanomechanical behavior of cryogenically rolled martensitic TiNi alloy

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Shape recovery of nearly equiatomic TiNi alloys has been found to be sensitive to the thermo-mechanical treatments. In martensitic TiNi alloys, the shape recovery is essentially the result of martensite-austenite transformation during heating. The amount of recoverable strain is the convoluted measure of the deformation accommodation due to martensite reorientation, variants coalescence and detwinning process. The mechanisms related to reorientation and coalescence of martensite variants are manifested by the overall microstructural constitutions. On the other hand, detwinning process is determined by the intrinsic mechanical response of the individual martensite variant. Therefore, mechanical behavior of the individual martensite variant is an important component of the observed shape memory effect which is sensitive to the thermo-mechanical treatment. Therefore, designing the process schedule for the alloy warrants understanding of the influence of thermo-mechanical treatments on the mechanical and shape recovery behavior of the single martensite variant. In the present study, nanoindentation technique has

been extensively employed to examine the mechanical response of single martensite variant formed in the microstructure of cryogenically rolled (30% reduction) martensitic TiNi alloy treated under appropriate ageing schedules. Influence of ageing on the mechanical behavior of cryogenically rolled martensitic TiNi alloy has also been studied at microstructural scale. The nanoindentation of martensite variants of the alloy has been found to be maximum at the peak aged condition i.e., 60 min at 400°C, which implies the influence of precipitate phase (Ti2Ni) on the mechanical properties of martensite. A hardening effect of the martensite variants has also been measured by conducting cyclic indentation with incremental loads. The improvement in the shape recovery response of the martensite variants has been attributed to the delay in the onset of plasticity. An attempt has also been made to correlate the results of the nanoindentation to the corresponding bulk recovery behavior.

Room temperature plasticity in nanocrystalline nickel of different grain size

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Nanocrystalline (NC) nickel of grain size 25 and 60 nm were produced by electrodeposition technique. The 60 nm samples were heat treated at 300°C and 370°C for 30 mins. The thermal grain growth resulted in the average grain size of 200 and 500 nm respectively. Nanoindentation tests were performed on nc-Ni samples of different grain size (25 nm, 60 nm, 200 nm and 500 nm) varying the loading rate from 4.8 to 240 mN/min at a constant maximum load of 8 mN. The hardness (H) was found to increase with decrease in grain size from a value of 2.53 GPa for 500 nm to 4.5 GPa for 25 nm grain size. Strain rate sensitivity was computed from the variation of H with strain rate which showed an increasing trend with decrease in grain size. The experimental findings suggested dislocation-grain boundary interaction to be the rate controlling mechanism. Creep properties of the nc material was studied by nanoindentation by holding the sample at the maximum load for an extended dwell time of 5-300 s while hardness and penetration depth were monitored as a function of the hold time. Different creep parameters like creep strain rate and stress exponent (n) were estimated. While creep strain rate did not vary much with grain size, stress exponent was found to increase with increase in grain size. 'n' exhibited very high value and no conventional creep mechanism could be associated with it. Finally, cyclic nanoindentation tests were performed on the nc Ni samples and hysteresis loops were formed. The loop area was found to increase with increasing grain size which might be attributed to the easy reverse plasticity in materials with larger grains.

Effect of thermo mechanical treatment on production of ultrafine grained 6082 Al alloy through cryorolling

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In the present work, 6082 Al after homogenization at 550˚C for 24 hrs was subjected to thermo mechanical processing (TMP) using Gleeble simulator followed by cryorolling up to 90% deformation. The applied TMP include isothermal compression at 125, 150, 175 and 200˚C under various strain rates of 1, 10 and 100 S-1.The maximum ductility and strength of the sample were achieved at 200˚C under strain rate of 1 S-1 and 100 S-1 respectively. To investigate the effect of pre strain rate on production of UFG 6082 Al

alloy, the material compressed at 200˚C was subjected to cryorolling up to 90 % thickness reduction. The sample compressed at 200˚C with the strain rate of 100 s^{-1} upon cryorolling gives maximum strength and ductility as compared to other samples. Detailed TEM and EBSD analyses of cryrolled samples were carried to substantiate the improvement in tensile properties observed in the present work.

Tribological behaviour of friction stir processed aluminium alloy-alumina surface composites

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Friction stir processing (FSP) is relatively a new technique for fabrication of surface composites. In this study, 5083 aluminium alloy – Al_2O_3 surface composites were prepared via FSP at low heat input (710 rpm rotational speed and 100 mm.min-1 traverse speed) and high heat input (1400 rpm rotational speed and 40 mm.min-1 traverse speed) conditions. The tribological behaviour of aluminium alloy, FSPed aluminium alloy and FSPed aluminium alloy-alumina composites against bearing grade steel ball in unlubricated conditions was studied at 5N, 10N and 20 N load. While no influence was found on the frictional behaviour, the wear resistance of FSPed composites was superior to FSPed alloys in any heat input condition. FSPed composites fabricated at high heat input condition showed improved wear resistance as compared to low heat input condition. The wear rate decreased with sliding load. The worn surface analysis revealed that plowing and delamination of deformed layer was dominant at lower load of 5 N, while adhesion and delamination were dominant at 10 or 20 N load.

Strain hardening behaviour of pearlitic steel wires

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Present work applies of Ludwik, Swift and Hollomon analysis for determining strain hardening behavior of drawn wires of pearlitic steel, having high proportional limit and work hardening rate, immediately without exhibiting discontinuous yielding in stress-strain curve. In particular these analyses have been applied to pearlitic steel wire in patented/unpatented conditions to observe effect of patenting on strain-hardening behavior. The obtained results have been analysed critically on the basis of logarithmic plot of true stress-true strain data. Additionally an attempt has been made to access residual drawability using yield and tensile strength data at different stages of reduction. Comparatively finer pearlitic grains are formed as a result of patenting process, as compared to controlled cooling used in wire production. The transverse sections for both unpatented and patented wire rods, after drawing, exhibited a wavy or curly pearlitic microstructure, whereas their longitudinal sections exhibited deformed pearlitic grains aligned in the direction parallel to wire axis. The differential C-J analysis assuming the Power Ludwik relation is less sensitive to the changes in deformed pearlitic grains at different stages of drawing. This analysis is also insensitive to changes in pearlitic structure of patented wire after drawing. In contrast, the modified C-J analysis based on Swift equation appears to be most suitable for describing strain-hardening behavior for both types of wires as well as drawn wires at different stages of drawing. This analysis reveals two stages of deformation, associated with deformation of ferrite and then ferrite and cementite respectively. On the other hand, the Hollomon curves deviated from linearity over all the uniform strain range regardless of

unstrained as well as pre-strained wires. The yield strengths of the patented drawn wires decreased in the later stages of drawing, possibly, due to increase in temperature in deformation zone indicating inadequate cooling.

Discrete dislocation dynamics simulations of dislocation interactions with low angle tilt boundaries in FCC crystals

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The interaction of dislocations with grain boundaries influence strain hardening in metals, particularly in nanocrystalline materials where the mean free path of dislocations is of the same order as the grain size. An intricate examination of the interaction of dislocations with boundaries could yield valuable information on the hardening behavior and would help in grain boundary engineering applications. The present work studied the interaction of dislocations with low angle tilt boundaries (LATB) in fcc metals using discrete dislocation dynamics. A symmetrical LATB was constructed using glissile edge dislocations. Free glissile dislocations on different slip systems were allowed to react with the boundary to produce an array of binary junctions of the type collinear, Lomer, and two variants of the glissile junction glissile-a and glissile-b. The simulations were run at constant shear strain rate as computed on the slip system of the incoming dislocation. The transmission resistance was taken as the minimum resolved shear stress required to break the array of junctions. The transmission resistance was higher for collinear and glissile-a junctions, which had a 90o angle between glide plane of the incoming dislocation and the LATB plane, as compared to Lomer and glissile-b for which the angle was 35:2o. With increasing dislocation density within the boundary the transmission resistance increased for all interactions, with the increase being higher for collinear and glissile-a, than for Lomer and glissile-b. The transmission resistance was found to be inversely related to the unreacted arm length of the incoming dislocation. This work assumes importance in materials where geometrically necessary dislocations exist and are arranged as dislocation walls.

Microstructural analysis and indentation behaviour of coconut shell (cocos nucifera)

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Coconut is a member of the monocotyledonous family Arecaceae (Palmaceae), subfamily Cocoideae and the monospecific genus Cocos. Coconut is considered as an important fruit crop in tropical countries. Coconut is the most extensively grown and used nut in the world, playing a significant role in the economic, cultural, and social life of over 80 tropical countries. Currently, coconut is mainly an oil crop; rich in lauric acid, with a variety of other uses in addition to commercial oil production. Dehusked coconut shells are agricultural byproducts that are often used as particle reinforcements in various polymer composites. This study aims to understand the microstructure and indentation behaviour of coconut shells. XRD results shows peaks that correspond to the presence of crystalline cellulose. Results obtained from FTIR indicate that the shell is made up of cellulose, hemicellulose and lignin. The density of the shell was measured using Archimedes principle and was calculated to be 1.21 g/cc. The Young's modulus and shear modulus as determined by ultrasonic contact testing were 1.042GPa and 0.383GPa. SEM

micrographs reveal the presence of fibres and certain other unique features throughout the sample. Fracture surface clearly shows the presence of very fine nano-scale porosity. Macro-indentation was done at different loads (10, 20 and 30 kg) and the hardness was found to be- 10MPa. SEM micrographs reveal the presence of cracks within the indent.

Application of numerically corrected master curve for determination of reference temperature of in-RAFMs

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In-RAFM's is a prime candidate for blanket material application in fusion reactor. Irradiation of high velocity neutron in fusion reactor is responsible for upward shift of transition temperature which makes the investigation of fracture behavior of In-RAFMS in ductile brittle transition (DBT) region of utmost priority. In this work the fracture toughness of In-RAFMs is evaluated in DBT regime and reference transition temperature is estimated using both single and multi-temperature Master Curve (MC) approach. The conventional MC fails to estimate reference transition temperature due to its small scale yielding (SSY) assumption in small size samples. A numerical correction is proposed to rectify this lack of conventional MC and the same has been used to evaluate the reference temperature of In-RAFMs. The numerical correction is based on critical stress–critical volume approach. Utilizing the fact that, for a material two samples having same amount of stressed volume ahead of crack tip represents same cleavage failure probability, numerical transformation of experimental non-SSY condition data to equivalent SSY condition was done using finite element analysis. The proposed numerical correction is found to be successful in estimating justifiable reference transition temperature.

Tensile properties of zircaloy fuel cladding tube after high temperature steam oxidation and quenching

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The present study estimates the tensile properties of pressurised heavy water reactor (PHWR) fuel cladding tube made of Zircaloy-4 after a postulated loss-of-coolant-accident (LOCA) simulation. During postulated LOCA condition the fuel cladding is liable to be severely oxidized in high temperature steam environment resulting in significant degradation in ductility causing fragmentation due to the action of re-wetting by emergency-core-cooling-system (ECCS) water. The abrupt change in heat transfer conditions during ECCS cooling induces large stress due to thermal shock in the cladding and its eventual failure. For LOCA simulation, the isothermal steam oxidation experiments were conducted on 3mm wide rings of cladding tube (outer dia. 15.2 mm and wall thickness 0.4 mm) at temperatures ranging from 850 - 1200°C at the interval of 50°C with soaking at each of the temperatures for 1, 2, 5, 10 and 15 min followed by quenched in water. The specimens that survived the water quench test were subjected to oxide layer thickness measurements and ring tension test (RTT). The oxide layer thickness were measured under optical microscope and found to vary from 9.7 µm at 850°C for 1 min to 102 µm at 1150°C for 5 min of

soaking periods. All the RTTs were performed at ambient temperature to estimate the transverse tensile properties of the cladding. The estimated ductility values showed a decreasing trend with increasing oxidation temperature whereas the strength values had increased to some extent. The scanning electron microscopy (SEM) was carried out on fractured surfaces of tested specimens that depict the varying mode of failure from ductile (cup & cone, 45o shear lip and dimples) to brittle (flat faces) fracture as the oxidation temperature increases. The hydrogen analysis of tested specimens were carried out using Inert Gas Fusion (IGF) technique and pick up was determined to be in the range of 5-30ppm.

Effect of microstructure on fatigue crack growth behaviour of Ti-6Al-2Zr-1Mo-1V

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Ti-6Al-2Zr-1Mo-1V is used for manufacturing load carrying bulkframe of supersonic fighter aircrafts. The bulkframe, which is a welded assembly of forged coupons, experiences high fluctuating loads during aircraft operations, and thus makes fatigue studies on the material important. The fatigue crack growth behaviour of this alloy was studied in three different microstructural conditions: lamellar, bimodal and acicular. Both increasing and decreasing stress intensity factor range fatigue crack growth tests were performed at ambient temperature and at a loading ratio of 0.3 using compact tension samples. Fatigue crack growth rates were found to be lower for lamellar and acicular microstructures as compared to bimodal microstructure. EBSD maps in conjunction with fractographs and surface crack profiles were studied to understand the crack growth mechanism. Fractographs suggested that striated fatigue crack growth occurred in lamellar microstructure, facetted crack growth in case of primary alpha grains of bimodal microstructure, and intergranular fracture of acicular alpha in case of acicular microstructure. The boundaries of lamellar colonies in lamellar microstructure and acicular grains in acicular microstructures caused significant crack deflections leading to improved fatigue resistance. In case of bimodal microstructure, the advancing crack front was insensitive to microstructural features. The less deflections of the crack front and facetted crack growth in case of bimodal structure were found to be the primary reasons for its lower fatigue crack growth resistance. The results agreed with previous work on lamellar structure of VT-20 alloy.

Influence of grain size and twin density on LCF behaviour of annealed OFHC copper

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Effect of grain size and twin density in annealed OFHC copper on low cycle fatigue behavior has been investigated by testing both under completely reversed true strain-control and true stress-control mode. In case of true-strain control mode tests have been done at six different strain amplitudes (0.25 to 0.75 pct.). True stress-control mode tests have been done at stress amplitudes which correspond to the stable

stress state of strain controlled cyclic tests. In this case stress amplitude varied from 130 to 170 MPa. It is found that during stress-control cyclic both cyclic fatigue damage and accumulation of tensile plastic strain occur. Further, it is found that for strain-control tests of coarse grained annealed OFHC copper the hardening during cyclic straining is more as compared to fine grained copper. This happens because of more twin boundaries in coarse grained copper. But, the fatigue life of coarse grained copper is less as compared to fined grained copper. However, Coffin-Manson relationship is perfectly followed in both cases. Comparison of cyclic fatigue resistance has also been done using the variations of hysteresis loop area and plastic strain range with number of cycles. It is found that test control mode highly influences the fatigue damage and more number of cycles is required for fatigue damage during true stress-control cyclic loading as compared to the true strain-control mode of loading. Fracture surfaces of low cycle fatigued specimens observed under SEM reveal that failure occurs through formation of fatigue striations and the striation spacing has been found to increase with increase of strain/stress amplitude. Fine microvoids are also observed on fracture surfaces.

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Serrated flow behaviour in tungsten heavy alloy

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Flow behaviour of a tungsten heavy alloy namely 90.5W-7.1Ni-1.65Fe-0.5Co-0.25Mo alloy has been investigated in a temperature range of 300-973 K and strain rate range of 10-5- 10-1 s-1. A broad band enclosing the flow stress values at different strain rates as a function of temperature shows an overall trend with two regimes. In the regime I at low temperatures, the flow stress decreases rapidly and in the regime II at intermediate temperatures (ranging from 673-973 K), it reaches a plateau. The width of the band, which is an indicator of strain rate sensitivity, decreases at intermediate temperatures. This is further corroborated with decreased strain rate sensitivity with increasing temperature. Decreasing strain rate sensitivity is associated with serrations in the stress strain curves suggesting a Portevin-Le Chatelier effect. A detailed study has been undertaken to understand the serrated flow behaviour. The study is of considerable technological significance primarily because of two reasons: (1) tungsten heavy alloys are generally swaged in this temperature regime in order to improve the mechanical properties that include strength, ductility and impact toughness; (2) these alloys are also subjected to a low temperature ageing heat treatment in order to enhance the tensile strength. Attempt has been made to estimate the activation energy in this strain-rate temperature regime and correlate it with activation energy for diffusion of different elements in tungsten. The results are discussed in relation to the existing understanding of dynamic strain ageing especially in tungsten heavy alloys.

Solder joint reliability of Sn-Cu and Sn-Ag-Cu lead-free solder alloys solidified on copper substrates with different surface roughnesses

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In the present work, bond strength of Sn-0.7Cu, Sn-0.3Ag-0.7Cu, Sn-2.5Ag-0.5Cu and Sn-3Ag-0.5Cu lead free solders solidified on Cu substrates were investigated. The bond shear test was used to assess the integrity of Sn–Cu and Sn–Ag–Cu lead-free solder alloy drops solidified on smooth and rough Cu substrate surfaces. The increase in the surface roughness of Cu substrates improved the wettability of solders. The wettability was not affected by the Ag content of solders. Solder bonds on smooth surfaces yielded higher shear strength compared to rough surfaces. Fractured surfaces revealed the occurrence of ductile mode of failure on smooth Cu surfaces and a transition ridge on rough Cu surfaces. Though rough Cu substrate improved the wettability of solder alloys, solder bonds were sheared at a lower force leading to decreased shear energy density compared to the smooth Cu surface. A smooth surface finish and the presence of minor amounts of Ag in the alloy improved the integrity of the solder joint. Smoother surface is preferable as it favors failure in the solder matrix.

Aspects of time dependent fatigue in superalloys

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Superalloys belong to a special class of high temperature materials predicted to contribute the highest in terms of usage in supercritical and ultra supercritical power generation establishments. Mainly, depending upon the practice, these materials are used in the components where time dependent fatigue is a critical structural integrity issue. Various attempts have been made and are still being done to simulate the environment of steam turbine power plants depending upon the temperature, loading waveform and different control modes (strain control, stress control and strain ramp with stress hold). The present research interest mainly intends to predict the component life and critical observation of the contribution of time dependent damage component in microstructural degradation. Therefore, a detailed understanding of effects of loading waveforms and holding position is necessary because these matters have not been looked into with proper emphasis. Microstructural variabilities associated with typical superalloys also interact differently with different damaging variables and manifestation of defect substructure needs a careful understanding. Some attempts have been made in those aforementioned regards and determination of interaction intensity of time dependent and independent component has been done using testing and characterization efforts. Two superalloys (Haynes 282 and IN 718) were chosen for the present study and they were subjected to different waveforms like slow fast, fast slow, non peak strain hold, peak strain hold, peak and non peak strain hold etc., to compare the degrees of damage introduced by them. Different control modes were also chosen (strain control, stress control and strain ramp with stress hold) to investigate effect of control modes in damage evolution of IN 718. Finally, two different microstructures for each of these two alloys were chosen with variable precipitate size and their

creep fatigue responses were investigated at operating temperatures (i.e., 650°C for IN 718 and 760°C for Haynes 282).

Effect of thermo-mechanical processing on the microstructure and mechanical properties of NiTiCuFe high-entropy alloy

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Alloying can make drastic changes in mechanical and chemical properties. In conventional alloys, addition of other elements is usually restricted to only small amounts. High Entropy Alloys having generally more than five elements in equiatomic compositions show an enormous increase in configurational entropy making it thermally stable even at high temperatures. NiTiCuFe high entropy alloy was prepared by a medium frequency induction melting furnace. To study the effect of annealing temperature on the microstructure and mechanical properties, samples were prepared from the ingots and were subjected to annealing treatment at 600 °C, 700 °C and 850 °C for 2, 4 and 6 h. The phase evolution on annealing was studied using XRD. As cast structure showed a mixture of BCC and FCC phase. Annealing favoured the formation of FCC phase. Chemical compositions of the ingots were analyzed using Energy Dispersive Spectroscopy (EDS). Microstructural characterization was done using SEM and TEM. Thermo-mechanical processing studies were carried out using Gleeble 3800 to understand the effect of high temperature deformation on the changes in the microstructure. Gleeble was also used to determine the high temperature stability and properties of NiTiCuFe alloy.

Study of serrated yielding during nanoindentation of Mg-Li-Al based alloys

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Nanoindentation measurements within the hcp α-phase and the bcc β-phase were carried out on two thermomechanically (TM) processed Mg-Li-Al based alloys, namely Mg–9Li–7Al–1Sn (LAT971R) and Mg–9Li–5Al–3Sn–1Zn (LATZ9531R), to study the individual mechanical behavior of the phases. Commercially pure Mg and AZ31 Mg alloys were also analyzed to study the contribution of alloying addition on the mechanical strength in nano-length scale, such as hardness and elastic modulus of the constituent phases, in Mg–Li based alloys. The average nanohardness and reduced elastic modulus values of α-phase of LAT971R and LATZ9531R measured by nanoindentation were observed to be higher (80% and 30%, respectively) than that of pure Mg. The elastic modulus values of α- phase of both alloys were about 58 GPa, which is much larger than that of other Mg-based alloys (~45 GPa). Pop-in events (serrated yielding due to the Portevin–LeChatelier effect) were observed in both α and β phases of LAT971R and LATZ9531R alloys and were quantified using models developed by Taylor and Morris.

Influence of stress state on microstructure and mechanical behavior of Ti-6Al-4V and CP titanium

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Titanium and its alloys are extensively used in aerospace industries, because of its attractive mechanical properties such as high specific strength coupled with good corrosion resistance. Most often, these alloys are used in critical applications (pressure vessels, cryogenic tanks), where it experiences complex, biaxial loading conditions during its service. Hence, it is necessary to study and understand the mechanical behavior of such alloy under equi-biaxial tension as well as under different biaxial loading ratios for improving the reliability and integrity of such structures. Cruciform (cross-shaped) biaxial testing is one such technique, which offers the possibility to test under in-plane condition by subjecting tensile loads to arms of the cruciform specimen in each perpendicular direction. A biaxial test rig of 250 kN capacity was in-house custom designed to carry out biaxial tensile testing of titanium alloys till failure. Strainsmart system 5000 data acquisition system (DAQ) was used for recording load cell as well as strain gage readings. In addition to strain gauges, state-of-the-art, non-contact digital image correlation (DIC) technique was used to capture biaxial strain contours in the gage section of cruciform specimens. Optical and scanning electron microscopy (EBSD) analysis was carried out to understand the mechanical behavior of failed samples under biaxial state of stress.

Mechanical behaviour of 2D and 3D weaved SiC-matrix, carbon continuous-fibre-reinforced composites: flexural strength under static loading conditions

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Advanced materials such as Continuous Fibre-reinforced, Ceramic-matrix Composites (CFCCs) offer significant enhancements in variety of properties, especially tensile stress to fracture, flexural stress and most importantly, the fracture toughness (especially the propagation fracture resistance), as compared to their bulk monolithic counterparts. However till date, there are hardly any scientific studies reported in the open literature on the effects of fibre weaving conditions on the flexural strength properties of carbon-fibre based advanced ceramic composites, where SiC is used as the matrix. The present paper discusses the flexural strength behavior of these CFCCs in 2D and 3D woven conditions, obtained by static loading using 3-point bend/flexural testing. Fracture behavior and principal fracture modes are also evaluated and analyzed to account for the differences in the flexural properties (both stress and maximum strains to fracture). The study showed that the relative difference in the flexural strengths is more or less controlled by the nature of fracture, which is dictated by the differences in the fibre/matrix interface characteristics and to a greater extent the weave condition.

Damage quantification methodologies at varying length scales

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Quantification of damage and prediction of service life are of prime importance for engineering structural components. Hence, investigating the evolution of damage in structural materials under various loading conditions at all length scales is essential to predict or prevent failure. Damage, typically initiates in materials at the micro scale level as changes in dislocation density or grain boundaries or through formation of other phases which can be detected and quantified by X-ray diffraction analysis. At the meso scale level manifestation of damage can be quantified through indirect methods, wherein various damage parameters such as elastic modulus can be used to measure damage using the continuum damage mechanics approach. Fracture mechanics is the other possible approach which is well described for analyzing damage at the macro scale level. The present investigation focuses on exploring various methods for quantifying damage at all length scales and correlating them. For analysis of the same, different destructive and non-destructive evaluation (NDE) methods are explored. For non destructive evaluation, ultrasonic & thermography techniques are employed and for destructive testing, monotonic tensile loading and LCF were used. The damage evolution has been connected to changes in microstructure at early stages of loading though XRD, TEM or ultrasonic.

Creep behaviour of MRI230D Mg alloy produced by two different casting processes

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The MRI230D magnesium alloy was produced by two different casting processes i.e., ingot-casting (IC) and high pressure die-casting (HPDC). The alloy in both casting conditions was tested for creep at various stress levels and temperature. A detailed microstructural characterization of the alloy before and after creep tests was carried out. The microstructure of the as-cast alloy consists of α-Mg, (Mg, Al)2Ca (C36) and Al-Mn-rich phases in both casting conditions. A relatively finer grain size and higher volume fraction of finer, denser network of C36 phase was present in the microstructure of the HPDC alloy as compared to that of the IC alloy. The HPDC alloy exhibited superior creep resistance and it was attributed to the presence of finer and denser network of the C36 phase. The microstructures of the alloy in both casting conditions revealed increased amount of C36 phase following creep tests.

Grain refinement of Mg-Zn alloy processed through hot rolling for orthopaedic applications: mechanical and corrosion properties

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Metallic materials, like, stainless steel, titanium alloy and cobalt-chromium based implants have found their place in orthopedic applications in the last few decades due to their unique mechanical properties and compatibility. However, these material systems have much higher elastic modulus and strength than human bone. This dissimilarity in properties causes a major problem of stress shielding in human bone, leadings to disturbance in bone growth and often fracture and delamination at bone-implant interface. The use of magnesium and its alloys can be a possible solution to this problem, due to its similar density and elastic modulus to that of human bone. Magnesium alloys exhibits good biocompatibility and biodegradability also, but show poor strength which restricts their use in load bearing biomedical applications. Hence, improvement in strength is needed for these alloys, while keeping the elastic modulus unchanged. One of the popular techniques, used for improving the strength of bulk metals, is by grain refinement through severe plastic deformation. In the present study, the grain refinement of Mg-3Zn alloy has been achieved through hot rolling. Mechanical properties of this grain refined alloy are investigated to check its compatibility with human bone. Another problem exhibited by magnesium and its alloys is high corrosion rate in body fluid environment, which contains abundant chloride ions. Rapid degradation of Mg alloy, and subsequent accumulation in blood leads to increase in local pH. It results in cell death and tissue inflammation and more importantly mechanical integrity of the implant is lost quickly. Solid solution of Zn can increase the electric potential of matrix and improve corrosion resistance and increase the life of an implant. The corrosion properties of the grain refined alloy are also investigated to understand the effect of grain refinement on the same.

Microstructure and mechanical properties of Al6061-graphite composites fabricated by stir-casting process

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In this study, the experimental results of microstructural characterization and mechanical properties evaluation of Al6061-Graphite composites is presented. The composites containing 6 to 9 wt% of graphite were prepared using liquid metallurgy route in particular stir casting technique. For each composite, reinforcement particles were preheated to a temperature of 250˚C and then dispersed in two steps into the vortex of molten Al6061 alloy to improve the wettability and distribution. Microstructural characterization was carried out by optical and scanning electron microscopy (SEM). Tensile and hardness tests were performed in order to determine mechanical properties of composites. The results of microstructural study revealed uniform distribution of graphite particles and low porosity in micro composite specimens. The experimental results showed that the density of the composite decreased with increased graphite content and agrees with the values obtained using the rule of mixtures. The results of this study revealed that as graphite percentage was increased, there was significant increase in ultimate

tensile strength, yield strength and ductility, accompanied by a tremendous drop in the hardness of the material.

Elastic plastic fracture behavior and effect of band-overload on fatigue crack growth rate of an HSLA steel

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Study of fracture toughness and fatigue crack growth behavior are important parameters of structural materials. These parameters can be used to predict their life, service reliability and operational safety in different conditions. The material used in this investigation is an HSLA steel. In the first part of this investigation elastic plastic fracture toughness (JI_c and δI_c) were measured, by resistance curve method. Tests were carried out on CT specimens, using unloading compliance technique. These tests were conducted at three different displacement rates. It is observed that fracture toughness decrease with increasing rate of displacement. In the next part of this investigation effect of single overload and band-overload on fatigue crack growth of same steel were studied. These tests were conducted on CT specimens. Single overload and band-overloads were applied under mode-I condition, during constant amplitude (tension-tension) fatigue crack growth test. It is observed that overload and band-overload applications resulted retardation on the fatigue crack growth rate in most of the cases. It is also noticed that maximum retardation took place on application of seven successive overload cycles.

Comparative study on prediction of metastable amorphous phases using Ab-initio molecular dynamics and evolutionary algorithm

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Ab-inito Molecular Dynamic (MD) simulations are usually implemented for obtaining the structure of amorphous solids, where the interatomic potentials are obtained from first principles calculations using Density Functional Theory. In this method, a material in solid state is heated beyond the melting point and allowed to equilibrate at that temperature in the form of a liquid. Then, the liquid is rapidly cooled to room temperature so as to obtain the metastable amorphous phase, which corresponds to a configuration of atoms trapped in local minima. The method actually mimics the melt-quench technique used in experiments for obtaining the amorphous solids. This computational technique fails if the liquid phase obtained through melting differs significantly from the amorphous solid to be obtained. Instead of ab-initio MD, we can use the evolutionary algorithm technique for predicting amorphous phases through random structure generation and variational operators such as mutation, heredity, permutation and selection in a way analogous to that of biological evolution. As a benchmark, simulations are carried out to find the structure of amorphous silicon, which illustrates both the advantages and disadvantages of using these two techniques. We will also present some interesting results on predicted structures of newly developed zinc oxynitride amorphous semiconductors.

Session 6: Nov 15, 2014: [14:00-16:00]

Study of toughness of LRPC material at low temperature

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There is a steady demand of high strength steel wires for bridge cable and PC strand. In prestressed concrete composites, steel wires with higher elastic limit and tensile strength are required. PC strands are used in pre-stressed concrete girders for road, river & railway bridges and flyovers, pre-stressed atomic reactor domes, slabs, silos, hangars, aqueducts, high-rise buildings, viaducts and railway sleepers. In this paper we have studied the effect of toughness of LRPC grade at low temperature. In case of LRPC grade material pearlitic microstructure has poor impact properties due to weak carbide joints, as at the joints the dislocation starts piling up leading to increase in flow strength, which results in increase in the Peierls Nabarro forces, but due to presence of brittle cementite phases, the material easily reaches the fracture strength resulting in low impact toughness of the material. Thus the above study indicates that toughness property of the LRPC material is very low even at room temperature and the material is inferior for impact loading condition in room temperature as well as at lower temperatures. So it is infered that the LRPC type of material is not suitable for cryogenic application.

High strength, ultra-low friction and wear properties of MoS2 nanosheets coatings

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Bulk MoS2 has already been exploited for its actions as an efficient lubrication in components of blends and composites. In the recent years, the focus has shifted to the more efficient lubrication by 2D-MoS2 nanosheets. The formation of 2D-MoS2 nanosheets has been done by spin-coating a solution of Ammonium tetrathiomolybdate and PVA on a metallographically treated standard cylindrical disk shaped steel sample of 5 cm diameter and 0.8 cm height. This was followed by annealing under a suitable atmosphere and conditions. The formed 2D-MoS2 has been confirmed by characterization techniques like indexing, TEM, SEM, Raman spectroscopy and PL done on the 2D-MoS2 deposited SiO2/Si substrate. The indexing of the formed film shows the reference to be like 2D-MoS2. SEM and TEM analysis detected its layered occurrence and proved that a layered compound of 2D-MoS2 has been formed. To determine the number of layers, Raman spectroscopy and Photoluminescence was done which resulted in Δk value to be around 21.6 cm-1 and 2 PL peaks indicate that 2D-MoS2 formed was either a monolayer or a bilayer or a trilayer. Layers of different thickness were deposited on this metallographically treated sample of steel. These different samples were analyzed for mechanical properties as well as tribological tests for the optimization of the strength as well as friction coefficient respectively. High values of strength and very low values of friction coefficients were obtained.

Micromechanisms of creep-fatigue interaction in P92 grade of martensitic steel

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Tungsten added 9Cr-0.5Mo steel is being used as header pipes in coal fired power plants for the last decade. At present, the performance of this steel grade is reviewed and reassessed. Owing to the cyclic operations in present day power plants creep-fatigue (CF) interaction is identified as a potential degradation mechanism and a thorough assessment has been proposed by several utilities. In the present investigation, high temperature isothermal strain controlled low cycle fatigue (LCF) tests are conducted at 600 ^{o}C at various total strain amplitude values: 0.2, 0.4, 0.6, 0.75, 0.9, 1.05 and 1.2 with a strain rate of $1x10^{-3}$. The alloy is found to exhibit complete cyclic softening from the beginning of fatigue life for all the employed values of total strain amplitudes. Extensive transmission electron microscopic (TEM) studies and fracture surface examination under scanning electron microscope (SEM) are done to ascertain the micromechanism of fatigue failure. The CF interaction tests are carried out to understand the effect of hold time, strain rate on the CF life. The effect of hold time on micromechanism of failure will be discussed.

Effect of boron addition on sliding wear charecteristics of titanium alloys

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Titanium alloys are extensively used in aerospace industry due to their high strength to weight ratio. Near alpha titanium alloys are gaining priority due to their improved creep strength, oxidation resistance, reasonably good ductility, and excellent mechanical properties at cryogenic temperatures over α-β and β titanium alloys. The properties get altered in heat treatment conditions viz., as-cast plus aged and solution treated plus aging conditions. Present study has been carried out to investigate dry sliding wear behavior of near alpha titanium alloy IMI-685 with and without boron. Wear test experiments were carried out on pin-on-disc wear testing machine with varying loads (5Kg, 7.5Kg and 10Kg) and sliding speeds (0.52-3.66m/s). The characterization of investigated materials was done by using optical microscopy, scanning electron microscope (SEM), and X-ray diffraction (XRD). Micro hardness tests were carried out with a Vickers diamond pyramid indenter. Under the present experimental conditions it has been observed that maximum wear rate corresponds to a sliding velocity of 2.62 m/s at all loads and minimum wear rate corresponds to a lower sliding velocity of 0.52 m/s even with increasing load. It has also been observed that IMI-685 alloy without boron presents better wear resistance in solution treated plus aged conditions at low load and sliding velocity combination compared to as-cast plus aged alloy and boron combination.

Development of micro alloyed reinforced bars at light and medium merchant mill (LMMM) of RINL

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The technology of micro alloyed steels is expanding beyond its original emphasis on low-carbon, thermo mechanically treated reinforced-bars which conventionally have high yield strengths of about 500MPa with good ductility. The manufacturing process involves a billet that is heated to the rolling temperature in the walking beam furnace and is then subjected to rolling in the mill. After being reduced to the required final size (16 mm rebars) at the hot rolling temperature, it passes through a Temp-Core box. In the Temp-core process, the reinforced bar surface is cooled faster (with water) and gets tempered by the heat dissipated from the core followed by self-tempering on a cooling bed to obtain the required properties. The bar transforms to having an outer surface of martensite with a ferrite-pearlitic core. In this experiment Vanadium (0.06% max) with 0.2% carbon nominal chemical composition is used to produce rebars with optimized Temp-core parameters to attain required properties. Vanadium of this composition imparts higher strength levels (yield strengths of about 600MPa combined with good ductility) accompanied by better high temperature properties. This is due to the presence of Vanadium carbonitrides - V(C, N), formed as fine precipitates in the matrix of the rebars. Such fine precipitates are not easily resolved in a regular Optical/Scanning Electron Microscope, and will need characterization by high-resolution microscopy.

Analyzing the various parameters leading to grain coarsening in low carbon aluminium killed EDD steel

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The main purpose of this study was to analyze the various parameters which are the probable cause of grain coarsening in low carbon aluminium killed EDD steel. Extra Deep Drawing steels have better formability (n> 0.22) but lower strength (> 110 MPa) due to their lower carbon content (< 0.03 %). The formability, especially deep drawability of cold rolled low carbon steel strip is strongly influenced by the concentrations and distribution of solute elements (especially carbon and nitrogen), and their relationships to the formation, composition, size, morphology, crystallography and distribution of precipitates during annealing. These are controlled by the slab reheating temperature, finishing temperature, coiling temperature, laminar cooling, cold reduction, batch annealing parameters like heating rate, soaking time, etc. From the experiments it was inferred that the finishing temperature (FT) should be in optimum limits i.e., neither very high nor very low. Abnormal coarse grains mixed with fine grains were formed when the finishing temperature is lower than dynamic transformation temperature (DTT) due to large difference in surface energies and too large grains formed when the FT is too high. The time after rolling and before coiling should be kept to minimum as aluminum nitride formation occurs. Presence of aluminum nitride is undesirable in rolling stages, as it would result in the formation of equiaxed grains which are not desirable for EDD grade. Hence coil should be moved at a fast rate after rolling and before coiling. Microstructures of four coils were analyzed after hot rolling and effect of FT was established. Then corresponding to these coils, eight more coils were selected and the effects of Batch

Annealing Furnace (BAF) parameters were analyzed. The effect of FT and heating rate during BAF was successfully studied through these findings.

Study of mechanical properties of constrained groove pressed Cu-Zn alloy

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Material properties depend mainly on its microstructure and chemical composition. Modifications in microstructure of material can be made by the selection of suitable processing conditions. Mechanical strength can be increased many fold by reducing grain size using processes such as Severe Plastic Deformation (SPD). Present work involves use of Constrained Groove Pressing (CGP), a SPD technique, for improving mechanical strength of Cu-Zn alloy by imposing high strains. Microstructural characterization of the processed samples was done using optical microscopy as well as SEM. X-ray Diffraction was used in order to estimate the lattice strain as well as grain size of the samples after deformation. Changes in mechanical properties such as tensile strength and hardness were observed using uniaxial tensile test and micro-hardness testing respectively. Results show that constrained groove pressing effectively improved the mechanical properties of the alloy. Although the decrement in grain size with the number of passes in CGP was not as huge (45.6μm to 27.3μm) as it is expected from a SPD technique, there was a drastic decrease in elongation and concomitant improvement in ultimate tensile strength (~ 230MPa to~ 400 MPa) which shows the effectiveness of this particular SPD process. Ultimate tensile strength as well as hardness increased up to first pass but after that remained approximately constant with the number of passes in CGP. However, yield strength showed anomalous behavior. It increased in the first pass as compared to as-received condition, but decreased slightly in later passes, there after indicating signs of softening as well as existence of some micro-cracks. The evolution of deformation homogeneity was analyzed in detail using optical micrography, EBSD, as well as fracture surface morphology. In this work, we explore the cause of sharp increase in strength and drastic drop in ductility with only nominal change in grain size.

Elastic rebound and dynamic indentation behaviour of titanium alloy at high strain rate

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Evaluation of high strain rate behavior of materials using dynamic indentation technique involves a non-deformable ball impacting a sample material over a wide range of velocities. In a typical dynamic indentation test, the kinetic energy of ball indenter is balanced predominantly by i) elastic rebound, ii) plastic deformation and iii) stress wave propagation. The energy balanced by only plastic deformation represents the dynamic hardness. In order to estimate the energy required for plastic deformation, it is imperative to examine the energy dissipation in rebound and stress wave propagation. The present work essentially addresses these aspects. In the present study, the dynamic indentation tests have been carried out for titanium alloy samples (Ti-64) using tungsten carbide ball in the impact velocity range of 4 ms-1 to

55 ms-1 and corresponding rebound velocities were also measured. The elastic rebound characteristics have been analyzed in terms of variation of coefficient of restitution which is the ratio of rebound velocity to incident velocity of ball, and its effect on dynamic hardness. The coefficient of restitution was found to vary from 0.7-0.45 with variation in the impact velocity which suggests significant energy dissipation during rebound. The fraction of energy loss through stress waves was found to vary from about 4 % to 14 % which is again significant proportion of the incident kinetic energy and hence cannot be neglected. Thus, the dynamic flow stress-strain plot representing high strain-rate flow behaviour have been evaluated after making necessary corrections for energy loss through stress waves and rebound during dynamic indentation tests. The strain-rate for these tests was of the order of 10^4 s^{-1}. The data obtained using dynamic indentation technique compares well with the high strain-rate data obtained using split Hopkinson pressure bar technique.

Improvement in life of vibro-feeder liner of sintering plant of Bhilai steel plant using superior grade of wear resistant alloy

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The liners of vibro-feeder tray meant to discharge sinter from sinter cooler to conveyer belt gets damaged within 45-50 days of operation due to wear and abrasion. Liners are changed in sets and one set consists of three plates of different designs. Thickness of liners is 10 mm as per original plant design. To achieve better equipment performance of sintering plant of Bhilai Steel Plant, it was thought of using better material for vibro-feeder liners. Literature survey was carried out to find out the best combination of techno-economically and commercially viable material to improve the service life of the vibro-feeder liners. Based on the previous work experience and literature survey, Cr-carbide hardfacing alloy was selected for fabrication of the liners. A layer of 2 mm thick Cr-carbide hardfacing alloy was deposited over 8 mm base plates made with SAILMA-350 grade steel. For the side liners 4 mm thick layer was deposited to take care of severe abrasion & wear. Six liner plates were fabricated and put into operation for performance monitoring. The wear resistance improved significantly due to hardfacing in the top layer of the liner plates. This is due to high hardness (more than 550 BHN) achieved in the top layer causing significant improvement in the abrasion resistance property. Presence of high percentage of primary and secondary carbides coupled with well distribution of the elongated carbides in the matrix of the hardfaced layer has resulted in overall improvement in the wear resistance characteristics of the liner plates. The life of these hardfaced liner plates has improved from an average of 45 days to 180 days.

Crashworthiness of aluminium alloys

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The need to reduce the weight of automobiles has taken the center stage due to the rising fuel prices and environmental concerns. It has been seen in recent studies that the increased use of aluminium parts in automobile can reduce its weight by 20-25%, which in turn can reduce fuel consumption by 22%. The main concern with increased use of aluminium in car body is its crashworthiness i.e. its ability to absorb energy

upon impact. The research done on the crashworthiness of aluminium has been explored in this paper. Furthermore, a comparative analysis with crashworthiness of steel has also been studied. It can be concluded from the literature that although aluminium has poor crash energy capability, it can be improved by optimizing the geometry of crash absorbing members. The primary parameter under study is Specific Energy Absorption (SEA). Researchers employed both aluminum extrusions and hat shaped members to evaluate SEA. The SEA of aluminium hollow tube, single celled and multiple celled extrusion members were also measured. Furthermore, aluminium extrusions and hat shaped profiled members reinforced with aluminium foams and aluminium honeycomb structures were subjected to dynamic impact and static loading to evaluate SEA. It was observed that reinforcing extrusions and hat profiles with foam and honeycomb yielded optimum result in terms of SEA. Researchers also carried out three point bending tests followed by compressive loading of empty and reinforced aluminium members to evaluate SEA in real crash scenarios. Furthermore, it was seen that aluminium extrusions and hat shaped members underwent collapse in a very predictable manner, which helps in estimating the behavior of aforementioned under axial impact. A lot of research still needs to be done to increase the crashworthiness of aluminium so that widespread use of aluminium in cars can be achieved.

Investigation of insitu tensile deformation behaviour of interstitial free high strength steel under scanning electron microscope

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It is a known fact that it is impossible to study the plastic deformation processes at the microscopic level during tensile loading. Therefore, to study the tensile deformation stages at the microscopic level, tensile tests directly under scanning electron microscope are gradually getting popular. Present investigation has been done to study the progress of tensile deformation of automotive grade cold rolled IFHS steel of 1 mm thickness received from TATA Steel Ltd. Polished and etched miniature sized tensile specimen was tested under tensile loading inside a scanning electron microscope (FEI, Quanta 450). Two Vickers micro-indentation marks within the parallel length of the specimen have been used to measure the amount of deformation. Tensile loading was done in steps and a series of secondary electron images were captured. It is found that, although deformation occurs by slip, slip lines are not observed in all the grains at a time. This happens because of the orientation difference of the grains with respect to tensile deformation axis. Surface slip features gradually become prominent with increase of deformation and at higher level of deformation, out of plane movement of grains is found to occur, resulting in undulation of the surface. Besides, intergranular cracking of the surface grains were very much significant. However, the scanning electron fractography reveals that tensile failure of the specimen occurs by void nucleation and growth. It is believed that grooving of the grain boundaries due to deep etching is responsible for intergranular cracking of the surface grains. Finally, it is found that, although the yield strength, tensile strength and strain at maximum load agree well with the tensile test results of conventional specimens, the total elongation to failure becomes extremely large in case of miniature sized specimen because of its very small gauge length.

METAL FORGING

Invited talks

Session 1: Nov 15, 2014: [11.00- 13.00]

Recent Advances in Metal Forging

G. V. Prabhu Gaunkar

The art of forging using heat and impact or pressure to mould metal mass into desired shapes was known to our ancestors some millennia back. From craft work, hand tools and primitive weapons to components for automobile, aerospace and energy sectors has been a long journey in the development of techniques, equipment and technologies of forging process. The size, shapes, functionality and durability of forged parts depended on the power of the hammer and skills to produce and transform suitable metals and alloys into desired shapes. Increasing demands on the performance of the forged components in terms of high service stresses, exposure to elevated or low temperature environments, their durability, etc., led to advances in the design of high performance equipment, development of new alloys and their processing schedules. Development of materials with good formability, high strength to weight ratios, good machinability, high toughness and fatigue strength, ability to maintain surface integrity in aggressive environments along with design of superior equipment and production processes have received particular attention in relatively recent times. From carbon and low alloy steels to precipitation hardening steels, titanium and magnesium alloys, there has been a quantum enhancement in the complexity of the alloys systems developed and converted into components by forging process. Forgings are produced today with ever increasing complexity in shapes and sizes, dimensional tolerances, performance assurances and compliance with standards and specifications for wide spectrum of applications. The closed die forging equipment designed to produce large number of components or high capacity forging presses, sometimes exceeding 50,000 tons, producing large size high value forgings employ CAD/CAM for design and computerized controls for operations along with process simulation tools for the design of processing schedules. Near net shaped products are now produced using relatively new processes such as forging of cast products and squeeze casting, where pressure is applied during solidification itself for special alloys with quality and economic benefits. We propose to examine some of the key heir developments and challenges faced by the forging industry in relation to the equipment and production processes, complexities associated with the processing of high performance materials, optimization of yield, economics and global trends in energy saving measures, environment management, education and training strategies, etc.

Multistage closed die forging technology for the production of aircraft main base plate undercarriage fitting component

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The key driving forces in the manufacture of components for the aircraft industry are weight reduction, application-specific performance improvement and affordability. The most important factor is the strength to weight ratio and performance reliability. Apart from these primary factors, wider process windows (accommodating easier manufacture), improved structural reliability and suitability for periodic inspections for structural health monitoring are also desirable. Development of a large size aluminium alloy multi-stage closed die forging process technology for production of an aircraft main base plate undercarriage-fitting component was taken up. Presently, such components are machined out of a slab feedstock. Al alloy raw material stock of weight 221 kg is machined into a finished component of weight 12 kg with a buy to fly ratio of 18.4:1. This process also results in inferior mechanical properties due to the absence of continuous grain flow and considerable material wastage. Through a newly developed close die-forging process technology, the finished component can be realized from a billet stock of 50 kg, resulting in a buy-to-fly ratio of about 4:1. The component manufactured by multi-stage closed die forging process technology has met the following objectives.

- Uniform mechanical properties, clearly above the AMS specified values for the given thickness – The results fall within a narrow band with the longitudinal tensile strength marginally higher than in the other two orthogonal directions.
- Improved buy-to-fly ratio from 18.4:1 to 4:1: The feasibility of the process to produce such an intricate shape proves that the method can be adopted / adapted for mass production with reproducible quality.
- Shorter manufacturing cycle: When mass-produced, the cycle time will be much less than that needed for machining the component from a plate / slab.
- Forged components exhibit superior properties as compared to machined components and this is expected to lead to vastly improved long term and reliable performance.
- Structural integrity achieved in closed-die forging extends the design limits of the forged parts.

Contributory papers

Session 1: Nov 15, 2014: [11.00- 13.00]

Simulation of radial forging process of Zr2.5% Nb alloy for pressure tube manufacturing and application in PHWR

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Pressure tube is a major life limiting structural tube used in the pressurized heavy water reactors (PHWR), and requires high resistance to creep and irradiation damage for the long term operation. Zr-2.5 wt% Nb alloy is presently being used as a pressure tube material in pressurized heavy water reactors (PHWR). In recent times, there has been a greater interest towards increasing the life of pressure tubes in advanced reactors. So a forging has been introduced for the effective breaking of cast structure in case of Zr2.5%Nb alloy in the modified manufacturing route for pressure tube. The established process route for the manufacture of pressure tube for the 700MWe PHWR reactors includes the primary breakdown of the cast structure using Radial Forging. Considering the criticality of the product, it is desirable to understand the role of process parameters in the Radial forging process. A Finite Element (FE) based modelling was carried for the Radial Forging process to understand the effect of process parameters such as ingot preheat temperature, die velocity etc. The effects of these parameters were studied for the overall distribution of the strain, strain rate and temperature across the cross section of the forged billet. The model was made

in Deform 3D commercial FE package, and the model was validated using the actual Radial Forging load for the same pass. The current simulation study was involved in understanding the parameters for the first pass forging from 500 mm to 475 mm. The material flow stress data of cast Zr2.5%Nb with respect to varying temperature and strain rate were generated using the Gleeble uniaxial compression testing. The result obtained shows that the best region for working of Radial Forging is in β phase field of the material, which starts above 900°C. The temperature rise due to deformation heat generated, and all other outputs are more uniform across the section in β phase region compared to α+β phase region.

Characterization of hot workability of A48P2 steel using dynamic materials model

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In roof slab of liquid metal cooled Prototype Fast breeder Reactor (PFBR), A48P2 steel is to be used in form of plates and hence necessarily to be produced by hot rolling. In addition, the rolled plates of A48P2 needs to be free from all kinds of micro structural defects as the in-service mechanical properties needed in the final component such as strength, ductility and fracture toughness depends on the microstructure. Presence of any flaw in microstructure will not only minimize the load bearing capacity of the plate but may serve as a initiation point of crack propagation and in turn will enhance the chances of failure before the designed life of the roof slab. To avoid these consequences, it is necessary to take advance measures to manufacture defect free A48P2 steel plates. This needs a proper design of manufacturing process. It is known that optimization of processing parameters for efficient thermo-mechanical processing needs detailed characterization of high temperature flow behaviour of the steel. Towards this end, isothermal hot compression tests has been carried out in a wide range of temperatures 1223 K–1473 K and strain rates 0.001–10 s^{-1}. The experimental data has been used to develop processing map based on Dynamic Materials Model. The stable domain of the map exhibits three major peaks P1, P2 and P3. P1 with peak efficiency 40% occurs at around 0.001s^{-1}, P2 with peak efficiency 40% occurs at around 10 s^{-1} and 1273-1350 K. P3 with peak efficiency 35% occurs at 1s^{-1} and 1375-1450 K. Even though P3 shows lower efficiency of power dissipation, the variation of efficiency around this peak remains within 35-30% over a wider range of strain and strain rate and can be considered as the domain for hot deformation. Extensive microstructural analysis has been carried out to validate the domains exhibited by processing map. The specimens corresponding to the unstable domain shows prominent signature of flow localization in the microstructure, whereas the specimen deformed at stable domain shows defect free equiaxed grains.

Challenges in manufacture of heavy stainless steel forgings – thick plates: LTSSHF experience

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This article covers the steel making, ingot casting and forging challenges in making the largest stainless ingot in India. Unlike other mass process of stainless steel production, here each heat is designed to meet the specific requirement of the special forgings required for particular application.In this case the challenge involved obtaining a low magnetic permeability (less than 1.05) for special application in ITER

project. Starting with meticulous selection of scrap for ensuring precise chemistry control and process controls during Vacuum Oxygen Decarburization (VOD) and controlling casting is described in this paper.While casting a good ingot makes a good beginning for forging, soaking at high temperature to homogenize chemistry, reduce segregation, soaking for forging and forging operation also put challenges to technologists and production engineers for making such high thickness plates. Two routes one with only forging and another with partial forging followed by rolling in plate mill were used to obtain final dimensions. Heat treatment and testing including ultrasonic testing and controls to ensure low magnetic permeability were other challenges that were addressed during manufacturing of these high thickness plates of these SS 304/304L plates.

Control of end defects in open die forging using finite element method

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In forging of huge and bulky ingots into cogged bars in open die forging, defects formed like fishtail shapes in front and back end shapes which results in cropping losses. In this paper the work deals with to minimize the end cropping loss and optimize the material by designing pre-form end shapes using finite element method. A three dimensional simulation carried was carried out for cogging of ingot in open die forging in FORGE 3D software with actual press parameter. With help of point tracking simulation tool in FORGE 3D software we predict material flow behavior of cogged bloom. Similar simulations are performed with varying press parameters like die width and anvil edge radius. On the basis these simulations, different analysis like strain rate analysis, vector analysis and stress analysis are carried out. Simulation results shoes reduction in end defects.

METAL FORMING

Invited talks

Session 1: Nov 13, 2014: [13.30- 15.30]

Role of thermo-mechanical simulation studies in High temperature metal forming

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Thermo-mechanical simulation studies can be successfully employed to study the hot deformation behavior during high temperature forming, like hot rolling, forging, extrusion, etc. It involves study of flow softening behavior due to recrystallization and recovery, flow stress determination, process simulation, determination of no-recrystallization temperature, stress-relaxation studies, understanding the influence of deformation on microstructural evolution, hot workability studies, edge cracking, and so on through hot compression studies. It can be divided into two categories, (i) uni-axial flow stress compression and (ii) plane strain compression depending upon the stress condition. Both these tests find different applications: whereas, uni-axial compression is more close to forging operation, plane strain condition is experienced in hot rolling. In a steel industry, hot rolling is the main finishing mill. It involves reheating of slab at high temperature, multi-pass deformation at high temperature in the roughing stand, multi-pass deformation at lower temperature in the finishing stand followed by post-cooling and coiling in case of hot strip mill. Selection of parameters for each of these steps is normally done based on some previous knowledge either from the literature or from practical experience. However, optimization of these steps is must to utilize the mill to its fullest extent in producing quality products with desired properties. Some of the common problem encountered during hot rolling is the mill load, edge cracking, formation of undesired microstructure, and poor mechanical properties. Gleeble System can be effectively used to study and overcome these problems. The present paper highlights the research work carried out and the application of such studies in solving some real world problems in steel industry.

Session 2: Nov 15, 2014: [14.00- 16.00]

Thermo-mechanical and microstructure evolution during hot rolling

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A transient thermo-mechanically coupled Finite Element Method (FEM) based model for hot rolling of AA 5083 alloy is developed where the material is considered to be of thermo-viscoplastic type while hot rolling is considered to be under plane-strain conditions. The temperature and strain prediction of the FEM simulation is incorporated in a microstructural model where dynamic recrystallization through particle stimulated nucleation (PSN-DRX) and static recrystallization (SRX) is taken into account. The SRX model is

centered on Avrami equation while the PSN-DRX model is a modification of discontinuous dynamic recrystallization model. An analysis of the results suggest that accurate assessment of constitutive behavior of the alloy, conversion efficiency of plastic deformation to heat and heat transfer at the roll/metal interface are critical for precise prediction of experimental results.

Contributory papers

Session 1: Nov 13, 2014: [13.30- 15.30]

Manufacture of wire rod with defined oxide scale formation at JSW Steel, Salem works

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JSW Steel Limited, Salem Works (JSWSL), is an integrated steel plant, having a production capacity of 1.0mtpa (million tons per annum) of high-grade automotive special steels. The steel making route comprises of: Blast furnace – Energy Optimizing Furnace – Ladle Refining Furnace – Vacuum Degassing – Continuous Casting. At JSWSL, scale formation in the hot rolled wire rod was controlled by controlling the laying head temperature. Hot rolled steel wire rods/bars, normally further subjected to various processes like phosphating, coating and forming process. However before further processing the oxide layers formed while processing needs to be removed. A study carried out at different application and method of descaling thrown light on the requirement of different types of scales on surface to avoid further complications at customer end. On experimental method by varying laying temperatures it was made possible to vary the scale formation and made suitable with the application requirement. The present paper discusses the manufacturing of wire rod coil with defined oxide scale formation to satisfy our customers.

Simulation of strain path diagram of low- Ni austenitic stainless steel incorporating evolution of microstructural features

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Demand for austenitic stainless steel has been increasing in industrial sector mainly in automotive industry because of its good corrosion resistance and favorable mechanical properties such as high strength and ductility. Most of the industrial metals forming processes are characterized by a complex strain path history. A change in strain path has a significant effect on the mechanical behavior of materials. Physical origin for this strain path dependency may be due to evolution of microstructural features such as twinning, phase transformation (α martensite) and local misorientation development. In this study strain path diagram (SPD) of low nickel austenitic stainless steel was plotted experimentally and numerically. Different Strains & Strain paths were determined experimentally through limiting dome height (LDH) tests. Microstructual studies were performed at these strain and strain paths using EBSD and X-RD techniques.

During deformation austenitic fcc phase was transformed to bcc phase martensite. The phase transformation had strong strain and strain path dependence. Finite Element (FE) analysis was performed to simulate SPD using available materials models. A large difference between experimental and simulated SPD was observed. A new empirical model was developed based on microstructural development at different strain & strain paths and incorporated in FE simulation to improve the SPD predictability.

Improvement of ductility of high carbon steel wire rods- a case study in Visakhapatnam steel plant

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High carbon steel wire rods of PC115 grade are drawn to 3mm size wires for prestressed concrete applications. The wire rods can be drawn to wires smoothly without breakage if their ductility is more. Higher the reduction of area of wire rods, higher will be the ductility. An attempt is made in Visakhapatnam Steel plant to study the effect of retaining time of wire rod coils on the reduction of area. Wire rod samples from 7mm size wire rod coils of PC115 grade were collected during rolling and subjected to tensile test in Universal testing machine on 7th, 15th day after rolling. The reduction of area continuously improved by 35% maximum and remained constant or decreased further. Hydrogen content was also measured and found to be decreasing with increase of retaining time. The metallography study showed no variation in the microstructure of steel after retention period. The improvement of reduction of area can be attributed to gradual release of hydrogen from steel with time. The customers also informed that drawability of the coils improved after retaining them in yard for 15-20 days.

Cost optimization through the material yield improvement in new generation passenger cars

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Approximately 70% of cost of a vehicle is being contributed by the materials used. To remain cost competitive, all the auto manufacturers are continuously exploring various techniques to optimize the material consumption. It is essential for an Auto OEM to innovate and mostly these are the trade secret for the manufacturer. Tata Motors, the largest automobile manufacturer in India is also continuously innovating the new ways to optimize the material consumption and material cost reduction. Use of advanced material grades, commonization of steel grades, use of tailor welded blanks, advanced tool design to optimize the material input, multi cavity tooling for production of multiple parts etc. are some of the methods which optimize the material input and reduce the material cost.This paper will explain how Tata Motors adopted the part design simulation techniques along with Advanced Quality Planning Systems from project conception to actual manufacturing of the product. This has helped to improve the material yield substantially in the future models of both passenger car and commercial vehicles. Through this rigorous and eventful journey, Tata Motors could achieve a 35% improvement in material yield for its new generation passenger cars. This gave substantial cost advantage and weight advantage. Needless to mention that, all along this process all the safety norms and regulations were adhered

Design of die and punch for sheet metal forming and optimization using GA

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Formability engineering analysis is an application technology used in Sheet Metal Forming industries. Its technical foundation includes metallurgy, engineering mechanics and forming die structure. Stamping is a manufacturing method used to form a sheet metal blank into a stamped part in the plastic deformation state, commonly using dies and stamping machinery such as mechanical presses. Die and punch design parameters are die and punch material, die corner radius, punch nose radius, sheet metal thickness, clearance, draw beads, coefficient of friction. Die component must be made of a material that has properties suitable for the conditions of service. The machining and fabrication play a critical role in the design of die and punch, since all the individual parts are machined separately. The main objective of this research is to determine the values of the controllable process variables that improve the output of the Sheet Metal Forming process. Rules of thumb and trial-error methods without the use of scientifically based engineering methods are very costly with regard to money and time and therefore it is a great need to developthe optimization and experimental engineering methods which can solve the problem easily and effectively to reduce the production cost and time. In areas like automobile, aerospace and marine, the Industry requires optimally designed die and punch. This study deals with the design and manufacturing of die. The design parameters considered are punch shape, clearance, punch speed and force, die material and operating pressure. Genetic algorithm is used to optimize the important sheet metal forming parameters like deformation velocity, die and punch geometries, drawing load, blank holder pressure and coefficient of friction. Such analysis is important to reduce the development time and cost for the process and improve upon the quality and productivity.

Improving the slab temperature profile by using the datalogger test and validating the heating model in the reheating furnace

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Hot Strip Mill in Tata Steel at Jamshedpur has three walking beam type slab Reheating furnaces of 300 T/hour capacities each. Furnace -1 &2 are mixed gas (Coke oven gas and Blast Furnace gas mixture) fired and the newly installed third furnace is using Blast furnace gas with the help of regenerative burners. Furnace 1&2 both has level-2 controls which are running since years successfully. Time to time the model is deviating because of the changed environment inside, particularly for the buildup scale on the hearth and for the deteriorated refractory condition. Subsequently the model validation is done in both the furnaces by conducting the “Datalogger” test, where, a test slab is fabricated with installed thermocouples and the datalogger in it. After the slab fabrication is complete, it is charged into the furnace by prior planning. This paper demonstrates an innovative temperature profiling method for the slab reheating furnace. The data logger captures all the temperature data and the tracking is done simultaneously during the entire slab residence inside the furnace. The actual data obtained by the datalogger along the slab location inside the furnace is then compared with the existing model. This helps in deciding the new

offsets to reduce the gap in the actual and calculated temperature and the furnace model is tuned accordingly.The improvements are observed after applying new nodal offsets in the furnace process. These offsets are implemented in the heating curve of both the furnaces.

Technology for the production of TMT bar in the new wire rod mill of ISP, sail

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The new Wire Rod Mill is one of the main production units of the 2.5 MT Expansion and Modernization Program of IISCO STEEL Plant, SAIL and started production since 2013-14. Though the mill is capable to produce 5.5 mm to 22 mm plain round of different quality and TMT bar 6/8/10 mm sizes, but presently are producing TMT bars only. The billets are charged at the walking beam type Re-heating Furnace for heating the bars to 1150^{0}C and then pass through the roll grooves of sixteen stands to be reshaped and reduced in oval-round sequence before being fed into 2-pass No Twist Block and then subsequently in 8-pass No Twist Block. In no twist blocks the successive precision pass grooves in the Tungsten Carbide rings are oriented at 90^0 to avoid any twisting during rolling.The finished bars at 870-880^{0}C temperature are passed through the specially designed cooling tubes to attain the desired physical properties. Finally the quenched bars are coiled through laying head and automatically compacted and strapped before being dispatched. All the pass grooves are cut in high precision CNC machine with an accuracy of less than 10 µmm to enable the mill to roll in negative tolerance only. As the notches in the finishing groove is maintained at same depth (0.7 mm for 8 mm TMT) along the entire length, so the transverse rib height is uniform from one side of the longitudinal side to the other side. This ensures better bonding strength when used in concrete. A typical microstructure analysis below revealed that a uniform dark colored rim of tempered martensite with a rim thickness of 0.98 mm is formed at the surface whereas the core constitute of a coarse ferrite-pearlite structure. The superior quality TMT bars (Fe500D) of more than 50000 T has already been produced with precision dimesional, rib & brand mark quality.

Changing land open extrusion of aluminum and aluminum alloys

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Open extrusion is an operation in which a short billet is pushed through a narrow opening that determines the cross section of the final product. This technique is basically a combination process characterized by extrusion and free forming. The geometry of the operation is described by the semi-cone angle, reduction and the initial aspect ratio. Due to the absence of container, the container wall - billet frictional force is eliminated and this leads to a large reduction in the total force required for extrusion. In changing land open extrusion, the land diameter is changed in stepwise manner and large strain is induced in the material. In the present research work, aluminum / Al-Mg-Zn alloy billets of varying length to diameter ratio (*l/d*) were extruded through the open dies with stepwise decreasing land diameterand different die angles to obtain the diametric change from 25mm to 21mm. The samples were extruded at room temperature using Universal Testing Machine of 40kN capacity with graphite powder as the lubricant. The velocity of the ram was maintained at 3 x 10^{-4} m/s. Hardness variation before and after extrusion and also as a function of die angle was studied in detail. Microstructural characterization of the extruded sample was done using optical microscope (OLYMPUS) and Scanning Electron Microscope (CARL ZEISS).

Development of single pass family of rolls in bar and rod mill to reduce section change time

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JSW Steel Limited, Salem Works (JSWSL), is an integrated steel plant, having a production capacity of 1.0 MTpa (million tons per annum) of high-grade automotive special steels. At JSWSL, Bar and Rod Mill is having facilities for rolling coils, spring steel flats and bars through different routes such as No Twist Mill Block , Garret Coiler and Cooling Bed. To cater to stringent customer size requirements with reduced tolerances, we also have the RSB supplied by M/s Kocks, Germany. An average of 70 sections is rolled every month with a section change delay of 12.5 %. There are different families of passes for rolling products in the range of 5.5 mm to 60 mm. Due to the existence of different families of passes, every section change require a significant pass change time. For every section change, section setting contributes to 25% of the section change time, which in turn reduces the possibility of producing first bar right. Due to more changes required between product families, the probability of misroll is very high. Different families of passes require a wide range of rolls, which increases the roll inventory and the number of stands being prepared. To minimize the losses such as delay, inventory, misroll, etc as stated above, we developed single pass family for the six input sizes required for RSB. The present paper discusses the development of single pass family and its advantages.

Rolling of high strength rebars (IS1786FE600) at VSP

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With rapid pace of development in infrastructure sector there will be a huge demand for high strength rebars in India for high rise buildings and other infrastructure projects. In the 2008 issue of IS1786 a new sub grade was introduced (Fe 600) to cater to these needs. In the thermo mechanically treated low carbon rebars there was limitation in achieving the high yield strength coupled with good ductility and bendability as brittleness increases with increasing % Martensite. Hence micro alloying route was selected to produce high strength rebars. To achieve good ductility the process parameters of tempcore was changed with an aim to increase the tempering temperature of the rebar. A trail run was made successfully by rolling 16 mm dia rebar by micro alloying to low carbon steel and treated with variation in tempcore process parameters at bar mill, VSP. The steel making process and rolling details are discussed in this paper.

Ageing behavior and mechanical properties of aluminium – magnesium – silicon alloy (AA6061) processed byequal channel angular pressing

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Equal channel angular pressing (ECAP) is a severe plastic deformation technique used to obtain ultrafine-grained microstructures. In the present investigation, solid solution treated specimens of 6061 aluminium alloy were processed by ECAP up to two passes using processing routes BC and C with solid solution treatment as the pre-ECAP treatment and ageing as the post-ECAP treatment. Differences in mechanical behaviour were investigated and ageing behaviour was studied at different ageing temperatures ranging from 120°C to 175°C for various time periods. Hardness increased for specimens processed by ECAP through route BC where as in processing route C, the hardness first decreased and then increased at all ageing temperatures. In ECAP processed material, the peak hardness was achieved at 140°C with ageing duration of 6h for route BC and 5h for route C. On the other hand, in T6 treated commercial 6061 alloy not processed by ECAP, the peak hardness was achieved at 175°C with ageing duration of 8h. The material processed through route C exhibited superior tensile strength when compared to that processed throughroute BC. Post-ECAP ageing treatment did not result in a significant increase in the tensile strength. However, it decreased the ductility when compared with the ECAP processed material without post-ECAP ageing treatment.

Scientific approach to reduce roll wear during hot rolling

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Rolls in hot rolling mills are most important tools for producing hot rolled steel strips. During hot rolling of flat products, the material being rolled varies in geometry, resulting in variation in temperature of roll surface from pass to pass. The rolls are required to sustain higher rolling load, which is necessary for deformation of the metal to the desired size. For maintaining the good surface of the rolls, the roll cooling system should be effective and efficient. Due to progressive wear of the rolls, loading condition of the mill varies. Wear of the rolls may be along the barrel length or may be localized to a particular length. Rolls wear affects the product quality, roll life and input cost of production. The wear rate of rolls is highly dependent on its temperature. The heat picked up by the roll body during hot rolling require to be extracted by cooling with required amount of water sprayed at adequate pressure over the roll body. There were higher grinding off-take, higher specific roll consumption and generation of fire-cracks on roll surface due to inadequacy of earlier cooling system of roughing stand of Hot Strip Mill, Bokaro Steel Plant. A scientific roll cooling system with double row spray concept, over-lapping of spray, optimum location of spray headers, intense and uniform cooling of work roll and mild cooling of back-up roll, higher impinging pressure by using flat jet nozzles was designed and commissioned to take care of heat generation at roll bite through maximum heat transfer effect with optimum density of the coolant. The modified system had resulted in reduction in grinding off-take by 25-30%, specific roll consumption by 25% and fire-crack formation in work rolls. Roll thermal profile improved by maintaining the temperature difference between centre and edges from 8°C to 4°-5°C. Improved roll thermal crown had also resulted in improvement in productivity, shape and quality of rolled product.

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FEM & experimental study of void closure behavior during hot rolling of an ingot

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Every cast steel ingot product hassolidification shrinkage [primary & secondary in some cases] associated with central looseness containing pores and cavities. The as-cast ingot has varying degree of micro-segregation depending on the depth of the solidification layer and grain structure. Such heterogeneous structure has to be broken down and porosities have to be well consolidated during the subsequent hot deformation operations. Hot rolling is one of the processes employed for deformation of small as-cast ingot. The process gives higher productivity, dimensional control and higher yield of the product. It is of importance to assess the extent of void closure and soundness during the hot rolling process. In this study, an effort was made to examine the hot rolling of a typical 4MT carbon steel as-cast ingot, C45 grade, with a pre-defined level of porosity using finite element based FORGE metal deformation software. The effect of varying hot rolling parameters such as initial cooling of ingot, draft and roller rpm on the void closure behaviour was examined theoretically till a reduction ratio of 4.3. The consolidation achieved in the rolled billet, rolling torque and strain penetration were validated by experimentation. The initial cooling of ingot gives better core strain penetration which will be helpful for reducing porosity. Higher draft and lower roller rpm gave the rolled billet with minimized porosity. Thus, it is possible to optimize the hot rolling parameters using the FEM based simulations as a virtual experimentation tool.

Effect of processing techniques on the microstructure and tribological characteristics of al-10Cu-Fe alloys

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The present study deals with the effect of processing techniques on the microstructure, mechanical properties and tribological characteristics of Al-10Cu-Fe alloy. Resistance heating furnace was used for melt preparation and the melt was cast into metal mould of the centrifugal casting machine mould at pouring temperature of 700°C. Same furnace with stirring and bottom pouring arrangement was used for rheocasting and poured into the metal mould in the semisolid state i.e. at 600°C. In Strain Induced Melt activation(SIMA) process, the as cast alloy was subjected to warm working at 400°C for 50% reduction in thickness with the help of Power Hammer and was then annealed in semi-solid region i.e. at 580°C held for different times varying from 5 to 55 minutes. The microstructural features were examined with an Optical Microscope. Mechanical properties and tribological characteristics of the alloys were evaluated. Microstructure of cast SIMA processed alloy showed semi globular to globular morphology of the primary α-phase compared to the dendritic morphology of the metal mould and centrifugal cast alloys. The grain size and hardness of SIMA processed samples increased with the holding time. SIMA processed samples showed fine grain size varying from 23 µm to 61 µm with change in their hardness from 118 to 124 VHN compare to a lower values of hardness and to relatively higher grain size of alloys processed by other

routes. These changes brought by SIMA processing are presented and discussed in the light of microstructural modification

Mathematical model based software system for optimizing ingot heating time with intermediate holding to minimize crack formation

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Reheating of steel ingots in batch furnace like soaking pit and box furnace is an important step for further thermo-mechanical processing like forging and rolling operations. The ingots are heated up to 1100 – 1200°C, and since this is a high temperature and energy intensive process, prolonged or excess heating time will cause productivity and energy loss, as well as oxidation and scale loss. On the other hand if it is heated very fast causing significant temperature difference between the surface and core temperatures, will lead to thermal stress generation, distortion and cracking. Furthermore, rapid heating of the surface to attain the specified temperature, without thermal homogenization of the slab can causes problems during hot rolling or forging operations, which may lead to cracking, roll stuck and forging problems. Therefore the aim of the heating process is to avoid any excessive thermal stress, particularly in the vulnerable ferrite to austenite phase transformation range, and also to minimize the time for thermal homogenization. To numerically simulate the process, a detailed two dimensional finite difference model is developed by using generalized Cartesian and cylindrical axi-symetric equation, and Crank-Nicholson technique. To accurately simulate the process we also have to consider all the complexities of the process like phase transformation and anomalous behaviour of thermal conductivity.

Performance improvement of pusher type reheating furnace by enhancing life of discharge end slots and skid pipe insulation in plate mill, BSP

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Plate Mill of Bhilai Steel Plant (BSP) is having 3 pusher type reheating furnaces (RHF) of capacity 120 t/hr. In each furnace there are 8 refractory slots at discharge end to facilitate movement of extractors for lifting and placement of heated slabs on rolls. These slots were made of 70% alumina low moisture castable (LMC) with mechanical anchoring system and getting damaged within 4-5 months restricting movement of slabs. Main reasons for low life of slots were high thermal shock and mechanical abrasions by moving slabs. Inside the furnace, slabs move over water cooled skid pipes. These pipes were insulated with 70% alumina LMC. Owing to low thermal shock resistance, castable falls increasing flowing water temperature inside the pipes. This results higher steam generation leading to higher energy consumption. The furnace was therefore put down for about 10-12 days for repair of slots and skid insulation in every 4-5 months hampering production. In order to improve the performance, all the slots in RHF no.3 were dismantled. New slots were made after building anchoring system using SS-304 rods. Water cooling system was also

introduced. Casting was done with 85% alumina Ultra Low Cement Castable (ULCC) having HMOR of 70 kg/cm^2 using steel formats. Skid pipes were thoroughly cleaned to remove old castable and burnt anchors. Newly designed "Y" shaped SS-304 anchors were welded at a distance of 250 mm in 4-9 layers across the dia depending on type of pipe. Casting was done using RDCIS developed 60% alumina andalusite based LMC and formats. After 24 hrs of air curing, slow heating was done to avoid cracking. The furnace has been commissioned in September 2013. The steam generation of 5 tons/hr at the time of commissioning has not increased since last 10 months of continuous operation of furnace. Condition of all the slots and skid pipes are found to be good. The improved performance has eliminated furnace shutdown in every 4-5 months increasing production by about 20,000 tons/annum.

Formability of third generation advanced high strength steels (AHSS)

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Automotive industry is currently focusing on using advanced high strength steels (AHSS) due to its high strength and formability for closure applications. Quenched and partitioned steel (Q&P) is one of the material for this application among other AHSS. The present work is focused on formability analysis of third generation advanced high strength steels. To mimic complex strain path condition during forming of automotive body, LDH tests were conducted and samples were deformed in servo hydraulic press to find the different strain path. FEM Simulations were done to predict different strain path diagrams and compared with experimental results. There is a significant difference between experimental and simulation results as the existing material models are not applicable for Q&P steels. Micro texture studies were performed on the samples at different strains and strain path using EBSD and XRD techniques. It was observed that austenite is transformed to martensite and texture developed during deformation had strong impact on limit strain and strain path. A new material model is developed based on microstructural evolution during deformation to improve the formability.

Metal coating on work rolls in blooming mill to avoid pass slippage

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JSW Steel Limited, Salem Works (JSWSL), is an integrated steel plant, having a production capacity of 1.0 mtpa (million tons per annum) of high-grade automotive special steels. At JSWSL, Blooming Mill produces Rounds and Round Cornered Squares (RCS) of various sizes from 55 mm to 240 mm predominantly for forging applications pertaining to automobile sector. During rolling of blooms using the newly dressed work rolls, there was difficulty in biting of metal by the rolls. The metal slips at the surface of the rolls due to smooth finish obtained through machining, which resulted in mill stoppage for 2 hours during every pass/section change. The practice of pass burning through conventional sampling method is also not possible due to larger stock size. Several measures such as reducing the input stock size, V–shaped cutting at front end of the input bloom, adjustment of roll gap and mill tension were taken to solve the pass slippage problem. All these measures were not fruitful. A special metal coating on the rolls was tried to

make the surface of the work rolls rough. The present paper discusses the method of metal coating suitable for Blooming Mill rolls and its advantages.

Evaluation of forming limit diagrams considering non-linear strain paths: state of the art and challenges

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Sheet metal forming processes are among the most important metal-working operations in automobile and aircraft industries. The industrial process of sheet metal forming strongly depends on numerous interactive variables: material behavior, lubrication, forming equipment, surface roughness of sheet and die, grain size of material, sheet thickness, etc. One of the main limitations in industrial stampings is the appearance of localized necking. Hence, it is necessary to determine the limit strains that can successfully be imposed upon the sheet metal during stretching. The limit strains determined from the biaxial tests are represented pictorially in the form of Forming Limit Diagram (FLD). The conventional strain based FLD proposed by Keeler and Backofen (1964, 1965) is widely used to predict the onset of localized necking failure in sheet metal during material deformation. FLD is evaluated by conducting Limiting Dome Height (LDH) tests such as Nakazima or Marciniak test which assumes linear strain path. However, in practice the material deformation in sheet metal forming processes is almost nonlinear. In the sheet metal forming industries, sheets are often deformed under various dies; hence they experience different strain paths. In operations such as deep drawing under complex loading conditions, the loading paths can deviate significantly from linearity. Furthermore, in multi-stage forming processes, the loading direction will change. Therefore, it is important to study the influence of strain paths on the FLD in various metallic alloys. Experimentally, the non linear strain paths can be generated by imposing uniaxial tension followed by equal biaxial stretching or equibiaxial stretching followed by uniaxial tension. For this purpose, some blanks are pre-strained by uniaxial tension in rolling direction (RD) and transverse direction (TD), and some are pre-strained by biaxial stretching over a hemispherical punch. The out-of-plane formability test is used for obtaining the FLD. The present study describes the experimental strategies involved in evaluation of FLCs considering non-linear strain paths and highlights the challenges involved in evaluation of FLCs experimentally.

Development of an automatic cut to length system for the improvement of productivity at shearing line of Rolling Mills

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The coils from HSM of BSL are cut in the range of 4.5 to 10 meters at Shearing Line of HRCF shop. These cut sheets are stacked in a piler, which are finally packed and despatched to customer as a consignment. The previous sheet shearing operation was based on traditional servo roll, encoder and dedicated hardwired controller for measuring desired length on running sheet and initiating the cut command accordingly. The system was very old and outdated. There were several issues in maintenance and

operation of the system such as mis-cut, length variation in first and the last cut etc. All these operational abnormalities led to rejection of sheets and also jamming in the piler, leading to downtime of Shearing Line.In view of above, an automatic cut-to-length system is designed, developed and implemented successfully at Shearing Lline-1 of HRCF, BSL. This is based on state-of- the-art Laser Doppler Velocimeter (LDV) as non-contact speed sensor for accurate calculation of length, and PLC for control and introducing various interlocks. In addition to above, tail end sensing and displaying cut length are also achieved. The implemented system has been interfaced with existing flying shear drive and motor for shearing operation. The system completely eliminates backlash and length variation associated with earlier system. The new system is in continuous operation since commissioned. Accurate cut lengthwithin +0.65% of set length value has been achieved as against a variation of 1% earlier, resulting in yield improvement. Further, implementation of the reliable and maintenance friendly control system has enabled reduction in downtime by 70%.

Performance of TMT and tor steel bar in construction work at hill and coastal region

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TMT (Thermo-mechanically treated), Tor steel bars are evaluated for use in flood affected hill region and coastal areas. There is huge loss of material and money due to breakage of building, bridges and flyovers. For natural calamity such as cloud burst, land slides and earth quake material loss are enormous. Hence these research work was undertaken to evaluate performance of SAIL TMT bar in construction work.TMT bars (PC, Cu and Cu-Cr Grades) 16 and 20 mm diameter were produced in steel plant Tor steels (PC) of 16 and 20 mm diameter were also procured from out side to evaluate their performance at virgin stage and also in the form of concrete blocks and beams. Mechanical and corrosive properties of 16 and 20 mm diameter TMT and Tor Steel bars and concrete reinforcement beam were evaluated at RDCIS, Durgapur steel plant, SE RC, Madras and FITT, New Delhi.TMT rebars (YS: 500 and 550 MPa) have a ductile ferrite-pearlite core and a tough tempered Martensite rim whereas Tor steel has ferrite pearlite Microstructure. Basic design of the concrete and structural column and beam along with size and quality of stone chips, amount of cement and its grade, size and quality of steel bars along with quantity of water to be used etc. was standardized in the laboratory before use at site. The mechanical properties meet requirement of standard (IS:1786-1985) concrete reinforcement cubes (150 X 150 mm) and beams (150 X 400 X 3000 mm) were cast and cured for at least 28 days before test.Fatigue test of reinforcement bars and concrete beams were performed at 10H_z frequency, slip test and static flexure test of concrete beam (150 X 40 X 300 mm) were also were tested in computer controlled servo hydraulic dynamic testing machine.RCC beams of both TMT and Tor steels during constant amplitude fatigue test passed 2 X 10 6 cycles.Corrosion rate decreases in the following order: Tor steel, PC TMT. Cu TMT and Cu-Cr TMT bars.Cr-Cu TMT bars are more suitable both in coastal and flooded hill areas. TMT bars have in general more seismic (earth quake) resistance than Tor steel. The above results are compared and suitability of TMT bar for use in costal and hill region are evaluated.

A review on: efficient energy optimization in reheating furnaces

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In Iron and Steel Industry Semi-finished products from blooming-billet/slabbing mills and continuous casting shop such as slabs, blooms & billets are reheated in reheating furnace to a temperature of 1260°C to 1280°C,using mixed gas (Coke Oven gas & Blast Furnace gas/BOF gas) having calorific value around 2400 kcal/m^3 along with hot combustion air for plastic deformation in rolling. During the process reheating of the semi-finished products, scale formation (metal loss) takes place and it depends on the variation of the time -temperature cycle with rolling rate and mill delays, presence of CO2, SO2, moisture and unburnt oxygen etc in the flue gas. The aim of this paper is to analyze the possibilities for energy efficiency improvements through utilization of measurement and automatic control; this includes both direct fuel savings and indirect savings due to product quality improvements. Focus is on energy use in steel reheating furnaces for rolling mills. The demands on the reheating process and the operational conditions that are essential for its control are described. There is another area for reducing energy consumption is by waste heat recovery from flue gases and the reduction in specific fuel consumption/ton of finished products, scale losses during reheating and rolling process and the advantages of walking beam furnace over pusher type reheating furnaces. The paper also deals the control of surface oxidation by improving the combustion efficiency, controlling temperature for minimizing fuel consumption and better utilization of energy in terms of specific fuel consumption/ton of finished steel.

Effect of alloying on mechanical properties and microstructure of cryo-deformed brass

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In the present study, we have reported the effect of zinc content on the mechanical properties of copper after cryorolling at liquid N2 temperature. The cast structures of Cu-3.84wt.% Zn and Cu-9.63wt.% Zn were homogenized by annealing at 800˚C for 4 h. The annealed samples of 8mmX24mmX72mm were rolled under LN2 temperature to obtain 50, 75 and 90% reduction in area (RA). The samples were kept under LN2 before and during the rolling process in each pass to maintain a constant LN2 temperature. Mechanical properties were evaluated by Vickers hardness measurements and tensile tests. The tensile tests were carried out as per ASTM E8 standard with 12.5 mm gauge length specimens at a constant strain rate of 5×10-4 s-^{1}. Tensile yield strength of the annealed samples increases with increase in the Zn content, and the same trend is observed for the cryorolled samples. The maximum tensile strength was obtained for the 90% cryorolled specimens for all the compositions. The maximum yield strength (YS) of the 90% rolled samples of Cu-9.63wt.% Zn alloy was found to be 672 MPa with a very low ductility of 3.6%. The YS is 165% higher compared to that of the annealed counterpart (254 MPa). A short time low temperatures annealing (20 min, 225-300°C) of the 90% rolled specimens showed an improved ductility with minor decrease in the yield strength. Microstructural analysis was carried by optical microscopy, X-ray diffraction (XRD), SEM and TEM analysis. Short-time annealing after cryorolling found to produce ultrafine equiaxed grained brass in contrast to the coarse grained annealed samples. Fractography analysis of the 90% cryrolled samples showed a brittle fracture after tensile test. The presence of dimples marks on the fractured surface of the

short-time annealed samples indicates a ductile failure. Increasing Zn content in the alloy decreases the stacking fault energy (SFE) of Cu. Thus, there is an opportunity to increase the plastic deformation by twining mechanisms rather than by slip. The dynamic recovery and recrystallization can effectively be prevented in the low SFE alloy during deformation at low temperature. This restricts cross slip to occur thereby increases dislocation density during the cryorolling process. Finally, the ultrafine grained brass can be prepared by short-time annealing of the cryorolled samples. Thus, formation of ultrafine grains and nanotwins possibly play the pivotal role for the improved YS and ductility.

METALLURGICAL HERITAGE OF INDIA

Invited talks

Traditional Iron Making Practice of the Agariyas

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Indigenous iron and steel technology in India has a long history. Recent archeological finds push the date for established iron making practice to about 1800 BCE. The Delhi pillar, of course, was the celebrated high point of the technology in the first millennium of the present era. Historical and archeological records however abound for periods before and after. The iron for these master pieces, including that for making the famous Wootz, seemingly came out of unassuming mud furnaces. These furnaces were operated, probably in an unbroken tradition, for making agricultural tools, till as late as the last decade of the 20th century, by the Adivasis of Chhotanagpur (the contiguous area between Madhya Pradesh, Chhattisgarh, Jharkhand and Orissa). These people, broadly called as the Agariyas by Verrier Elwyn, identified themselves with this technology and probably therefore kept the technology intact over the ages. The process is conceptually different from the modern iron making processes; the temperature has to be such that the slag melts but the iron does not. Trying to understand the technology was therefore an interesting challenge due to its historical context. Equally important was the modern metallurgists' encounter with a traditional technology developed from a different knowledge base. This talk aims to explain the efforts made to document the existing practice, mainly in the Agariya belt of Central India. Efforts were also made to study some of the metallurgical aspects at IIT Bombay. The experience has indeed been rewarding: one could get some fresh insights into the modern process, and it has led to new avenues for research.

Copper/Bronze and Iron Age Cultures of India

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The settled life began in India around 7000 BCE in the Northwestern and Northern India. Most of the basic technologies were developed in the beginning of settled life. The concept of pottery came into existence around 6500 BCE and the copper technology was developed around 6000 BCE. There was a gradual growth in these technologies and they were perfected in the middle of the third millennium BCE. Since then there has been continuous use of these technologies till the modern times. Different cultural traditions were developed in different parts of the country during the copper age period. In the Northwest and Western part of the country the cultural development which began around 7000 BCE culminated into the formation of the Harappan Civilization. The Harappans were the first in the Indian subcontinent to develop bronze technology. The Harappan Civilization developed urbanization, leading to the formation of cities and towns with provisions of civic amenities and water harvesting facilities at major cities. Contemporary to the Harappans were Ahar culture in Rajasthan, Neolithic/Copper Age in the Ganga plain, Malwa in Madhya Pradesh, Jorwe in Maharashtra and Neolithic farmers in South India. The characteristic features of the copper age cultures of India include the presence of circular or rectangular mud houses of wattle-and-daub, handmade and wheel made painted and pain pottery, restricted use of copper, presence of stone

blade tools, stone beads and a variety of terracotta objects. The main source for the Copper Age cultures of India was the Khetri in Rajasthan, which was traded almost all over the country.The Early Iron Age culture developed around 1500 BCE simultaneously in North and South India. The Iron Age culture that flourished in North India is called Painted Grey Ware Culture whereas the one flourished in South India is termed as Megalithic culture. The life of the people of the Painted Grey Ware culture was same as that of the Copper Age cultures of India, except that the later developed iron technology. The Megalithic people in South India, including the Vidarbh part of Maharashtra, lead simple rural life, but built circular burials, on the periphery of which were placed large stone boulders. The dead body was buried sometimes with horse, pottery, tools and ornaments, which indicates that the people believed in life after death.This presentation will take review of the Copper-Iron Age cultures of India.

Survey of iron and wootz steel production sites northern telangana

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Iron mining, smelting and manufacturing of wootz steel was very brisk, in many villages of Adilabad, Karimnagar, Nizamabad and Warangal districts in Northern Telangana, since ancient time. In these works, several thousands of people employed, in various activities. These included mining, smelting, processing of iron and wootz steel, manufacturing and production of arms, armour and iron implements. These products catered to the demand of the local buyers and external traders. Indur, Nirmal, Konasamudram, Elgandal and Metpally mentioned by several medieval travellers and particularly in medieval Persian literature. This region is in present Andhra Pradesh state. The deposits of iron ore found in several places until this day. Iron ore mining, smelting and manufacturing of wootz steel was in vogue. The village names in Telugu language etymologically identified with iron and iron related works. They noted in medieval literature and mentioned by the travelers. There are good numbers of villages not identified yet. An extensive survey conducted in 2600 villages, recorded the information and ample evidences collected regarding iron smelting and wootz processing in more than Four hundred and twenty five villages. Remains of iron smelting furnaces seen in the deep forests of Adilabad. Slag remains, crucible remains and wootz implements collected from four districts. The findings of the survey in iron and wootz production sites presented. The crud local methods in iron smelting and wootz production recorded on basis of the old smiths, who revealed their memory. Large numbers of artifacts from different sites have been collected and they need further laboratory analysis. The standing testimonies for the iron and wootz manufacture are the heaps of slag remains, existing in hundreds and thousands of acres in the region. The extensive use of wootz steel implements can be seen till this day by the different occupational groups in this region.

Contributory papers

Session 1: Nov 14, 2014: [08.30- 10.30]

Archaeometallurgical studies of ancient sword

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Metals played a significant role in society throughout the ages, their use strongly influencing the course of civilization and human history. The ancient people observed that the properties of metals could be seriously improved by alloying or mixing them with other elements. Through metallurgical procedures, melting points can be lowered, leading to final products of enhanced hardness; other characteristics, such as strength, workability and resistance to corrosion, can be similarly improved. A very old artifact found from the remains of Kondapalli Quilla (fort) area, was experimented to understand the possible way of manufacture of ancient Indian tools and the craft of the blacksmiths. Various observations, of microstructures and strengths were taken and have been compared with the archaeometallurgical studies and the possible reasons for the differences in the patterns or structures observed, with present day tools have been tried to be identified and studied. With the advent of the carburization of iron, a special type of high carbon steel was produced in India from as early as the fourth century BCE. This steel was named as wootz steel and it was much prized by warriors because tough swords could be wrought from wootz steel. The possible resemblances have been tried to be recognized.

Microstructural characterization of three ancient wootz steel implements: A comparative study

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This presentation describes our efforts at comparative characterization of three different ancient wootz steel implements collected from Dharmapuri in Karimnagar district of Telangana by Dr. Jaikishan in his field survey on Archaeo-metallurgical study i.e. Iron and wootz steel making and smelting centres in Telangana districts between 2005-2010. The implements, namely, a sickle, a sword, and a chisel have been manufactured, supposedly from wootz steel that has been made by a process adopted by the local artisans for centuries. Wootz steel is essentially a hyper-eutectoid high carbon steel and normally contains between 1.2 to 1.8 wt.% C. The main objective of the present study was to characterize the microstructure of the three implements to find out differences as well as similarities in the presence, morphology and spatial distribution of the various constituent phases. A wootz steel ingot, recovered in-tact from a small crucible, was also characterized to serve as the reference. Chemical composition analysis of theimplements revealed that the ingot, sword and chisel had carbon content typical of wootz steel, but the sickle contained only 0.6 wt% C, making it the only sample with hypo-eutectoid carbon content. This was expected to reflect in interesting differences in the microstructure between the different implements. Basic microstructural characterization carried out using optical microscopy and SEM, revealed ferrite and cementite to be the predominant phases. However, the morphology and distribution of the two phases was found to vary within and amongst the different samples. Investigation of micro-texture and phase analysis was carried out using electron back-scattered diffraction (EBSD). ThermoCalc^{TM} calculations were employed to predict equilibrium phases and their compositions as a function of temperature, and attempts were made to compare these with experimental results to better understand the microstructure. The paper will present and describe salient highlights from the work described above.

Understanding ancient iron technology from the vidarbhan megalithic sites through archaeo-metallurgic and ethnographic approaches

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Naikund datable between 700 B.C – 555 +/- 100 B.C, has yielded the earliest evidence for smelting and smithery practices of the early Iron Age Megalithic Society within the Vidarbha region, though its artefacts are limited in variety. This may be due to the fact that Naikund functioned as a centre of production and distribution of iron objects. The aforesaid hypothesis needs verification. For this, a typo-technological analyses followed by an ethnographic survey was carried out. Typological analysis of a variety of artefacts from Naikund and its surrounding regions revealed that the production at Naikund followed a degree of standardisation. The methods of study employed to characterize the materials include wet chemical analysis, optical microscopy and scanning electron microscopy. The comparisons of results from Naikund and the other regions suggest that the raw materials used were from within the surrounding vicinity. The presence of high carbon (1.06%) in the objects indicates that there was a deliberate attempt to steeling. This can be considered as the earliest example of steel production within the sub-continent. To reconstruct the mechanism of production and distribution of objects from Naikund, ethnographic surveys were carried out among the present day traditional iron smiths of Vidarbha. This exercise revealed carbon is deliberately added here while smithery process is done. It is also noticed that most of these traditional industries have lost the knowledge of procuring ores from a natural deposit and therefore are dependent on other agencies for raw materials. This paper reconstructs the metallurgical activities at Vidarbha by integrating the scientific analyses data with the ethnographic data.

Invited talks

Session 1: Nov 14, 2014: [08.30 - 10.30]

Graphene Coating and Nanocrystalline Alloy Structure: Novel Approaches for Remarkable Corrosion Resistance

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This talk will present two novel approaches for mitigation of corrosion. The associated research has also involved considerable degree of surface analysis.

Ultra-thin Graphene Layer for Corrosion Resistance

Graphene research was awarded Nobel Prize in 2010. A monolayer or a few atomic layer thick graphene coatings on metals have shown to improve their corrosion resistance by nearly orders of magnitude. Though there are very few studies reported on the topic of corrosion resistance due to graphene coating, there is still considerable variability in the degree of improvement. For example, improvement in aqueous corrosion resistance of copper due to graphene coating is reported to vary from insignificant to nearly 2 orders of magnitude, whereas the improvement for nickel can be in excess of an order of magnitude. This presentation will review the most recent research on graphene that has been claimed as 'the thinnest known corrosion-protecting coating', and potential application of such disruptive approach to corrosion resistance of steels.

Nanocrystalline Structure for Remarkable Oxidation Resistance

The second part will demonstrate remarkable resistance to oxidation as result of the nanocrystalline alloy structure. This will include an elaborate description of the author's own hypothesis that nanocrystalline structure can impart extraordinary oxidation resistance, and the validation of this hypothesis. A thorough surface/subsurface characterization of oxidized alloys, using secondary ion mass spectrometry has provided a sound mechanistic understanding of the remarkable improvement in oxidation as result of nanocrystalline structure. The data to be presented will include the results establishing that a Fe-Cr nanocrystalline alloy with only 10 wt% Cr can provide as much oxidation resistance as a Fe-20Cr alloy, suggesting possibility of Fe-Cr alloys with the necessary corrosion resistance at much lower Cr contents. As another exciting potential application of this work, the nanocrystalline powders of Fe-Cr alloys synthesized in this study could be used for developing corrosion resistance coating having considerably low Cr contents.

Session 2: Nov 15, 2014: [11.00 - 13.00]

New approaches for the preparation of nanoparticle dispersions and superlattices

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During the past few years our group at NCL has been working on the synthesis of metal nanoparticle dispersions in different media. It is now well documented that some of the potential applications of the nanoscale particles requires them to be as monodisperse as possible. Thus there is lot of interest in controlling the shape and size of these nanoparticles and there are many studies towards achieving these goals. In the colloidal regime it is well documented large particles grow at the expense of smaller ones and this process is commonly known as 'Ostwald ripening'. In spite of the thermodynamic stability associated with bigger particles, many synthetic protocols have been developed that could provide very small NPs with narrow size distribution. In literature, these are being described with different names, such as size focusing, digestive ripening or inverse Ostwald ripening. At NCL, we have been working on the process termed as "digestive ripening" in which a colloidal suspension in a solvent is refluxed at or above the solvent boiling temperature in the presence of the surface active agent results in the conversion of a highly polydisperse colloid into a nearly monodisperse one ($\sigma < 5\%$). Digestive ripening has now been established as a very convenient route to obtain monodisperse nanoparticles from polydisperse ones by refluxing the latter in the presence of an excess ligand. Many ligands including long chain thiols, amines, or phosphines have been shown to be effective digestive ripening agents. It is hypothesized that the surface active groups of such digestive ripening agents bind and remove reactive surface atoms/clusters from big nanoparticles and redeposit them on smaller nanoparticles. In this way, large particles become smaller, while small particles become larger and eventually, an equilibrium size is obtained that is specific to each of the digestive ripening agent used. We will also present some studies wherein particles with narrow size distributions tend to self-organize into exciting crystal structures (superlattices). Very recently we have shown - by undertaking a systematic investigation by varying the time and temperature of the refluxing step- that the trends in the final particle size distributions suggest a cross over from digestive ripening to Ostwald ripening process.

Contributory papers

Session 1: Nov 14, 2014: [08.30 - 10.30]

Effect of boron addition on grain size stability and hardness in nanocrystalline Cu-Al alloys

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Nanocrystalline Cu-14Al and Cu-12Al-2B alloys has been synthesized by mechanical alloying through cryogenic high energy ball milling. Present study concerns assessment of grain size stability at elevated temperatures by substituting B in place of Al, in Cu-Al alloys. The as-milled alloys were subjected to annealing at various temperatures between 200-800˚C. The resulting grain morphology has been studied in detail using X-ray diffraction and transmission electron microscopy. The addition of B significantly reduced susceptibility to grain growth compared to CuAl alloys. The hardness is substantially increased due to B addition in the as-milled CuAl powders. Accordingly, the efficacy of grain size stabilization by B addition at high homologous temperatures has been explained on the basis of a recent thermodynamic stabilization models.

On densification mechanisms during spark plasma sintering of nanocrystalline B2 aluminides

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In the present study, nanocrystalline FeAl and NiAl powders were synthesized using high energy ball milling.Mechanically alloyed powders were consolidated using spark plasma sintering (SPS) in the temperature range of 750 to 900°C and stress range of 25 to 100 MPa. The densification mechanisms during SPS were studied based on the analytical model for creep deformation studies in the final stage of densification. The values of stress exponent and activation energies obtained using this analysis are compared with those obtained using conventional compressive creep experiments conducted on dense SPS pellet. The detailed characterization of mechanical alloyed powders and dense SPS pellets were carried out using XRD, SEM and TEM.

Grain growth kinetics of austenitic ODS steel

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In the current scenario of conserving and producing more energy, there is a lot of scope for the energy derived from nuclear reactors. For fast breeder reactors, fuel pins are of major concern and these pins, at their service temperature at about 500-800°C, undergo void swelling. Due to void swelling, pins undergo change in shape, volume and cannot be used for a longer period. In present work, austenitic steel (SS316L) have been chosen as a candidate material for FBR fuel pins as they have higher creep strength and oxidation resistance. But a major drawback of austenitic steels is their poor swelling resistance than the ferritic steels. This limitation can be overcome with the usage of dispersoids by means of oxide dispersion strengthening (ODS). In this work, SS316L with varying percentage of yttria (0.25, 0.5 wt %) and titanium (0.4 %) are studied in terms of their grain growth behavior at various temperatures from 500-800°C for different holding times from 30 min to 4 hours to know their thermal stability. From the kinetic study, the grain growth exponent (n) and activation energy (Q) is determined. The n values for SS, SS-0.5Y_2O_3-0.4Ti are 0.2369 and 0.0728, respectively. The lower the n value, higher is the resistance for grain growth. The grain growth of alloy with dispersoids is reduced due to the Zener pining of grain boundary by the

dispersoids. The activation energy of SS, SS-0.25Y_2O_3-0.4Ti and SS-0.5Y_2O_3-0.4Ti is 243, 485 and 496 kJ/mol, respectively. The activation energy increases due to the presence of nano scale dispersoids. The formation of Y-Ti-O complex oxide helps in reducing the swelling problem and hinders grain growth. The study of grain growth kinetics is important in selecting correct alloy composition that has lesser grain growth at its service temperatures, for irradiation studies.

Evolution and interaction of defects in nanostructured α-brass processed through cryorolling

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Bulk nanostructured materials have been successfully synthesized by severe plastic deformation (SPD) processes. Recently cryorolling has been employed to produce nanocrystalline Cu and its alloys. Severe rolling at cryogenic temperature helps in accumulating high defect density, thus restricts dynamic recovery and refines the overall microstructure. The grain refinement in bulk solid with low stacking fault energy involves formation of twin and their fragmentation by high-density dislocation flux that generate during severe deformation. Therefore, microstructural refinement in α-brass during severe rolling proceeds by extensive twinning due to its low stacking fault energy (14 mJ/m^2). Moreover, with the increase in rolling strain both the twin lamellae thickness (λ) and twin spacing (d_{twin}) decreases. A high-density dislocation flux interacts with TBs at higher rolling strain and dissociate into the twin lamellae into equaxed structure. Therefore, formation and subsequent fragmentation of twin lamellae refines the microstructure in α-brass during cryorolling.The present work explores the micromechanism of grain refinement in α−brass (Cu–30 wt.% Zn) during cryorolling (CR). The evolution of structural defects and their interaction have been evaluated through x-ray analysis, transmission electron microscopy, resistivity, positron lifetime and doppler broadening measurements. The twin lamellae width of α−brass has been refined down to <40 nm and the refinement saturates at $\varepsilon_{CR} \geq 0.9$. The lattice strain increases up to 2.1×10^{-3} at $\varepsilon_{CR} = 0.6$, and remains constant upon further rolling. The variation of twin density (β), their interaction, the evolution of other defects such as stacking faults and dislocations density on extensive grain refinement in α-brass (Cu–30 wt.% Zn) during cryorolling will be discussed in detail. The authors thank P. Das, K. Sahoo, R. Basu, and S. Bhattacharya for technical assistance. Financial support provided by DST, SERB, Govt. of India for the project entitled “Processing and characterization of bulk nanostructured brass” is gratefully acknowledged.

Face centered cubic titanium in Ti/Al multilayer thin films: not an artifact of sample preparation

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The present investigation is the first comprehensive study of high-resolution XRD to address the important issue of the fcc-Ti formation in Ti/Al multilayer thin films. Ti/Al multilayer thin films with precise thickness have been deposited using a combination of dc and rf magnetron sputtering technique. Increasing the power of magnetron guns alter the rate of deposition, which in turn enhanced the formation of fcc-titanium phase in the titanium layers. However SIMS and X-TEM studies exhibited the formation of fcc-Ti that was rejected by a group of researchers. Further to this, they suggested that this should be established

with the help of high resolution XRD nondestructive technique. The basis behind this was, the stress acting on the multilayer thin films could induce lattice defects, which in turn covert the hcp to fcc phase (stacking fault). The synchrotron xrd experiments exhibited the fcc-Ti phase at different **ψ** rotations. In this report, we are going to explain the formation of fcc-Ti on glass substrate and show the presence of fcc-Ti using high-resolution xrd.[2]

Phase evolution in $Al_xCoCrFeNi$ (x= 0, 0.3, 0.6, 1 mole) high entropy alloys synthesized by mechanical alloying and spark plasma sintering

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$Al_xCoCrFeNi$ (x=0,0.3,0.6,1 mole) high entropy alloys with multiprincipal elements and varying Alcontent were synthesizedthrough mechanical alloying. Elemental blends of CoCrFeNi, $Al_{0.3}CoCrFeNi$, $Al_{0.6}CoCrFeNi$ and AlCoCrFeNi powders were subjected to high energy ball milling in Fritsch Pulverisette P-5 until the formation of solid solution. The as-milled HEA powders having grain sizesof around 10 nm were consolidated by spark plasma sintering to obtain pellets of very high relative density without significant grain growth. Characterization of milled powders and consolidated pellets has been done byX ray diffraction, scanning electron microscopy, transmission electron microscopy and differential scanning calorimetry. The quaternary alloy showed an FCC phase, while the quinary alloy showed a BCC phase, indicating the stabilization of BCC phase with the addition of Al. Variation in Vickers hardness between the sintered sampleswascorrelated to the presence of FCC and/or BCC phases present after SPS. Phase and microstructural evolution during mechanical alloying and after spark plasma sintering has been investigated and the differences have been correlated with the Al content of the alloy.

Synthesis of nano-structured aluminum 2024 alloy by milling route

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Nanostructured (ns) Al-2024 was prepared via attritor milling in wet medium under protective argon atmosphere, followed by vacuum hot pressing. The milled powders were characterized by differential scanning calorimetry (DSC), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The crystallite size of Al-2024 powders was 25 nm at around 4h of milling, as calculated from the XRD patterns. Formation of a super saturated solid solution was observed upon milling, which resulted in nanosized precipitatates of the intermetallic Al2Cu and Al2CuMg phases upon thermal treatment. The consolidation of the powders was done via vacuum hot pressing. Through this method samples almost fully consolidated were obtained in only 15 min at 500ºC and 50MPa, preserving the nanometric scale of the grains as indicated by elevated hardness values.

Synthesis and characterization of multi-component alloy

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The AlMgFeNiCuCr based nanocrystalline multicomponent alloy was synthesized by mechanical alloying. The microstructure, hardness and thermal properties of the AlMgFeNiCuCrHE alloy were studied by utilizing XRD, SEM with EDS, Vickers hardness tester and thermo-gravimetric analyzer. XRD analysis of powder has confirmed that the alloy is basically composed of FCC phase with minor BCC phase. Phase evolution as a result of sintering is discussed. Based on the present study structure-process-propertiescorrelation of AlMgFeNiCuCr alloy is critically addressed.

Synthesis of nanocrystalline complex metallic alloys by mechanical milling

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The synthesis of nanocrystalline phases in complex metallic alloys appears to be beneficial in order to overcome the brittleness problem of these alloys. Mechanical milling among the various processing techniques has been adopted for the purpose of synthesizing nano-phase/amorphous and their composites. The aim of the present work is to investigate on some Al-based complex metallic alloys/quasicrystalline alloy and process their microstructure through mechanical milling. The milling is carried out in a high-energy mill at various milling intensity with varying the ball to powder ratio in a suitable milling medium. The milled and the annealed samples are characterized with the help of DTA, TGA, XRD, SEM, EDX and TEM. It is found that these alloys can give rise to the formation of nanocrystalline phases of grain sizes to a certain minimum value. At present, it is not clear why, given a milling energy, some alloy systems restrict to the formation of nanograin of a particular size and do not lead to the formation of amorphous phase. Attempts will be made to discuss the micromechanisms for the formation of nano phases.

Development of environment friendly new generation Mgo-C brick using nano carbon

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The basic oxygen steelmaking (BOF/BOS) is an overwhelmingly popular process for steelmaking and MgO-C refractories are widely used as working lining for BOF vessel. Combination of graphite with MgO increases resistance to thermal shock, controls thermal expansion, improves corrosion resistance, and

decreases wettability with corrosive liquid phases. On the other hand, due to the increased demand for high quality steel such as API steel or ultra- low carbon steel, the carbon content in the MgO-C bricks have greater importance. The problems with conventional MgO-C brick are carbon pick up by steel and increase in heat loss, which has resulted in demand for low carbon MgO-C bricks. Reducing the carbon content, however, may decrease the thermal spalling resistance, which is contributed by carbon's high thermal conductivity and low thermal expansion. The thermal spalling resistance of the said brick can be improved either by adjusting the particle size distribution or by additives. In this study we investigate the role of Nano Carbon for the development of low carbon MgO-C brick with better properties. This review paper comprises of the study in which amount of Nano-C was varied from 0.3 % to 1.5 % along with pitch and liquid resin as other source of carbon. The results of the samples prepared were compared with the conventionally prepared MgO-C (10%) bricks. Finally the suitable anti-oxidant and the amount of anti-oxidants were fixed by a no. of experiments has been discussed.

Development of nano carbon coated water wettable graphite by applying nano technology and its application in refractory

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Carbon coated graphite with high resin and water wettability characteristics could expand the refractory and carbon–carbon composites application in different fields.Graphite is a flaky non wetting material widely used for making carbon containing refractory for relining of steel making vessels. Making of carbon containing castable is not possible without making graphite water wettable so that it can be distributed homogeneously in the body.In the present work, graphite was made water wettable by coating nano carbon above the surface of graphite. Nano sized carbon is a form of para crystalline carbon that have a very high surface area (80- 120 m^2/g) and particle size is of 15 – 280 nm. The structure of this carbon is similar to graphite which was confirmed by X-ray diffraction. An optimum colloidal solution was prepared in which nano carbon was dispersed by using some surfactant and dispersant. The coated graphite has the same characteristics as like natural graphite which was confirmed by the X-ray diffraction. Scanning Electron microscopy of the coated graphite also confirmed the coating of nano material above it. The High Resolution Transmission Electron Microscopy of the nano coated material has shown presence of graphene type structure of the coating. The nano carbon coated water/resin wettable graphite has been successfully used in preparation of refractory castable and block samples.

Effect of rare earth and transition metal elements on the glass forming ability of mechanical alloyed Al-TM-RE based amorphous alloy

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Bulk metallic glasses (BMGs) exhibit excellent mechanical and good corrosion resistance owing to the absence of long-range atomic periodicity, and defects. In this context, aluminum-based metallic glasses with high Al content (Aluminum - transition metal - rare earth element, with Al>80%) are drawing much attention owing to their superior specific strength and corrosion-resistant properties. However, synthesis

of these alloys is extremely difficult due to low glass forming ability (GFA) of this alloy system. The present work aims to compare the amorphous phase forming ability of ternary and quaternary Al based systems (Al86Ni8Y6, Al86Ni8La6, Al86Ni6Y6Co2 and Al86Ni8Y4.5La1.5) synthesized via mechanical alloying by varying the composition, i.e. fully or partially replacing rare earth (RE) and transition metal (TM) elements with either varying atomic radii, or similar coordination number solutes. The GFA of the alloys has been investigated with respect to the Al-X bond distance or atomic size ratio, coordination number and enthalpy of mixing of each binary system. X-ray diffraction study revealed complete amorphization of the alloy powders in between 100 to 150 hr of ball milling. However, addition of lanthanum resulted in lesser degree of amorphization attributed to higher atomic radius and higher enthalpy of mixing of La with Al. Transmission electron microscopy along with selected area diffraction exhibited incomplete amorphization with partially insoluble La distributed as nanoparticles in the amorphous matrix. Degree of amorphization was correlated with strain hardening and consequent fracturing of the powder particles during milling leading to decrease in particle size. The glass transition and the multistage transition temperature of the alloys were characterized by DSC which showed four-stage transition in case of Al86Ni8Y6, while all other composition showed two-stage phase transition.

Session 2: Nov 15, 2014: [11.00 - 13.00]

Modified CdSe nanowires, radially grown on TiO_2 nanowires, for realization of "Rainbow" solar cell

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Achievement of low-cost-high-efficiency photovoltaics has become possible due to use of Quantum Dots (QDs) as photosensitizers due to their lucrative properties of size tunable band gap energyand composition control, high molar extinction coefficient and large intrinsic dipole moments for enhanced charge separation. Recent efforts has seen introduction of recombination barrier layer between TiO_2 and QDs, protective layer between QDs and electrolyte for enhancing photovoltage, grafting molecular dipoles between QDs and a nanometric protective layer, to facilitate electron injection without suffering loss in open circuit voltage (V_{oc}). Fabrication of a "Rainbow" solar cell consisting of QDs stacked inside a TNT to absorb visible light was proposed recently, indicating that stacking QDs of different sizes shall allow maximum visible light absorbance thereby placing us in the vicinity of theoretically reachable quantum efficiencies. However, nanotubes of wide bandgap host oxides have dimension typically 10^3 times higher compared to the size of the QDs. Thus, the proposed architecture in this form (vertical stacking) is not practical due to the disparity in the scale of the deposited QDs and the host oxide nanowire. Radial stacking is a theoretically feasible idea, but not easy to achieve experimentally. Multi segmented semiconductor nanorods/nanowires with coupled electronic properties are reported and can be prepared by sequential VLS/SLS growth or ion exchange methods. Radial growth of these 1D nanostructures on oxide nanotubes can alleviate the problem of QD stacking and make a large stride towards "Rainbow" solar cell. In the present work, we use Bi nanoparticles as nucleation sites for CdSe nanowires as well as segmented CdS/CdSe nanowires with photoelectrochemical properties better than CdS or CdSe alone. Also, there are many modifications which can be made to the nanowires to improve their absorption energy range.

Synthesis of graphene oxide nanoparticles and its application in composite and blend cellulose acetate membranes

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Cellulose acetate (CA) and its derivatives are commonly used materials for synthesizing membranes. However, CA membranes interact with hydrophobic groups leading to organic and biofouling. Thus, modification of CA gains importance. Recently, nanomaterials are playing an important role as hydrophilic additives in membranes due to the significant improvement of membrane permeability, antifouling property and mechanical property. Moreover, unlike water-soluble polymers, nanomaterials as the additive could stably exist in the prepared membrane. In the present work, we have synthesized graphene oxide (GO) nanoparticles using modified Hummer's method with KMnO4 as oxidizing agent and developed hydrophilized composite and blend membranes using CA and GO. GO increased the surface functional (e.g. epoxy, carbonyl, carboxyl) groups on membranes as found in FTIR graph. GO mixes readily with many polymers, forming nanocomposites and greatly improving the thermal, chemical and electrical properties of original polymers. The composite and blend membranes showed drastic increase in its pure water flux (PWF) from that of pure CA membrane of base composition with same thickness. In blend membranes, when the concentration of GO nanoparticles to the cellulose acetate increased from 0 to 0.5%, pure water permeate flux increase from 19.7 to 110 LMH/bar. The membranes were also characterized for their water contact angle and tensile strength. Additionally, membranes were used to clarify and de-colorize effluent containing primarily indigo dye obtained from Arvind Mills. GO is negatively charged on account of presence of carboxylate groups, hence, anionic dye molecules were repelled by the GO particles present in the membrane top layer. The filter cake formed was loosely attached to the surface of the membrane and can be easily cleaned. Therefore, these membranes may find potential applications in dye industry for recycling spent dye from the process or in effluent treatment.

Beneficiation of nano-metric zirconia for the development of low cement castable (LCC) refractory matrix

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Low Cement Castable (LCC) contains white tabular alumina (WTA) aggregate, high alumina cement bond material (5–7%), silica fume and micro fine alumina as mullite forming additives and nano-zirconia as special additives. LCC are used for the construction and repairing of high temperature furnaces used in iron and steel industry, cement industry, glass and petrochemical industry.In the present investigation, WTA (80% of the batch) is used as aggregate; SECAR–71 bond material (6%) and micro fine alumina (10%) are added in the batch. Micro fine alumina is replaced by nano-zirconia synthesized by sol-gel technique through ammonium and tri-ethyl amine route.Important properties like apparent porosity, bulk density, cold crushing strength, strength retainment after spalling tests are performed. Scanning Electron Microscopy (SEM) studies have been utilized to investigate the microstructure of the castable matrix. Crystalline phases are identified by X-Ray Diffraction (XRD) techniques, major phases identified are mullite

and corundum and minor phases are cristobalite and tetragonal zirconia. Fourier Transform Infra-red (FTIR) spectroscopy is used to detect the presence of nano-zirconia in the castable.It is observed that incorporation of nano-zirconia in the castable improves the properties like cold crushing strength, strength retainment after spalling and dense microstructure. It is interesting to mention that 6% nano zirconia addition in the castable batch improves the strength by attracting micro cracks at the grain boundary. So addition of 6% nano-zirconia is beneficial for Low Cement Castable.

Polycrystalline textured large grained silicon "seed layer" by aluminum induced crystallization (AIC) on steel for solar cell application

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Investigation of the aluminum-induced crystallization (AIC) of amorphous silicon (a-Si) during the aluminum-induced layer exchange (ALILE) process was carried out , in which a stack of steel/Al/a-Si is transformed into a steel/polycrystalline silicon (poly-Si)/Al (Si) structure by an annealing step well below the eutectic temperature (T_{eu} = 577 ^{o}C) of the Al/Si system. The nucleation and the growth of the crystalline phase during the ALILE process were observed using an optical microscope while optimizing the process parameters with quartz/Al/a-Si stack for the investigated Steel/SiN/TiN/Al/a-Si structure. The experiment resultedin continuous large grained polycrystalline silicon layer which could act as 'seed layer' for large area thin film devices. We also investigated the feasibility of plasma enhanced chemical vapour deposition (PECVD) deposited Silicon Nitride (SiN) thin film as diffusion barrier over steel substrate for Fe, Ni, C, Si atoms by employing X-ray photoelectron spectroscopy (XPS) in concentration depth profiling (CDP) mode. TiN layer served as the back electrode and also provided two additional functionalities, it firstly acted as Fe diffusion barrier & secondly provided high-temperature compatible non-contaminating substrate layer.

CNT based 3-dimensional structure as high current density field emitter

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High current density field emitters have been in focus of researchers due to its application in microwave amplifiers and satellite propulsion system. Variety of structures has been investigated till date to achieve high current density. Carbon nanotubes (CNT) are known as promising candidate for next-generation field emitters. One of the ways to achieve high current density could be to enhance CNT density, while avoiding screening effect. However, in a conventional plate-type (2-dimensional) field emitter, it is extremely difficult to increase spatial density of CNTs without causing screening effect. 3-Dimensional architecture has the advantage of enhanced surface area so that more surface area is available for CNTs growth. Cupric oxide (CuO) nanotubes array and CuO nanorods were grown on copper substrate by simple chemical

method. The surface area of these nanostructures was measured to be much higher than the corresponding 2-dimensional structure. A comparative field emission study between 2-Dimensional structures, i.e. CNTs on copper foil and 3-Dimensional structures, i.e CNTs on CuO nanotubes and CuO nanorods, clearly demonstrate achievement of higher emission current density in 3-diemnsional field emitter.

Synthesis and characterization of silica nanoparticles

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Silica nano-particles were synthesized by Stober method (sol gel) using tetraethylorthosilicate (TEOS), ethanol (C2H5OH) and water in the presence of ammonia as catalyst at room temperature. The morphology and structure of colloidal silica particles formed depend on the molar ratio of reagents. The size dispersity of particles was found to depend on stirring and the mean size could be controlled by concentration of reactants. The nature and morphology of particles was investigated by transmission electron microscopy (TEM). Particle size distribution was characterized by DLS. It is expected that monodisperse particles may lead to sintered bodies with high density with superior transmission characteristics.

Synthesis and characterization of ZnO nanoparticle using electrodeposition method

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ZnO is a wide band-gap (3.37 eV) II–VI compound semiconductor with hexagonal wurtzite structure. Higher exciton binding energy of zinc oxide (~60 meV) enhances its luminescence efficiency. It is an n- type semiconductor material having several applications in optoelectronics, transparent electronics, spintronics, and sensor applications and widely used for the fabrication of transistors and FETs, light emitting diodes, dye sensitized quantum dot solar cells. In this work we have synthesized zinc oxide thin films using electrodeposition technique because of easy control of the thickness, simplicity, low equipment cost and the possibility of making large - area thin films. The electrodeposition was carried out using zinc plate as anode and cathode. In 40 ml of distilled water 0.1M sodium citrate and 30% hydrogen peroxide are introduced. The pH of the electrolyte was maintained 9-10 by introducing ammonium solution. The deposition was carried out at 4V at room temperature. The duration of the deposits was 15 minutes. The as-deposited films were annealed in air at 400°C.The solid state properties of as-deposited ZnO thin films were studied by X-ray diffraction (XRD), Field emission scanning electron microscopy (FESEM), Energy-dispersive X-ray spectroscopy (EDX) etc. The structural analysis clearly indicates the diffracted prominent peak at (101) plane. The average grain size using the Debye- Scherrer's formula is observed to be 48.3 nm. FESEM images illustrate the electrodeposited layers, showing compact, void-free surfaces, and varying orientation. The composition analysis clearly indicates the peak of zinc and oxygen is present. The ratio of zinc and oxygen is observed to be 5.92 by weight percentage as well as 1.44 by atomic percentage. The deviation can be minimized by optimizing the growth condition. The resistivity of the film is observed to be 30 Ω-cm. The carrier concentration of the ZnO film is observed to be 4.7×10^{17} carrier/unit volume.

Structural and optical properties of Cr doping ZnO crystalline thin filmsfabricated by PLD technique

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High-quality Cr-doped ZnO thin films of single phase with preferred *c*-axis growth orientation were formed on Si (100) substrate by pulsed-laser deposition. The structural analyses demonstrate that all samples have wurtzite structure and are preferentially oriented along the *c*-axis perpendicular to the substrate surface. The intensity of defect-induced mode frequency for Cr-doped ZnO film increased showing the formation of such high-quality crystal. CrZnO thin films consist of nanorod-shaped grains with different sizes. Each of rods has a width in the range of 85-93 nm and a length up to several hundred nm. Photoluminescence spectra of films confirmed PL characteristics with the peaks in the UV and defect-related deep level emission in invisible region (blue, violet, green) between 419 to 398 nm and 450 to 546 nm. In the visible region of the PL spectra, the green light emission peak of samples at about 567 nm was associated with defects in ZnO. It is observed that RMS roughness increases with Cr doping which means the surface becomes rough and grain size increases. Thus it reveals that our fabricated thin films can be used in different applications like dye sensitized solar cells (DSSC), catalysis etc.

Development of manganese ferrite nanosystems via hydrothermal method & its potential application as MRI contrast agent

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Magnetic oxide has always remained a very noble matter for study & research purposes. It serves multipurpose role in variety of fields like Biomaterials, Nanoelectronics and various others. One of the most important is its use as MRI contrast agent in the field of Biomaterials. The MRI contrast agent is broadly divided into two categories namely T1 and T2. Gadolinium Oxides are super paramagnetic in nature & is widespread used as T1 contrast agent in the MRI, but it has been found that prolonged contact with Gadolinium lead to a Nephrogenic Systemic Fibrosis.So, it leads to further study of other potential oxides which can be used as T1 contrast agents. Mn^{2+} ions are superparamagnetic in nature in nature, which is one of the criteria which suits for MRI T1 contrast agents. Hydrothermal Method was adopted for the development of manganese ferrite nanosystems, the precursors mainly used were Ferric Chloride, manganese Chloride, Ethylene Glycol, Ethyl Acetate, Sodium Acetate. The samples was kept in an autoclave and heated to various range of temperature from 160° C to 200° C and observed at varying range of time periods from 8-20 hours. After cooling & drying of sample the XRD was done and it confirmed the sample as $MnFe_2O_4$ by using JCPDS no. 742403. The samples which were kept for 12 hour and 16 hour gave distinct peaks and patterns. The peaks in diffraction pattern of 16 hour sample was wider that 12 hour sample indicating that with increase in time period the size of nanoparticle reduced.

From XRD data it had been confirmed the sample developed is MnFe2O4 & the size is gradually becoming smaller with increase in time period.

Flame synthesis of nano crystalline indium doped tin oxide powder and its characterization

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Tin oxide (SnO_2) is a wide band-gap, n-type semi-conductor whose wide electrical and optical properties as well as chemical and thermal stability have caused it to be used in various photocell and optoelectronic devices. The addition of dopants from the III-family elements such as indium would lead to a p-type semiconducting behaviour in SnO_2. Various synthesis methods such as sol gel synthesis, spray pyrolysis etc. have been used for doping indium in tin oxide. However, not much literature is available on the synthesis of indium doped tin oxide in the nanocrystalline form using a gas phase processing route such as flame reactor synthesis. During flame synthesis a suitable precursor is pyrolised within the self-purifying environment of a flame resulting in the production of nanocrystalline particles with a reasonable degree of control on the synthesised powders. In this study, flame reactor synthesis has been used to prepare nanocrystalline indium doped tin oxide powders starting from inorganic precursors.Undoped and indium-doped SnO_2 nanocrystalline powders with systematically varying doping concentrations were prepared from inorganic precursors using an indigenously developed flame reactor. Standard characterisation methods like X-ray diffraction (XRD), high resolution scanning and transmission electron microscopy (HRSEM & HRTEM) with energy dispersive spectroscopy (EDS) were used to ascertain the phases and phase-purity, crystal structure and morphology of the particles as well as determine the presence of indium in the final powder. XRD studies showed clear peaks of only tetragonal SnO_2 indicating a phase-pure product with ultrafine crystallite sizes, while TEM studies confirmed that the nanoparticles were in the 20-50 nm size range. The indium content in the final powders was obtained for different In/Sn ratios in the precursor. It was concluded that the flame synthesis process can be a viable route for the production of indium doped tin oxide nanopowders.

Flame synthesis, characterization and stability of gadolinium-doped, nanocrystalline anatase titania powders

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Titanium dioxide (TiO_2) has attracted a lot of attention due to its potential in various kinds of applications, such as photovoltaics and solar cells due to the semiconducting behaviour, waste gas and waste-water treatment and self-cleaning coatings due to the catalytic properties. Further enhancement in applications can be obtained when the powders are in the nanocrytalline form. Of the polymorphs of titania, the anatase phase has higher functional value, but is stable at sizes below 14 nm and transforms to rutile at 400–600°C in addition to having a larger band. Recent studies have shown that lanthanide ion doping increases the anatase to rutile transformation temperature as well as the photocatalytic activity of titania. TiO_2 particles, produced in flame reactors using halide or metallorganic precursors exhibit higher purities

compared to that obtained from a liquid phase route and no further treatments such as filtration, drying and calcination are required. In this investigation an attempt was made to synthesize nanocrystalline gadolinium doped anatase titania using the flame synthesis route with the aim of increasing the anatase-rutile transformation temperature while at the same time decreasing the band-gap energy, thereby making it more viable for photocatalysis. Titanium tetra isopropoxide (TTIP) in ethanolwas used as the precursor for titania while an additional precursor, gadolinium nitratewas used for doping with gadolinium. The nanoparticles formed on pyrolysis of the precursor were analysed by transmission and scanning electron microscopy (TEM & SEM), X-ray diffraction (XRD) etc. The stability of the powders were studies using thermogravimetry and differential scanning calorimetry (TG-DSC) and the band-gap energies were determined by diffuse reflectance spectrometry (DRS). The role of key process parameters such as molar feed ratio of precursors and dopant concentration on the powder characteristics, anatase stability and band-gap energy were analyzed and the results are presented.

Session 3: Nov 15, 2014: [14.00 - 16.00]

Bend sensitivity and interfacial activity in rapidly solidified nanostructured bilayered soft magnetic ribbons

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Development of nanostructured soft magnetic ribbons through rapid solidification has been basically intended for application in transformers, choke cores etc. The ribbons used in these applications are mostly single layered wherein the microstructure and magnetostriction are optimized through heat treatment to achieve enhanced properties. In recent years, development of bilayered ribbons through melt spinning technique has given new dimension to the application of rapidly solidified soft magnetic ribbons. In the application area, the bilayered ribbons are expected to be potential sensors in biomedical applications like monitoring chest wall displacements (cardio-respiratory activity), static curvatures (airplane wings, optical lenses) and dynamic measurements like airflow detectors.Even the bilayered ribbons are promising materials for energy harvesting. The present investigation addresses Fe/Fe, Fe/Co, CoFe/CoFe bilayered ribbons with compositional and stoichiometric variations. The ribbons have been prepared through melt spinning wherein Dual nozzle technique (DNT) has been used. The rapidly solidified bilayered ribbons comprising of two different alloys, manifest the crystallization stages representative of individual layers during devitrification process as measured using Differential scanning calorimetry (DSC). Thermomagnetic transitions determined using Vibrating sample magnetometry (VSM) show change in the profile of magnetic moments during nanocrystallisation. The bilayered interface is evidenced from electron probe microanalysis (EPMA). The ribbons show synergistic elemental profiles of dominant elements at the bilayered interface. These ribbons which are bilayered, reveal interesting interfacial activity and functional gradation. The elemental microanalyses indicate the diffusion zone across the bilayer interface. The bend sensitivity has been measured for the monolayer and bilayered ribbons. The bilayered ribbons also show interesting bends sensitivity which will be addressed in terms of their magnetostriction and microstructural developments.

Application of nanotechnology in steel industry

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Nanotechnology is predicted to be a main driver of technology and business in this century and holds numerous potential applications in steel industry as well. The use of nanotechnology in steel leads to improvement in the properties of steels e.g. the fatigue strength of steel due to cyclic loading is improved. The use of vanadium and molybdenum nanoparticles improves the delayed fracture problems associated with high strength bolts reducing the effects of hydrogen embrittlement and improving the steel micro-structure through reducing the effects of the inter-granular cementite phase. Welds and the Heat Affected Zone (HAZ) can be brittle and fail without warning when subjected to sudden dynamic loading. The addition of nanoparticles of magnesium and calcium makes the HAZ grains finer in plate steel and this leads to an increase in weld toughness resulting in a smaller resource. Two products are available today viz. Sandvik Nano flex and MMFX2 steel. Both grades are corrosion resistant, but have different mechanical properties and are the result of different applications of nanotechnology. Sandvik Nanoflex has both the desirable qualities of high strength and resistance to corrosion. MMFX2 steel has a modified Nano-structure that makes it corrosion resistant and also an alternative to conventional stainless-steel, but at a lower cost.The use of copper nanoparticles reduces thesurface roughness of steel which in turn limits the number of stress risers andhence fatigue cracking. By regulating the formation of a fine grain structure, taking account of self- organization of nano phases, not only the strength but also the plasticity and cold strength of the steel may be increased. In this paper an attempt has been made to briefly illustrate the advantages of application of nanotechnology in steel industry alongwith present status and at the same time suggest new potential area of applications.

Synthesis, processing and characterization of nanocrystalline Ni and Ni-Cu alloy

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In the present study, pure nanocrystalline Ni and Ni-5wt.%Cu alloy were synthesized using high energy ball milling. Microstructural changes during all stages of synthesis and processing were studied using XRD and SEM. In both alloys, during course of milling, the ratio of 200/111 peak intensity of Ni was observed to increase and then decrease with subsequent milling. Further,it was also observed that addition of Cu enhances this ratio. After 20 h of ball milling, mechanically alloyed powders were consolidated using spark plasma sintering (SPS). For similar SPS conditions higher relative density was obtained for Ni-Cu alloy as compared to pure Ni. Microstructural characterization of dense SPS pellets was characterized using electron back scattered diffraction (EBSD) studies.

Preparation and characterization of free standing nanoparticles by cryomilling

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The free standing nanoparticles or nanocrystals have scientific and technical interest due to shape and size dependent properties, which are closely related to important applications, like chemical sensors and catalytic properties. However, due to synthesis related difficulties, the bulk production of such particles remains challenging issue. Theoretically the preparation of free standing nanoparticles by milling is decided by competition between cold welding and fracturing during ball milling. These two factors are strongly dependent on the milling temperature. Cryomilling can be efficiently used to prepare the free standing nanoparticle. Also, cryomilling allows the significant particle size reduction with less contamination. Importantly, cryomilling suppress the cold welding due to low temperature, leading to rapid grain refinement. In this context, free standing nanoparticles of Cu, Zn, and Al have been prepared by mechanical attrition at cryogenic temperature. The decrease in grain size and generation of strain were explained by X-ray diffraction using Hall-Williamson method and the same was confirmed by the transmission electron microscopy (TEM) analysis as well laser particles size analyzer, and theory behind free standing and strain generation as well grain size refinement have been studied. The free standing nanoparticles were achieved within 270 minute of milling and remain free standing beyond 10 days. The particle morphology was investigated using FESEM and the contamination due to milling media was analyzed using ICP-MS (Inductive coupled plasma -mass spectroscopy). The results show the formation of nanosized (10-20 nm) free standing nanoparticles in case of Cu, Zn, and Al during cryomilling. The results will be discussed using theoretical understanding of the subject.

Synthesis of nanostructure hydroxyapatite by wet precipitation route for biomedical applications

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In recent years, hydroxyapatite, the main inorganic composition of natural bone, has been extensively researched for biomedical application due to its excellent biocompatibility and bioactivity. This phase is basically a derivative of calcium phosphate, with few molecules of water, which forms rod shape complex hexagonal crystals. Because of its affinity to create quick bonds with natural bone, it is extensively used for repair and replacement of injured bone tissue.For this reason, HA synthesis techniques have been explored a lot. These include techniques, like, hydrothermal synthesis, electrochemical deposition, sol-gel synthesis, combustion method, mechnochemical synthesis etc. In the present effort, we are using wet precipitation technique to prepare HA having calcium oxide as calcium precursor and ortho phosphoric acid as phosphorus precursor. The calcium oxide is available as a waste in many industries like steel, sugar, glass. So in other way we are developing a method to process this waste and use it for biomedical application. Also the wet precipitation approach includes low processing temperature, which range from room temperature to 95°C. Yield of HA obtained by this process is high. The precipitate obtained is dried at 100°C for 24 hours and sintered at 300°C, 400°C, 600°C and 800°C. The HA powder, thus synthesized, is evaluated for constituent phases and degree of crystallinity using x-ray diffraction technique. Morphology of powder is analysis by SEM. FTIR is done to check functional groups present in the powder.

Effect of different carbon nano-fillers on mechanical and photocatalytic properties of sintered TiO_2 composites

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Titania (TiO_2) based composites reinforced with 2wt.% of graphene nanoplatelets (GNPs), carbon nanotubes (CNTs) and single wall carbon nanohorns (SWNHs) were synthesized at 1000 ^{o}C using spark plasma sintering (SPS). X-ray diffraction (XRD) and Raman spectroscopic studies confirmed that the milled powders comprised of anatase phase which transformed into rutile phase during SPS. Nano transformation twins were also observed on rutile particles in transmission electron microscope (TEM) images. Fractured surfaces showed that fillers are well bonded with the TiO_2grains. Mechanical properties of the composites were evaluated using nano indentation studies. Indentation hardness and elastic modulus of GNP reinforced composites were found to increaseby 78% and 30% respectively, compared to TiO_2 compact. The hardness and modulus of the CNT reinforced TiO_2 increased by 22% and 5.4% respectively while that of the SWNH reinforced TiO_2 increased by 11% and 23% respectively.Effect of these nanofillers on photocatalytic degradation of methylene blue solution was investigated. It was observed that SWNH reinforcement improved the photocatalytic properties of TiO_2 while CNTs and GNPs did not affect the properties significantly.

Effect of rotating cathode speed on high yield of arc discharged multiwalled carbon nanotubes in open air

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Carbon Nanotubes were synthesized using direct current (DC) arc discharge method in open air condition. Arcing was achieved with a horizontal graphite anode and a vertical rotating cylindrical graphite cathode, with the use of Ni catalyst. The experiments were conducted at a constant current density of 150 A, whereas rotating cathode speed was varied in each experiment. The speeds of rotating cathode are 5 rpm, 10 rpm, 15 rpm, 20 rpm, 25 rpm, 30 rpm, 40 rpm, and 50 rpm and also in stationary cathode condition. As synthesized raw soot samples were washed with distilled water and purified. As synthesized and purified samples were characterized using Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Raman spectroscopy, and Transmission electron microscopy (TEM). The impact of the speed of rotation of the cathode on CNT synthesis was determined.

Comparison of $\alpha-Al_2o_3$ synthesized by sol-gel process using $AlCl_3$ and $Al(No_3)_3$ as precursors

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Nano-sized alumina has a unique combination of excellent mechanical properties and high thermal stability. The combination of structure, topology and dimensions create a horde of physical properties like high hardness and resistance to crack propagation thereby making it an ideal reinforcing agent. The applications emanating from the remarkable properties of nano-sized alumina include optical devices, high temperature materials, in biomedical engineering, as coatings against corrosion and abrasion, in electrical engineering for fabrication of fuel cells, sensors, piezoelectric devices etc. The present study deals with the synthesis and characterization of Al_2O_3 using two precursors by the Sol-gel method. Owing to the difficulty of synthesis of nano-sized α-Al_2O_3 powder due to grain growth at high temperatures involved during calcinations, Al_2O_3 was prepared by two different sol gel processes, one using aluminium chloride ($AlCl_3$), ethanol and 28 % NH_3 followed by calcinations at 1200°C for 2 hours, and the second process using aluminium nitrate$Al(NO_3)_3$, malic acid and PVP (polyvinylpyrrolidone) in the mass ratio of 10:3:1.5 followed by heating the gel at 60°C for 24 hours in an oven and then heat treating at 1250 °C for 2 h to form α-Al_2O_3. The crystalline degree and crystallite size of the alumina formed were analysed by XRD and particle size analysis. Microstructural analysis was performed using SEM and TEM. Further, characterization of particles was carried out by FESEM, DSC and FTIR techniques. The resultant alumina synthesized by the second process was found to have a higher aspect ratio and hence is more suitable as a reinforcing agent for high strength composites.

A bio-comparative study of copper nanoparticles synthesized by using different reducing agents

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In the present study, Copper Nanoparticles (Cu NPs) were prepared byreduction of copper nitrate ($CuNO_3$) using three reducing agents viz. sodium Borohydride (SBH), tri-sodium citrate (TSC) and oxalic acid (OA). In every experiment, copper nitrate and reducing agent were taken in 1:1 ratio. The concentrations of copper nitrate and reducing agents used are in the range of 0.02-1M. The synthesized Cu NPs were characterized by UV-visible spectroscopy, particle size analysis, x-ray diffraction, SEM/FESEM with EDAX and zeta potential. UV-visible spectroscopy revealed a typical surface plasmon resonance (SPR) in the range of 228-320 nm. The size of copper nanoparticles, as determined by the particle size analysis, is seen to be 37 nm when SBH was used as reducing agent, 14 nm when TSC was used as reducing agent and 46 nm when OA was used as reducing agent. The shape of the Cu NPs was found to be reducing agent dependent. Snowflake shaped Cu NPs were obtained when SBH was used as reducing agent, hollow tubes/rod shaped Cu NPs were obtained when TSC was used as reducing agent and ellipsoid/rice-grain shaped Cu NPs were obtained when OA was used as reducing agent. XRD and SEM-EDAX patterns indicated the presence of copper along with oxygen as an impurity. The antibacterial activity of Cu NPs dispersion was measured by Kirby Baeur method. The Cu NPs synthesized by using 3 reducing agents using six different concentrations showed varied antibacterial activity against gram +ve and gram –ve bacteria's.

Model-based design and scale-up of an aerosol flame reactor for nanoparticle synthesis

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Aerosol flame reactors are used for producing fine particles of titania, fumed silica and carbon black on the industrial scale and for synthesis of nanoparticles of metal oxides, metals and composites on the laboratory scale. But design and scale-up of flame reactors for nanoparticle synthesis has not received enough attention because existing knowledge on this technology has evolved through trial and error rather than through the principles of aerosol reaction engineering. Heuristics and correlations for design and scale-up of the laboratory and pilot scale reactors have been proposed in the literature. But their applicability was tested for pilot scale reactors in a limited manner only, and application for industrial scale processes still needs to be examined. Hence, there is a need for a generic method which can guide the designer for systematic design and for scale-up of the reactor for a given production rate and particle characteristics. Computational fluid dynamics (CFD) models for the flame dynamics inside the reactor and population balance models (PBMs) for predicting the powder particle size distribution have been developed and tested with varying degrees of success in the literature. We have recently proposed a model-based design and scale-up methodology for flame aerosol synthesis of nanoparticles using a coupled flame dynamics – monodisperse population balance model (CFD-PBM). The proposed scale-up methodology was tested using published experimental data for synthesis of silica and titania nanoparticles using burners of three different sizes. The design methodology was applied to establish a laboratory scale facility which was utilized for synthesizing silica and carbon black and also to study the effect of oxidant flow rate on axial flame temperatures. Results of the scale-up studies using the model and preliminary results from the laboratory scale experimental facility will be presented.

Synthesis of zirconia nanopowder using planetary ball mill

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This work deals with preparation of nano-zirconia powder using planetary ball mill and then characterization ofsynthesized powder to analyze the particle size. Powder is characterized using Field Emission Scanning Electron Microscope (FE-SEM), X-ray diffraction (XRD) and particle size analyzer. FE-SEM showed decrease in particle size from 200-500 nm to 50 nm with some amount of agglomeration. XRD showed that ball milled zirconia powder has a mixture of monoclinic and tetragonal crystalline structures. Laser particle size analyzer noted a significant decrease in particle size, i.e., from about 53 nm to 13 nm. Using the 20 h milled zirconia nanopowder, PAEK/zirconia nanocomposites were prepared using suspension method followed by hot pressing. The content of zirconia nanopowder was varied between 0 and 30 wt% (~ 9 vol%). It was observed that presence of zirconia enhanced microhardness, thermal stability and dielectric constant of the PAEK. SEM showed good dispersion of milled zirconia in the PAEK matrix as compared to as received zirconia powder in the matrix.

Preparation and reduction of free-standing graphene oxide – activated charcoal hybrid paper

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Graphene is the wonder material of 21st century because of its unique properties. Even though Graphene is produce on industrial scale, it is highly difficult to produce mono-layer graphene industrially due to the inherent difficulties in the procedures being followed. Thus we are not able to achieve the properties of pristine Graphene. In order to enhance the properties of as produced Graphene, we've tried to incorporate Activated Carbon (AC) and study its effects on properties. Activated carbon in the percentages of 10, 20, 30 & 40 was added to Graphene oxide on weight basis and the hybrid papers were vacuum annealed at 250° C to obtain reduced Graphene oxide-Activated Charcoal papers which were further characterized by FE-SEM, Raman Spectroscopy, Four Probe method & TGA. FE-SEM images have shown the layered structure with incorporated carbon particles. Raman spectroscopy has shown the defects and graphitic bands. Also the I_D/I_Gratio increased with increasing AC content indicating the induction of defects. Four Probe measurements showed increasing electrical conductivity with increasing AC. This can be attributed to the higher reduction that has occurred in higher AC content papers due to the catalytic activity of AC on the removal of superficial oxygen content. TGA indicated the reversal in mass loss, i.e. higher AC content paper lost less mass at a given temperature, which could be attributed to the AC content increase, which would have blocked the pores leading to the difficulty in the inner oxygen content to diffuse out.The enhancement in properties like electrical conductivity of the hybrid paper with increased AC content may lead to reduction in the cost of Reduced Graphene Oxide Paper production and make Graphene a more commercially viable product.

Imaging of nanoparticles by electron and ion beam microscopy

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Nanomaterials, specifically nanoparticles, are, without a doubt, key components in the development of new advanced technologies. Due to the unique optical, electronic, and molecular-recognition properties of gold nanoparticles, they are the subject of substantial research, with applications in wide areas of research, including electronics, nanotechnology, and materials science. To understand the potential of these nanoparticles, a deeper knowledge of their synthesis and characterization is needed. This study involves the image analysis of gold nanoparticles embedded on silicon substrate. Imaging of these nanostructures were performed by electron and ion beam using Scanning electron microscopy (SEM) and Focused ion beam microscopy (FIB) respectively. In addition to gold, small thickness film of silver nanostructures were taken into account. For both the samples, electron and ion beam imaging were performed and results were analyzed with variation in thickness of gold film and tilt angle of sample stage. Monte Carlo Simulation software was used to plot the ion and electron beam trajectories and projected range.

Fabrication of nano-porous alumina template via selective dissolution

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Porous materials have various applications, such as catalysts, drug delivery, energy storage and conversion, self- lubricating bearings, membrane and membrane support, heat exchanger, etc. In this present work, porous alumina template is fabricated using pressureless sintering via selective dissolution of Ni. Alumina and Ni powders of 99.9% purity were ball milled (300 rpm, 40 hrs, 10:1 weight ratio) in order to get homogeneous mechanical mixture. This powder mixture was characterized using particle size analyzer, BET, X-ray diffraction (XRD) and scanning electron microscopy (SEM). The milled powder was compacted and pressureless sintering was carried out in reducing atmosphere (H_2) at 1400° C. SEM studies along with energy dispersive spectroscopy (EDS), optical microscopy (OM) and XRD were performed to study microstructure evolution, bonding characteristics and distribution of Ni particles. Ni was then selectively dissolved from the 1 mm thick sintered disc of diameter 16 mm in 1M HCL+3wt% $FeCl_3$ solution in order to get the porous template of alumina. The size, distribution and nature of pores in porous templates of alumina were investigated using SEM along with EDS, optical metallography.

Synthesis, characterisation and antimicrobial activity of Cu NPS using oxalic acid as reducing agent for a range of concentrations

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Copper nanoparticles(Cu NP's) were synthesised using copper nitrate as metal precursor and oxalic acid as reducing agent with different molar concentrations 0.02M, 0.04M, 0.06M, 0.08M,0.1M and 1M by chemical reduction method. The as-synthesized Cu nanoparticles were characterised using UV-visible spectroscopy, zeta potential, particle size analyser (PSA), SEM-EDS and TG-DTA. The UV-Visible spectroscopy results indicated the presence of Cu NP's with SPR peak range between 230-240nm with respect to colour change from white to milky blue and colour turbidity increased with increase in concentration. The FESEM reveals the morphology to be ellipsoid/rice grain shaped particles in case of all concentrations. The particle size was around 46-110 nm. The zeta potential was found to be negative for all the CuNP's samples produced at different concentrations. The purity of the sample analysed by EDS was 73%. These Cu NPs were tested for their antimicrobial activity against gram +ve and gram –ve bacteria like E-coli (MTCC390, gram –ve), micrococcus luteus (MTCC7256, gram –ve), lactococcus lactis (MTCC440, gram +ve), bacillus species (MTCC10616, gram +ve). The CuNP's showed a good antimicrobial activity on lactococcus lactis bacteria.

Relative study of Cu NPS synthesized chemically using different reducing agents and their applications

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Copper nanoparticles (Cu NPs) were synthesized chemically by using copper nitrate ($Cu(NO_3)_2.3H_2O$) as metal precursor and sodium borohydride ($NaBH_4$) sodium citrate tribasic dihydrate ($C_6H_5Na_3O_7.2H_2O$, extra pure AR) (also known as Tri-Sodium Citrate) and oxalic acid $(COOH)_2$ as reducing agents maintaining the molar ratio as 1:1 and a concentration of 0.02M. The morphology of Cu NPs synthesized by these three reducing agents was found different. Synthesized Cu NPs were characterized by UV-visible spectroscopy, XRD, SEM-EDS, FESEM, Particle size analysis (PSA) and Zeta potential. The surface plasmon resonance (SPR) peak range was observed between 228-315nm with respect to the change of colour of the samples from olive green to dark brown in case of sodium borohydride as reducing agent; cyan to blue colour in case of trisodium citrate as reducing agent and white to milk blue in case of oxalic acid respectively. The average size of the Cu NPs was found to be in the range of 40-50nm for trisodium citrate, sodium borohydride as reducing agent and 14-15 nm in case of oxalic acid as reducing agent as determined from PSA. The FESEM images of Cu NPs revealed three different morphologies viz. snow flake in case of sodium borohydride, hollow tubes in case of trisodium citrate and ellipsoid/rice grain type in case of oxalic acid. SEM-EDS confirmed the presence of copper nanoparticles with less oxidised particles.The antibacterial activity of Cu NPs dispersion was measured by Kirby Baeur method. The Cu NPs synthesized by using 3 reducing agents showed varying degrees of anti-bacterial activity against gram +ve and gram –ve bacteriaand the results from antibacetrail activity will be discussed.

Synthesis and optical properties of undoped zinc sulphide nanocrystal embedded in aerogel matrix

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Combining sol-gel chemistry, a widely used technique for synthesis of glasses and ceramic materials, with semiconducting nanocrystals (NCs), could lead to the development of new materials to replace environmentally unsafe solid-state lighting and optical materials. ZnS semiconductor nanocrystals were synthesized by sol-gel technique at room temperature and embedded in aerogel. Here we use silica aerogel because it possess pores of all sizes and have high surface area to enable every embedded nanocrystals to sense the environment independently. Synthesized sample are characterized by UV-Visible spectrophotometer, Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM) and Photoluminescence Spectrophotometer (PL). UV-Vis absorption spectra revealed that ZnS and ZnS with aerogel samples showed absorption peaks at 332 nm and 389 nm respectively and indicates a red shift. Size of as synthesized nanoparticles measured by TEM and SEM was ~ 5nm.The samples were annealed at 100^0C , 200^0C , 300^0C respectively for 30 minutes each to study the morphology and size variation of the prepared samples with temperature. PL spectra shows a red shift for ZnS and ZnS with aerogel sample with increasing particle size. But after annealing, PL spectra shows red shift for ZnS and blue shift for the ZnS with aerogel sample.

Optimization of parameters of mechanochemical activation of fly ash by taguchi method and determination of fractal geometry of Agglomerates lacunarity

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In this study, statistical analysis by Taguchi method was used to optimize parameters of high energy ball milling to produce nano-structured fly ash. A class-F fly ash was subjected to high energy ball milling-induced mechanochemical activation in presence of a surfactant. Ball milling parameters, such as ball to powder weight ratio, type and quantity of surfactant and type of medium were varied as guided by the Taguchi design. The nano-structured fly ash was characterized by dynamic light scattering, BET surface area analysis, X-ray diffraction, Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy and transmission electron microscopy. The average particle size was 329 nm which reveals that the ethyl acetate as a medium, a ball to powder ratio of 12:1 and 2 wt.% of non-ionic surfactant (Triton X-100) improved the reduction in particle size and accelerated the suspension stability of nano fly ash particles. Morphological studies revealed that the smooth and inert surface of the fresh fly ash was converted to a rough and more reactive one after 48 h of ball milling.The fractal dimension determined the individual agglomerates from their planar projections andthe compactness of the initial geometrical arrangement in the agglomerate, this justifying the historical association with the lacunarity of the agglomerate particle. The surface modification of fresh fly ash was determined from FTIR spectroscopy. The nano-fly ash obtained by this method has application potential in polymer industries as reinforcement in composites.

Invited talks

Session 1: Nov 12, 2014: [13:30 - 15:30]

Steel industry in India: Prospects of growth

S. Banerjee

At first, the author recalls the inspiring contributions and achievements of late Dr. Bal Raj Nijhawan–in building, organizing, managing NML in its formative years and in delivering excellent R&D. More important, the values which he instilled in NML's colleagues and the remarkable foundation which he laid – continue to imprint NML's outputs even after five decades. This paper examines the prospects of growth of steel industry in India. During the last 60 years, Steel Consumption (Production + Import) in India has approximately doubled – every 10 years. A model for steel consumption was proposed and validated by steel production data of the different steel making countries of the world during the last one and a half century. Based on the above data and model, it is estimated that steel consumption in India would increase from the current level of ~ 95 million tons (mt) to about 435 mt in 2040. The inputs and resources required to produce the additional 340 mt steel during the next 25 years, would depend on technology routes that the new or existing plants adopt. The major alternative technology routes are two: BF-BOF; and Electric Process. During the last 5 decades, steel production using the two routes show different growth trends. Keeping this trend in mind, one can identify the Inputs and Outputs required when the two alternative routes are used to sustain the growth in steel production in the next 25 years. The growth has to be planned and projected in such a long time frame, since the Infrastructure, Raw Materials and sources required are rather large and therefore, the time required to obtain them on the ground will be very long. The feasibility of each route and location of the plant would depend on several factors: the availability of the primary Inputs and the Raw Materials required; the ability to adopt recent R&D-based technology in using the alternative Raw Materials; the capability of the route to make niche and special purpose products; and the manufacturing flexibility to deliver as per a customer's requirement. With the above in mind, the prospects of decentralization and dispersal of the industry is examined.

Mechanical activation of gibbsite and boehmite: New findings and their implications

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The minerals, gibbsite [Al2O3.3H2O] and boehmite (γ-AlOOH) are central to the production of various alumina based ceramics and chemicals having numerous applications. Nature of bauxite (e.g. gibbsitic, boehmetic, mixed, etc) and its processing in the Bayer process are also determined by the presence of these minerals. Mechanical or mechanochemical activation (terms coined by Smekal in 1940s) refers to enhancement in reactivity of a solid due to physicochemical changes induced during milling. This paper is an overview of our recent research on mechanical activation of gibbsite and boehmite. The focus is on

changes induced during milling in terms of physicochemical characteristics and reactivity; especially, in the context of thermal transformation to alumina polymorphs and leachability in alkali. The results obtained using planetary and attrition mills are compared. In the case of gibbsite, interesting finings covered include: enhanced amorphisation due to the presence of quartz and hematite during attrition milling; role of milling energy; and, texture induced zeta potential changes. Similarly for boehmite these are: anomalous reduction in surface during planetary milling of boehmite prepared by thermal transformation of gibbsite; differences arising in mechanical activation of boehmite depending on the methods of preparation (i.e. thermal and hydrothermal transformations of gibbsite); boehmite to bayerite transformation during attrition milling of boehmite. Further, it has been shown that the leachabity of boehmite is significantly enhance due to mechanical activation and, irrespective of method of preparation, near complete dissolution can be achieved below 100°C. The results presented have great practical implications from tailoring, through mechanical activation, properties of Al-oxhydroxides and oxides to the development of a universal Bayer process, and these are highlighted.

Session 2: Nov 12, 2014:[16:00 - 18:00]

Mitigating sustainability challenges in mining and mineral industry through technological innovations

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India is blessed with huge mineral reserves and thus mining industry plays a vital role in the economic growth of the country. Given the underappreciated but well-endowed geological base of natural resources, the anticipated increased natural resources demand can easily be met. Domestic production will not only play a crucial role given the demands of raw materials security challenges but will also have a transformational role in Nation's development. The Natural Resources industry contributes to Government's revenue on account of taxes and production levies. In contrast, imported raw materials result only in transferring the country's wealth to others.Given that maximum value can be created by encouraging domestic production in India, it is Exploration and Innovation that will shape the Nation's Natural Resources landscape. There are many global examples of successes derived from enhanced exploration and deployment of latest technologies in Natural Resources. In the 1950's and 60's, when Australia, as a nation was the same age as the Indian republic, the Australian national iron ore resources were 350 million tonnes. However, encouragement of exploration and adoption of new innovative technologies for resource mapping, managing environmental issues with ultimate objective of inclusive growth has added billions of tonnes of iron ore and now Australia exports more ore than it thought it had in 1950, with additional new ore being discovered every year. Similarly, the US Shale Gas is another excellent example of a case study where exploration and new technologies have reduced the country's dependence on imported oil. The Mining Sector today contributes a meager 1.3% to GDP. This contribution can easily be enhanced to 5.5-6.0% of GDP by 2020 and fully meet the demands of the Nation's future economic growth. The mining sector has the potential to generate additional employment (direct and indirect) of 2.3 million by 2020 and contribute at least USD 40 billion to the economy. The competitive advantage of the mining sector lies in large scale and low cost production and logistics which will make it possible for the industry to meet significant demand from sectors like construction and manufacturing at market determined prices. No doubt, to bring this great potential to fruition, enabling and forward-looking policies are required but such policies shall be effective only when there is a social license to operate from the society. Today, the faith of the community has been shaken and large scale protests are seen everywhere against any existing or new mining projects mainly on account of social & environment conflict. Large overburden heaps on the mine head, big tailings ponds in mineral processing

complexes, discharge of mine water and effluents to outside water bodies, non-utilization of wastes and tailings for value added products etc are some of the reasons for such opposition. These issues can easily be managed by adopting Innovative Technologies to make Mining and Mineral industry as a Zero Discharge, Zero Waste and Zero Harm Industry so that besides regaining people confidence, profitability of the business also can be improved. One such example is converting Power business into an integrated business of Aluminium, Power and Glass/ Cement where Fly ash can be used for production of Aluminium and Silicates or Geo-Polymer and thus cost of power can be reduced substantially besides eliminating need of any ash pond or dumping area

Environmentally benign bauxite beneficiation using mining microbes

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India has significant reserves of bauxites and is ranked third among the world's largest producers. Both high grade as well as lower grade bauxites find industrial applications in different ways such as metal production, refractory and ceramic manufacturing. Major impurities in Indian bauxites located in the eastern, western and the central belts of the country include iron oxides, calcium carbonates, clay matter and silica, all of which deleteriously influence their processing and end uses. For example, presence of silica and iron oxides are detrimental to aluminium production processes, where as the presence of calcium and iron cause many processing problems and lower the quality of refractories and ceramic products produced. Though several physicochemical methods such as flotation, gravity separation, high intensity magnetic separation and classification are used to achieve beneficiation of mined bauxites, all of them are not environmentally benign, cost-effective and energy-efficient.Since bauxite mineralization is biogenic, many types of microorganisms are indigenously present in the deposits bringing about biochemical reactions leading to mineral formation, conversion and transport. Such native microorganisms are capable of bringing about dissolution, dispersion and flocculation of mineral constituents such as iron oxides, silica, clays and calcium carbonates. Mineral-specific microbes can be isolated, cultured and grown in the laboratory and used to bring about selective removal of desired impurities from bauxites. Such a microbiological approach would prove to be cost effective, energy-efficient and environment-friendly.Biomineralization of bauxite deposits is illustrated with respect to biogenesis and formation of gangue constituents such as silica, iron oxides and calcium minerals interlinked with aluminium oxides and silicates. Microbial processes to remove calcium, iron and silica are discussed in terms of mechanisms, process optimization and quality of beneficiated products. Biotechnological innovations in bauxite beneficiation and environmental control are brought out with relevance to Indian conditions.

Session 3: Nov 13, 2014:[09:00 - 11:00]

Recovery of quality iron ore fines concentrate through incorporation of slime beneficiation system at SAIL mines

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SAIL iron ore mines processes ore mainly through 2 to 3 stage crushing followed by wet screening and classification using screw classifier. In treating ore in wet circuit, 15-25% (of run-of-mine ore) slimes (<0.2 mm) are generated, which consists of good quality fine iron ore liberated particles. Huge quantities of slimes have got accumulated in the tailing dam of Meghahatuburu, Kiriburu, Dalli, Barsua and Bolani mines over the years. SAIL R&D centre has implemented innovative online slime beneficiation systems in two iron ore mines, viz., Dalli & Meghahatuburu and are in the process of implementing at Bolani and Barsua mines. Also lot of work has been carried for developing process flowsheet of the accumulated slime at Dalli and Kiriburu mines, which are also in the process of implementation. At Dalli, slime with very low quality (50% Fe, 15% SiO2, 9% Al2O3) could be enriched to 62.5% Fe, 5.2% SiO2, 2.2% Al2O3 by treating in Fluidised Bed Classifier, followed by hydro-cyclones and modified Slow Speed Spiral Classifier (SSSC). This resulted an extra production of 40 tph concentrate with the final reject quality to <44%Fe. At Meghahatuburu also, the slime/process reject (~100 tph) with 57-58%Fe could be enriched to 62.6%Fe by treating in two stage hydro-cyclones at a product yield of 50%. The final reject slime has been brought down to ~48%Fe. Presence of Goethite minerals along with Hematite makes it difficult to recover the valuable minerals further from slime. As additional advantage of this modification, the load on the thickener has been reduced considerably and the water recovery from the thickener has also been improved. By way of additional recovery of fines the life of tailing dam would also get extended resulting in better environmental friendly mining activity.The amenability for the beneficiation of iron ore slimes deposited at tailing dam of Kiriburu (6 Mt) and Dalli (15 Mt) mines over the years has been investigated for utilization in sinter making process. Except for the lowest stream slime sample of tailing dam, most of slimes are very much amenable to beneficiation. Wet screening followed by Hydro-cyclone and WHIMS resulted an optimum iron mineral recovery of 75% with 64%Fe in product concentrate

Emerging iron ore beneficiation trends in eastern India

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India has large reserves of iron ores which are the basic raw material for iron and steel industries. Indian iron ores especially in Eastern Region, though rich in iron, suffer from two distinct disadvantages: (a) high alumina percentage in the ore and (b) softness of the ore which generates huge quantity of fines and slimes. In addition ,these slimes are stored in huge tailing ponds which pose enormous environmental hazard .Worldwide in general we observe alumina / silica ratio of the iron ore raw materials is <1.0 but most of the Indian hematite iron ores show a reverse trend. Alumina bearing minerals are inter-grown with iron oxide minerals and in general liberation takes place in fine size ranges (generally 90 % liberation at 60 micron). Clay and gibbsite are the predominant mineral phases present as surface coating, fracture filling and/or free occurring and are responsible for alumina content being higher. Washing is the most common steps adopted by Mining Industries for iron ore beneficiation and helped in removal of adhering

clays and silica to produce lumps and fines of desired quality to some extent. In view of gradual depletion of high grade deposit and as well as space constraint in mining lease to store fresh generated slime, mining Industry has started adopting complete beneficiation to reduce fresh generated slime while enriching product which in turn increasing productivity of Mines .This key note describes emerging trends of iron ore beneficiation flow-sheets in Eastern India–specific to Barbil zone to reduce product alumina which has detrimental effect in agglomeration process as well as in Blast Furnace operation .

Raw material sustainability for mineral processing industry-Issues, opportunities, and challenges

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While mineral resources are the key inputs for the mineral processing and primary metals industry worldwide, the processes for recovery of minerals and metals have been evolved over the years, for quality of resources as available. The quality of mineral resources, defined in terms of the main metal content, percentage of the primary mineral type, major impurities and minor element components, have been going through considerable changes, on account of the increasing global demand and hence the consumption rate, trends towards nationalization of mineral (and other) resources, and pressures for significant value addition to the resource at the place of origin. Increasing sustainability pressures, on account of the generation of large volume of disposable wastes, so characteristics of mineral processing industry, steep reduction in the availability of land for storage of waste solids and tightening environmental norms and long term producer liability for waste generation and environmental damage pose additional constraints. Mineral processing industry has responded to these challenges by adapting to the use of increasingly lower grades of raw materials, through multiple approaches such as-process changes, pre-processing of the low grade raw materials, or development of novel alternative processes for recovery. In addition, cost effective separation of minor components, hitherto lost with the process waste and process development for utilization of process wastes as resource through value recovery, so as to reduce the disposal load, have been additional steps taken by industry to manage the utilization of low grade resources. Such holistic approach has resulted into development of break-through technologies for the commodity materials, utilization of non-conventional resources, potential for reduction on cost of manufacture, often below the traditional costs, and combination of process technologies to multi-product plants etc. For non-ferrous industry, dealing in mining of bauxite, copper ore and coal, and manufacturing of aluminum and copper by conventional technologies, this approach of poses a set of challenges as well as formidable opportunities to create a sustainable business model for long term sustainability. Some of the technical challenges taken up by global non-ferrous metals industry will be discussed and move of the industry to future-proof the business of these materials, which are closely linked to the growth in the global GDP and standard of living, will be discussed.

Processing of coal fly ash for the recovery of Alumina

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Indian coal contains higher amounts of ash (35-45%) thereby generating huge quantity of fly ash. Coal fly ash (CFA) is an incombustible residue produced by burning coal in thermal power plants. The nature of the coal decides the composition of fly ash. The silico-aluminous fly ash contains various valuable minerals such as mullite, quartz, hematite, magnetite, amorphous silica, CaO, TiO2, etc. in a matrix of aluminosilicate glass. Alumina is mainly associated in mullite matrix. The average quantity of CFA generated in India at present is about 130 million tons. This means around 30 million tons of alumina is lost every year in fly ash considering 25% alumina in CFA.The use of fly ash for making roads, bricks, etc, is although is a relief for its partial utilization for low value application, by this we are wasting the valuable materials present in it. Alumina, the major value material is getting wasted due to unavailable of suitable processing options.The use of fly ash as source of alumina extraction has been tried through different methods such as lime stone sintering, soda lime sintering, high pressure hydrothermal treatment, ammonium sulphate sintering process and acid leaching processes etc. A number of disadvantages are associated with each of the process. Sintering process produces huge quantity of residue which is several times higher than the original fly ash. In ammonium sulphate process the requirement of ammonium sulphate is huge (10:1, ammonium sulphate: alumina). The sulphuric acid method requires 200-210°C, with volumetric ratio of acid to CFA is 5:1 and extraction rate is around 85%.In the present study different processing route were tried for the treatment of CFA to recover alumina. Sulphuric acid leaching of fly ash in presence of Fluoride ions solubilises alumina in CFA. Almost 90% alumina recovery was obtained in this route. XRD data also confirmed the dissolution of mullite phase. In another option amorphous silica was removed, prior to alumina recovery through hydrothermal leaching in alkaline medium and CaO treatment, as dicalcium silicate. The alkali is regenerated in this process. The residue was subjected to sulphation roasting followed by water leaching to recover more than 90% of alumina values. The alumina values ultimately recovered as pure alumina after precipitation and calcination. Further, in another option aluminium fluoride was produced from CFA by reacting it with hydrofluorosilic acid. The papers describe the issues and optimum conditions associated with the process flow.

Session 4: Nov 13, 2014:[13:30 - 15:30]

Technological challenges on low grade iron ore beneficiation and palletisation

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Iron ores are valuable natural resources being finite and non-renewable. They constitute the vital raw materials for iron and steel industries and are a major resource for national development. As per the recent National Steel Policy of Govt. of India, steel production will be enhanced to 250 MTPA in 2025 from current production of 82MTPA. For production of 250 MTPA, the country needs the high quality ore around 370MTPA. The country is not endowed with high grade requisite iron ore resources. It is, therefore, imperative to achieve the best use of available iron ore resources through scientific methods of mining, beneficiation and pelletisation. Challenges and solutions for upgrading Indian low grade hematite iron ore in association of goethite, kaolinite, gibbsite minerals as well as recovery of micro fines of iron phase minerals from slimes/tailings from iron ore washing/beneficiation plant respectively should be taken seriously to maximize the utilization of these resources for long term sustainability by using suitable eco-friendly beneficiation process through R&D backup. Similarly, the economic and eco-friendly process is essential to maximize the recovery of iron value from lean grade ore BMQ/BHQ/BHJ through physical beneficiation or combination with pyro-metallurgical process. The nature of iron minerals and associated

gangue minerals decide the process flowsheet of beneficiation to be adopted.To utilization of micro-fines concentrate, the Indian iron makers are sifting their focus from sinter intensive blast furnace operation to pellet oriented operation. Adoption of suitable pelletisation technology of varying capacity is necessary to encourage meeting specific needs with respect to availability and type of iron ore fines/concentrate in the country. The Indian hematite iron ore concentrate has high LOI because of the presence of kaolinite, goethite and gibbsite even after beneficiation process. As this ore is in fragile nature, the Blaine number of pellet feed material is high. To maximize the iron values from the slimes and tailings, flotation process may be adopted in future. It provides the hydrophobic characteristic on the surface of the particles, which has negative impact on pelletisation. It is the challenging task to handle the iron ore concentrate having high LOI, high Blaine and hydrophobic surface particles. It needs to improve the heating cycle of induration segment of the present pelletisation technology either grate kiln or straight grate processes through fundamental studies.

Scandinavian experience with 100% pellets - can Indian steel industry benefit?

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This paper summarizes the experiences for the Scandinavian Integrated Steel Plants (ISP) transferring form sinter based 100% pellets based production. The presented results are all collected from Scandinavian furnaces, meeting the specifications given by Indian Bureau of Mines in Vision 2020. Further, the paper show operational data from the Scandinavian furnaces. The operation with 100% pellets have resulted in operation with a coke rate of below 300 kg/ton HM and a PCI rate of 160 kg/ton HM. The slag amount in the blast furnace has been reduced to 150-180 kg/ton HM. The magnetite base of the Scandinavian iron ore allows for extremely low energy consumption in the pelletizer, less than 8 kg coal equivalents pr. ton pellets. The paper also outlines one possible/alternate route for blast furnace operators with limited availability of high grade iron ore. The basis for this discussion is information from the Iron & Steel Vision 2020 published by Indian Bureau of Mines in 2011. As the mining in India historically has been concentrating on the high grade parts of the ore bodies, large volumes of lower grade hematite and magnetite ores have been left. Time has now come to return to these ores and reclaim the iron units for the iron and steel production of India.Beneficiation of iron ore, hematite and magnetite by crushing, flotation and separation (gravimetric and magnetic) will remove the unwanted gangue and trace elements. Modern agglomeration technology will secure aggregates well suited for BF operation.A change from a lump/sinter operation to a pellets based operation of blast furnaces is possible and may well be beneficiary for the production rate of the ISP as well as for the DR-units of India.If results similar to the Scandinavian are achievable in the Indian Iron and Steel Industry are discussed in this paper

Session 5: Nov 13, 2014:[15:30 - 17:30]

Role of metals in meeting the challenges of 21st century

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Metals have been associated with the progress of humancivilization and economic development for the last 6000 years. The invention of steam engine by James Watt laid the foundation of modern metallurgical industries by highlighting the needs for precise borings and castings that were necessary

for trouble free operation of rail road cars which kick started the industrial revolution of 18th century. Till latter half of 20th century the world community remained focused on growth of industrial and economic activities quite oblivious of their deleterious impact on the environment . Thanks to the discovery of depletion of ozone in the earth's atmosphere, problem of global warming, satellite imagery depicting shrinking glaciers in the polar region of the earth and rising sea level, the search started for alternate source of carbon free energy. The everrising price of oil the production of whichis monopolized by a few nations in the Middle East gave further impetus to the above search for renewable energies.

Rare earths, a group of 17 elements belonging to the group 3 of periodic table plays significant role in alleviating the above problem of global warming. However, production of rare earths from the minerals in their separated forms is not easy to achieve and mining of rare earths minerals are not without problems of ecological importance. Rare earths often occur along with other elements mostly of radioactive in nature and their processing not only involves use of hazardous chemicals, but is also often associated with liberation of objectionable gasses and pollutants. In the presentation comprehensively covers the technological, economic, social, environmental and legal issues associated with the mining, mineral separation, processing and usage of rare earths compounds and metals for achieving the challenges of 21st century.

Application of ionic liquids in the green extraction of rare earths

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Ionic Liquids (IL) or molten salts, made up essentially of ionic species and occurring in liquid form with melting points below 100°C, are of late gaining wide attention from scientists and researchers all over the world in view of their special properties. If they exist as liquids in ambient temperatures, they are called Room Temperature Ionic Liquids (RTILs). RTILs are very interesting on at least three counts: (i) by appropriately choosing different organo-metallic complexes as constituent ions one can fine tune specific aspects of the governing chemistry, (ii) the overall reaction can be manipulated electrically and (iii) one can take the advantages of faster kinetics of liquid phase in room temperature itself which might lead to higher efficiency. Generally the ionic liquids have high electrochemical conductivity because of their ionic nature, high thermal stability, low flammability and very low volatility and these special properties give definite advantages in their use for a number of diverse applications. Significant research is reported in literature on ionic liquids and the volume of the scientific literature on ionic liquids have gone up exponentially from an extremely low value in 2001 to more than 3000 reported articles in 2011. Ionic liquids find applications in various fields, such as dissolution of cellulose and other biopolymers, de-polymerization of non-natural polymers, extraction of rare earth elements from primary and secondary resources, capture of carbon dioxide and other gases produced by the burning of fossil fuels etc. etc., and different levels of development have been achieved in all these. It is relevant to mention here that a fully industrial application of ionic liquids in any field is yet to commence, ionic liquids, nevertheless, show a lot of promise for their adoption in large scale use. In this talk we would direct our discussion to the extraction of rare earth elements using ionic liquids. In view of the recent shortage felt for the rare earth elements and their compounds, globally awareness has been created to increase the resource base for rare earths and India is no exception. With its reasonable reserves of light rare earth elements (LREE) bearing monazite in the placer deposits and heavy rare earth elements (HREE) bearing xenotime in the inland and river placers, efforts are already on in this country to maximize the recovery of rare earth elements. Spent e-wastes also are also another promising resource for rare earths. At IIT Bhubaneswar, a

strong research base has been created for intensive investigations on synthesis of ionic liquids and their characterization and separation of rare earth elements and precious metals using extraction with ionic liquids. Studies are in progress in the separation of La, Ce, Dy, Gd and Ru at the Institute.

Exploitation of rare earth materials for modern applications

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Rare earths are an important group of materials, abundant in India and it is required for high technology applications such as fuel cracking catalyst, permanent magnets, industrial Phosphors, ceramics etc. The production of these materials in the World is dominated by Chinese due to their deposits of bastnasite and xenotime minerals, unlike monazite mineral associated with thorium in India. Till recently, the Chinese strategies were aimed at achieving monopoly status. The Western World has realised the importance of the rare earth material for strategic uses and hence have revived rare earth extraction in the past three years at various facilities. The presentation would focus on applications of rare earth, the markets they are linked to such as the markets for magnets, Ni-MH batteries, Fuel cracking catalyst and Industrial Phosphors. The demand for rare earths is growing and in the Indian context the extraction of rare earth based metallic materials from rare earths have not matured to commercial levels. The technologies involved in rare earth metal extraction would be highlighted in this presentation. The author's experience in the extraction and application of some of these materials for magnet application, Ni-MH battery application and hydrogen storage container development would be presented.

Session 6: Nov 14, 2014:[08:30 - 10:30]

Beneficiation of low-grade barites by flotation column; from lab-scale studies to commercial production

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Barite deposit at Mangampet, Kadapa district of Andhra Pradesh is one the world's largest deposits containing 61 million tonnes of recoverable barite. Since the high grade barite resources are limited, utilization of low-grade dumps is essential to meet the demand. Beneficiation of low-grade barite interlocked with schist and slate gangue was studied by flotation process. Though barite can be separated by direct flotation using oleic acid as collector, flotation of silica using amine as collector (reverse flotation) was followed to obtain barite concentrate free from oleic acid coating. Instead of conventional flotation cells, flotation column was opted to derive better metallurgical results. Effect of process parameters like reagent dosage, slurry pH, liberation size, superficial air velocity, bias water etc on grade and recovery of barite was studied. Laboratory scale tests have indicated that the barite concentrate assaying 95% $BaSO_4$ can be obtained with a recovery of around 70% in a single stage flotation. The recovery was improved to 85% by incorporating scavenging flotation by conventional cells. Based on the laboratory results, commercial size flotation column was designed and commissioned for M/s Andhra Barite Corporation Pvt

Ltd, Kadapa, Andhra Pradesh. Beneficiation plant to process 700 tpd of barite was established at Mangampet and utilization of low-grade barite dumps was demonstrated.

Developments in oxidative alkaline processing of Indian uranium ores

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Nuclear energy constitutes an important component of overall energy mix planned for futuristic requirements of India. 10% of the nuclear power reactors under construction world-wide are from India. The fuel requirements for these reactors are planned from indigenous resources and from imports. Some of the prominent low-grade uranium ore deposits in India which form part of the fuel supply-chain (immediately or in near future) for nuclear power reactors are the carbonate hosted uranium ores from Tummalapalle in Andhra Pradesh and Gogi in Karnataka. Recovery of uranium values from such host rocks invariably involve processing under alkaline conditions, which is a cost-intensive process unless efficient regeneration and recycle of reagents is implemented. The complexity associated with uranium mineralisation in both these ores is completely divergent. While the nature of uranium mineralisation in the medium-grade Gogi ore (U3O8 0.18%) is simple, the difficulty stems from developing a single hydrometallurgical processing scheme viable for recovering uranium present in both limestone and granite host rocks which co-exists in the mineralised zone. Added to this is the need for subjecting the ore to very-fine grinding which has its own processing problems, chiefly the solid-liquid separation and tailings disposal. Processing of Tummalapalle uranium ore is also equally formidable due to physical refractory nature of uranium mineralization. Except for few discrete grains of pitchblende present in intimate association with pyrite segregates in dolomicrite, bulk of the uranium values (about 95%) is as ultra-fine disseminations in lighter minerals, mainly carbonates, of specific gravity < 3.3. The paper describes the intensive R & D efforts involved in development of oxidative alkaline processing scheme for obtaining high purity final product from both the ores imbibing concepts like conservation of fresh water, reagents and energy efficiency. These investigations led to setting-up of a commercial uranium recovery plant at Tummalapalle in Andhra Pradesh and pre-commercial project activities in Gogi, Karnataka

Session 7: Nov 15, 2014:[11:00 - 13:00]

The Copper Conundrum

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Despite being the oldest metal processed by mankind, the copper industry remains an enigma, even today. Price volatility has always been an area of concern. To protect against the fluctuations of LME, the copper mining industry has adopted the time-tested strategy of increase in scale of operations through massive mechanization resulting in higher productivity. However, like in other metals, with time, the grade has fallen. The industry has responded with improved mineral processing technologies that have raised recoveries, to partially compensate the fall in grade. Hydrometallurgy has also come handy for processing

oxide ores, leaner ores and secondary sulphide ore. Beneficiating the lean ores has caused accumulation of huge volumes of tailings in dams, using up extensive stretches of land and consuming lots of water required for handling of slurries. Some work is under way and a lot more needs to be done to extract metals and minerals of value from these dumps.Smelting and refining provides the least value addition in the value chain from ore to finished products. Treatment and refining charges have been traditionally determined on the basis of the gap between mining i.e. supply and smelting i.e. demand of concentrate and has no relation to cost of processing. Economies of scale and improved technologies have come to the rescue of smelters to overcome market forces and the effect of rising energy prices, through rise in recovery and current efficiency. Lately, new technologies such as the SKS process and chloride leaching of sulphide concentrate hold a lot of promise.Considering the energy intensive nature of smelting operations, recycling of scrap, more so, e-scrap could be game-changer. For this to materialize, an organized system of collection and sorting of scrap needs to be in place. While treating huge volumes of e-scrap, the issues of environment with regard to emissions must not be lost sight of. Development of new alloys and new applications of copper has not received as much attention as in the case of other metals. Probably, the absence of primary players in this field is the cause. The aluminium industry has grown tremendously, thanks to the development of finished products such as beverage cans, automobile and aerospace components, in which primary players have played a significant role. Attributes of copper such as its anti-microbial properties, excellent thermal and electrical conductivity, leading to energy efficiency and corrosion resistance have not been thoroughly exploited. There exist immense scope to utilize these properties through commercial applications that can be easily justified if life cycle costing is considered. Addressing of all these issues could significantly change the future of the copper industry in the long run.

Enhancement of flotation selectivity of sphalerite using mineral-stressed Paenibacillus polymyxa and its cellular components

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The selective flotation of sphalerite from a sphalerite-galena mineral mixture was achieved using cellular components of Paenibacillus polymyxa after adaptation to the above minerals. The soluble and insoluble fractions of the thermolysed bacterial cells adapted to sphalerite yielded higher flotation recoveries of sphalerite with selectivity indices ranging between 22 and 29. The protein profile for the unadapted and mineral-stressed cells was found to differ distinctly, attesting to variation in the yield and nature of extra-cellular polymeric substances (EPS). The changes induced in the bacterial cell wall components after adaptation to sphalerite or galena with respect to the contents of phosphate, uronic acid and acetylated sugars of Paenibacillus polymyxa were quantified. In keeping with these changes, a marginal morphological transition of Paenibacillus polymyxa from rods to spheres was observed. The role of the dissolved metal ions from the minerals as well as that of the constituents of extracellular secretions in modulating the surface potential of the mineral- stressed cells was demonstrated. These studies highlight that mineral stress lead to qualitative and quantitative changes in the cellular components, which facilitate enhancement of flotation selectivity of sphalerite.

Session 8: Nov 15, 2014:[14:00 - 16:00]

Tailings dewatering technologies to mitigate environmental hazards

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In order to prevent the release of tailings slurry from coal or mineral beneficiation plant into the environment, mines usually have a disposal facility in which tailings are impounded in pond or dam. This is a common practice of storage since tailings are mostly in the form of slurry when they are discharged from the plant to the tailings pond. In recent years, land acquisition for construction of pond for disposal of tailings is becoming increasingly difficult. In addition, disposal of wet tailings through trucks/dumpers are also not desirable options because they create nuisance of the area resulting in environmental hazard. Further, advance technology allows lower grade ores to be exploited, generating higher volumes of waste that require storage. Environmental regulations are also becoming stringent creating further pressure on the mining industry, particularly with respect to storage of tailings. This paper describes the possibility of tailings storage in dry condition which can alleviate the environmental issues arising out of wet disposal of tailings. This paper also discusses the current practices and the latest trend on dewatering technologies to produce dry tailings that mitigate the environmental damages.

Contributory papers

Session 2:Nov 12, 2014: [16:00-18:00]

Quantitative estimation of mineral phases from chemical assays and powder X-ray diffraction rietveld analysis

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Separation efficiency in most of the mineral processing plants is generallymonitored and/or reported in terms of chemical assays pertaining to various separated fractions/process streams. However, a true representation would be to track mineral-wise separation, as quite often, different mineral phases comprising of same element behave very differently under similar process conditions. With the advent of automated mineral analyzers (such as QEMSCAN and TESCAN), it has become possible to determine mineralogical composition of various plant streams. The mineral analyzers,typically basedon simultaneous processing of SEM images and corresponding X-ray microanalysis data from multiple detectors, are relatively expensive and time consuming at this point of time;thus, limits their wide spread applications. In this paper, we propose a method for quantitative estimation of mineral phases in ores and process streams. The methodusesconventional bulk chemical assay data and information on phases present as

determined by powder X-ray diffraction (XRD) pattern analysis.We illustrate the utility of our approach by taking example of selective dispersion-flocculation of iron ore slimes. Iron ore slime samples were subjected to selective dispersion-flocculation experimentsin our lab and feed, concentrate and tailswere analyzed using the approach. Chemical assays (iron, alumina, loss on ignition (LOI) and silica) were determined by wet chemical analysis and mineral phases (hematite, goethite, gibbsite, kaolinite and quartz) were quantified by Rietveld analysis of XRD patterns. Considering the elemental proportion in the minerals, mass balance of elemental assays and minerals, the method is developed for reconciliation of assays and determination of mineral composition in process streams. The estimated mineral compositions reasonably match with those obtained by quantitative phase analysis by the Rietveld analysis. The estimated mineral compositions in feed, concentrate and tails were also used to compute mineral wise recoveries. Our generic approach thus provides mineral engineers a simple and relatively easy method to convert elemental assays into mineralogical compositions and also compute mineral-wise recoveries, grades and distributions in the various plant streams.

Recovery of iron values from iron ore slimes of Donimalai tailing dam

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At present Indian steel Industry is passing through serious challenges to achieve targeted capacity. The National Steel Policy 2008 targets the estimated domestic steel production of 180MTPA by 2019-2020. It is a known fact that during production of calibrated (sized) ore, more than 50% fines and rejects (Slimes) generated which cannot be directly utilised in iron making due to its unfavourable granulometry, low iron content, high alumina and silica content. Approximately 10-20% of the process plant input is discarded as slimes in to slime ponds/tailing dams. Tailing dams are now considered as threat, due to lack of high grade iron ore and acute shortage of land for storing. More over these slimes also pose threat to the environment. Recovery of iron values from slimes results in economic benefit by utilisation of waste as a resource, minimizes the land requirement, surface degradation, ground water pollution, and destruction of forests. These slimes after beneficiation agglomerated (Pelletisation) and can be used as burden for the blast furnace. This research work presents the route for beneficiation of slimes by removing gangue in the slimes to a level which is acceptable for the blast furnace route production of iron. The slime sample was sampled from tailing pond of Donimalai Iron ore mine and transported to R&D Centre, NMDC Limited, Hyderabad. The 'as received' slime sample assayed 49.40% Fe, 13.51%SiO_2, 8.44% Al_2O_3 and 5.16% loss on ignition and all other trace elements are within the critical limits. It is possible to produce a pellet grade concentrate which assayed 65.93% Fe, 1.45%SiO_2, 1.94% Al_2O_3 and 1.56% loss on ignition with a weight recovery of 45.18% with a recovery of 60% Iron values.

Effect of an organic binder and industries waste slag on pelletization of hematite ore eliminating bentonite

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A bentonite has been using as a binder to iron ore pelletization from very beginning. It gives excellent binding property to green and dry pellet properties. However several drawbacks of bentonite have been

observed such as an impurity due to its acid oxide contents. In order to replace the bentonite, we thought to be use the organic material as a binder. An organic binder only gives sufficient green pellet property although it failed to give strength at high temperature. In addition to binder problem, Induration of hematite pellet would require very high temperature and longer firing time than magnetite. It has been thought that induration temperature and firing time can be lowered if enhancing the recrystallization, diffusion process or incorporating same low melting slag. We used industries waste slag along with an organic binder as an additive to overcome the above problem. It has been observed that only 0.5 wt% of an organic binder can provide very good green properties. From the properties of indurated pellet, it has been found that organic bonded pellet shows very high RDI (27%). Thus, use of only organic binder would not be acceptable. The varying amount of industries waste slag has been added with 0.5 % of organic binder and found to improve CCS to a great extent. The developed pellet shows around 300 kg/pellet strength at induration temperature of only 1250°C. However, the pellet of similar basicity with 0.5 wt% bentonite shows 300 kg/ pellet CCS at induration temperature of 1300°C; i.e. the induration temperature requirement for the developed pellet is 50 °C lower than the usual bentonite bonded pellet. The phase identification of both developed pellets and bentonite bonded pellet through XRD shows mainly Fe2O3, CaAl2Si2O8 phases which are in agreement with the estimation through thermodynamics software FactSage

Waste polymer encapsulated pellets (WPEP): Converting challenges into opportunities

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"Waste Polymer Encapsulated Pellets" are new raw material category for the production of pig iron. WPEP (Waste Polymer Encapsulate Pellets) are produced by the encapsulation of iron ore pellets by waste polymers and has demonstrated a huge potential of minimizing industrial dust due to raw materials handling from pellet making unit to the smelters at the same time this consume the waste polymers like (PET, LDPE, HDPE, etc.) to make the environment free from pollution caused by waste polymers. Encapsulation of iron ore pellet with waste polymer will result in isolation of pellets from the surrounding (moisture, dust, water etc.). 10% Increase in cold compression strength of pellets were observed as compared to the convectional pellets. WPEP has 95% better abrasion strength as compared to the convectional iron ore pellets. Microstructure evaluation of pellets discloses that irregular pellet surface and pores near to the pellet surface are the reason for the dust generation due to abrasion from pellets. Coating on the pellet with waste polymers fills these surface pores of pellets at the same time polymer diffuse inside the pellet surface pores and arrest the inherent cracks of the pellets which will result in enhancement in the pellet CCS and abrasion strength. Coating on pellet minimizes the shattering and abrasion of pellets inside the blast furnace also. Addition of waste polymer (hydrocarbons) in blast furnace will reduce the coke consumption for smelting and result in economical hot metal production.

Session 4: Nov 13, 2014:[13:30-15:30]

Reduction roasting of BHQ

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With the increasing demand of steel, need for exploring low grade ores has gained importance. One such low-grade iron ore is Banded Hematite Quartzite (BHQ), with iron content ranging between 30% and 38%. Apart from their low iron content, another problem with beneficiating banded iron formations is intricate mixing of ore and gangue mineral grains, which results in optimum liberation occurring at sizes below 100-150 microns. Para-magnetic iron ore, consisting primarily of hematite, limonite or siderite can be converted to magnetite by reduction roasting process. This magnetite can be further reduced by magnetic separation. In this study BHQ sample was reduced in the presence of coke oven gas in a Laboratory scale tubular furnace. The roasted material was ground to -150µm and feed into Davis Tube at different current. The magnetic and non-magnetic fractions were collected, dried, weighed and their iron analysis was carried out. The effect on yield on varying feed size, roasting temperature and roasting time had been studied. Reduction roasting of BHQ sample at optimized condition produced a concentrate with a grade of 60% Fe at a yield of 51.5%.

Physico chemical analysis of iron ore tailings

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Chemical analysis of iron ore tailing sample was conducted to ascertain the exact composition. The chemical analysis flow sheet has been elucidated here. Stoichiometric calculations were performed to determine the composition of the tailing sample. The chemical composition and crystallite size was confirmed by X-Ray Diffraction using different targets. The loss of ignition from the ore was confirmed using Thermal Gravimetric Analysis. The surface characteristics of the pellet mix were determined using a Zeta Probe Analyser. The size of the fines was determined using the laser based particle size analyzer. The ore sample was found suitable for pelletisation. The mineral beneficiation flow sheet for the tailing sample has been outlined from the analysis.

Dual flux iron ore pellets: quality and microstructure

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High alumina content in the Indian iron ore fines (2.5% Al2O3) resulted in poor quality pellets at the recently commissioned 6 MTPA pelletizing plant and a study was under taken to establish suitable pellet chemistry through proper fluxing agents. Limestone fluxed pellets were prepared at varying basicity (0 to

0.8) and pyroxenite fluxed pellets at varying MgO contents (0 to 3%) to study their effect on the metallurgical properties. Fired pellets were tested for their strength, RDI, reducibility and swelling. Microstructural studies, optical and SEM-EDS, were carried out to quantify different phases and their chemistry.Acid pellets exhibited poor quality whereas limestone pellets at 0.8 basicity and pyroxenite pellets at 1.5% MgO exhibited optimum metallurgical quality. Formation of widely distributed silicate melt in limestone pellets and high melting point slag & magnesioferrite in pyroxenite pellets found to improve their quality. However during commercial production of MgO bearing pyroxenite pellets, their room temperature and low temperature properties found to be inferior, but exhibited better high temperature properties, viz. softening-melting behavior. Contrary to this, commercially produced limestone pellets, comprising CaO, exhibited better strength and RDI but showed poor high temperature properties. To yield the benefits of both CaO (for good strength and RDI) and MgO (for low swelling and high softening), a dual flux combination was established by using both limestone and pyroxenite/olivine together that resulted in optimum metallurgical properties. Coke rate at the blast furnaces found to be reduced by 1kg for every 1% increase of dual flux pellets in the burden

Selective dispersion-flocculation studies for the beneficiation of Indian iron ore slimes

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Current practice of iron ore processing in India typically leads to the generation 15-20% of slimes. These slimes, despite having high amount of iron, are being discarded into tailing ponds due to higher alumina contents (10-15%). These tailing ponds not only occupy major lands but also pose an environmental hazard. Several research groups are working on development of viable processes for the beneficiation of slimes so as to make it amenable for iron and steel making. Considering the fine size particles present in these slimes (<150 microns), selective dispersion – flocculation seems to be one of the most appropriate processes for their beneficiation. Our research is directed at employing state-of-the-art molecular modeling techniques for the design/selection of selective reagents for dispersion-flocculation of iron ore slimes. We have identified several promising reagents based on our computational studies and currently testing them in experiments. In this paper, we present our results on the selective dispersion– flocculation of iron ore slimes using different combinations of dispersants (sodium hexametaphosphate, polyvinyl pyrrolidone and sodium silicate) and flocculants (starch and guar gum). With careful selection of process conditions such as pH, dispersant dosage, flocculant dosage and settling time we could obtain a concentrate containing >66% Fe and <3.5% Al2O3 with an yield of more than 60% from a natural iron ore slime obtained from one of the mines in India assaying 58% Fe and 7% Al2O3.

Design of experiments (DOE) studies to optimize the selective dispersion-flocculation of Indian iron ore slimes using guar gum flocculant

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Development of effective beneficiation processes for the utilization of high alumina containing Indian iron ore slimes has been topic of research for several years and more so in the recent times due to rapid depletion of high grade iron ores. These slimes, in spite of having good amount of iron values, are currently being disposed into tailing ponds owing to their high alumina contents (10-15%). Due to very fine sizes of the particles present in the slimes (<150 micron), selective dispersion flocculation seem to be a promising route for their beneficiation. At our lab, we are working on developing a selective dispersion flocculation process for the beneficiation of slimes. We have employed state of the art molecular modeling based design and selection methodology to arrive at selective reagents for the process. Based on our computational results and subsequent laboratory tests, guar gum flocculant was found to be effective in the beneficiation of slimes. The process thus developed needs to be optimized further for scale-up at pilot/plant scale. The present work involves a full factorial design of experiments study on the beneficiation of iron ore slime using guar gum flocculant. The experiments were designed to study the effect of the three key process parameters i.e., pulp density, guar gum dosage and settling time on the beneficiation of iron ore slimes. The experimental data is used to develop models for yield, iron grade, alumina grade and iron recovery in the concentrate. The models thus obtained are utilized to arrive at optimized process parameters to achieve better iron recovery and grades with less alumina in the concentrates. The optimized process conditions shall form base line for further scale-up of the process.

Raw material planning process for JSW Steel - Dolvi complex

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JSW Steel is an Integrated Steel Plant at Dolvi, having state of the art technology for making hot rolled coils and sheets. The facilities includes BF, SIP, Sinter plant, Pellet plant, Coke Oven plant & Lime calcination plant for iron making and CONARC- CSP for steel making. As there is no raw material sources proximity to the plant as well as captive source, raw material planning is very important and crucial activity for this plant. This activity involves interactions with the Plant Heads , Costing , Shipping , Jetty , Raw Material Handling Section (RMHS), Finance , Centralized Procurement Team , Site Offices at Vizag , Orissa and NMDC. The incoming raw material comes to the Dolvi plant mostly by sea route and there are lots of challenges and day to day activities involved while planning the logistics of raw material from various sources. Feed mix planning of iron making units is extremely dynamic due to variability of actual production of agglomeration units and coke oven. The Lead Time, Logistics from Mumbai Port to JSW Jetty through Barges and thereafter from Jetty to RMHS are also very crucial and complex activities as far as the material availability at plant and Vessel Demurrage are concerned. The Inventory optimization and proper Optimum Funds Planning are also the crucial activities which are being monitored by Raw Material Planning Section. This paper illustrates how the Raw Material Planning has been done for JSW Steel – Dolvi Complex on Annual Basis / Quarterly Basis / Monthly Basis to take care of the Raw Material requirement.

Mineralogical and characterization studies of low grade iron ores

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Upgradation of low grade iron ore was started due to depletion of high grade iron ore and strategic concerns. Chemical and physical properties of iron ore depends on mainly their geographical locations and constituents present in ore. With the change in location, properties of ores are also changed and content of constituents are varied. Low grade iron ore contain or made up of various compounds such as oxides, silicates, carbonates and in combinations with other minerals; which belong to solid solution processes during formation of host rock. During these formations, iron ore crystallizes with each other in randomize locked conditions.Present work was carried out on mineralogical and characterization studies of low grade iron ores. The mineralogical studies included ore mineralogy, phase analysis, structural, textural studies, ore and gangue distributions, photo-micrograph of sample. For chemical analysis and phase identification, different techniques were employed such as XRD, SEM, EDS, XRF. For liberation point of view, physical properties were also checked. These studies gave the clear picture of phase identification and also help in liberation and separation of valuable ore mineral for the low grade iron ore.

Session 5:Nov 13, 2014:[15:30-17:30]

Separation of rare earth metals by room temperature ionic liquid (RTIL)

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Use of conventional acidic or saponified extractants in the solvent extraction process releases H+, NH4+, Ca2+, and Na+ ions into the aqueous phase, which affects the extraction efficiency and generates the effluents of high salt concentration. The present investigation aims at separating the trivalent lanthanides by solvent extraction using room temperature ionic liquid (RTIL) as extractant which has a potential to obviate the above mentioned problem. A commercially available RTIL, Cyphos IL 104 comprising of an organic phosphonium cation and organic phosphinate anion, was used to study the effect of pH and extractant concentration on the separation of trivalent lanthanides in the hydrochloric acid solution. Extraction of lanthanides increased with increase in pH and RTIL concentration. Increase in pH during extraction indicates that acid competes with lanthanides in the organic phase without releasing any ions such as H+ or Cl- into the aqueous phase. McCabe Thiele plot shows that complete extraction of Lu (III) can be achieved in only two counter current stages at O/A ratio of 1/3 and pH 5.0 with 0.002M Cyphos IL-104. When simulation of counter current extraction was carried out, 93% Lu (III) was loaded in the organic phase leaving behind 1.23 mg/L of Lu (III) along with other rare earth metals (La, Nd, Gd) present in the aqueous solution. Extraction of lanthanides with the RTIL followed the ionic radii sequence: Lu (III)> Gd(III)>Nd(III)>La(III). It was also possible to recover the trivalent lanthanides as chlorides by stripping with HCl. The work emphasizes the potential of using room temperature ionic liquid such as Cyphos IL-104 in separating heavy lanthanides from lighter lanthanides as a group and addressing the drawbacks of using conventional acidic extractants.

Selective precipitation of yttrium as high purity oxide from leach liquor of phosphor powder

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Phosphor powder present inside the fluorescent tubes is a rich source of rare earths (REs). The present study deals with the selective precipitation of yttrium as yttrium oxide (Y2O3) from the HCl leach liquor of phosphor powder using oxalic acid (H2C2O4). The precipitation was carried out while varying the pH of the leach liquor and concentration of the oxalic acid. After mixing the oxalic acid with the leach solution the instantaneously formed slurry was stirred for a definite period. The precipitate was recovered by filtration, washed with distilled water and was dried in an oven overnight at 80 oC. It was noted that under the acidic conditions the recovery of yttrium and other rare earth metals such as La, Ce, Eu, and Tb by precipitation was high, and the precipitation of Ca, Al and P was almost negligible. However, at low acidity other metal ions were also precipitated along with these REs. Since yttrium behaves as a heavy lanthanide and its concentration in the leach solution being high, precipitation of yttrium was found to be selective at higher acidity. The dried oxalate powder containing mostly yttrium was calcined at 600 oC to convert it into its oxide (Y2O3). The yttrium oxide powder so produced was characterized by XRD, EPMA and chemical analysis. The XRD analysis of the product confirmed the presence of yttrium oxide only, although very minor amounts of Eu and La were also detected in the EPMA. The EPMA image of the synthesised yttrium oxide exhibited the flower petal shaped particles of 5µm size along with a few particles of other morphologies. The purity of the final product (Y2O3) was found to be ~95% (with 60 wt % Y) along with contamination of Eu and La.

Session 6:Nov 13, 2014:[15:30-17:30]

Effect of kimberlite dosage on green properties of iron ore pellets

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During diamond mining at Panna Mines a huge amount of tailing generated .This generated tailing is called Kimberlite tailings, produced after recovery of Diamonds from Kimberlite rock. The Kimberlite contains about 32 % [MgO+CaO] and rest are primarily Silica, Iron oxide [Fe2O3] & Loss on Ignition [LOI]. During green pellet making it is observed that strength of pellets improves with higher dosages of kimberlite. The further studies were taken to replace bentonite with kimberlite. The iron ore pelletization processes requires production of green pellets of size 9-16 mm, which can be fired in grate kiln or straight grate furnace. The green pellet should posses' sufficient strength to withstand stress and strain during handling. In the present study, the effect of dosage of kimberlite on the properties of green pellets has been studied. Iron ore fines from Bailadila-5 have been taken for study. The kimberlite obtained from mines as solid waste have very low surface area [700-900 cm2/grams], and subjected to grinding. The surface area need to be optimized for minimum grinding cost. This paper attempts to describe the effect of surface area of kimberlite and its dosage on green pellets properties and minimum binder (Kimberlite) surface area required to produce pellets with sufficient strength.

Recovery of quality iron ore fines concentrate through modification of slime beneficiation system at Meghahatuburu mines

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The wet processing circuit of Meghahtuburu iron ore mines consists of screens, rake classifiers and hydro-cyclones. The underflow product of hydro-cyclones is de-watered in Slow Speed Spiral Classifiers (SSSC) and conveyed to fines stock pile. While processing ores through wet mode, nearly 20% of ROM ore were getting lost as slime through primary hydro-cyclone overflow. This slime, though of inferior quality, still contains significant amount of rich quality iron mineral particles in them. The average Fe content in slime is around 59%. Coarse and heavy mineral particles settle faster in thickeners and serious jamming problems were faced while raking the particles out of it. Characterization study of the slime sample shows majority of gangue minerals exist in very fine size (less than 15 micron) range.For recovering these heavy high density iron mineral particles of such a fine nature in slime, de-sliming hydro-cyclones have been introduced for second stage of beneficiation. The overflow of primary hydro-cyclone is taken as the feed to the secondary hydro-cyclone whose cut size has been kept at 15 micron only. This modification in the beneficiation circuit has been able to segregate and recover iron mineral particles with the product having ~62% Fe. Further enrichment of the concentrate to 62.5% Fe could be achieved by installing a permanent magnet in the launder base, carrying the cyclone underflow product to SSSC. The entire modified slime beneficiation system is thereby generating an extra production of 50 tph of sinter grade fines from the slime. As additional advantage of this modification, the load on the thickener has reduced considerably and the water recovery from the thickener has also improved. By way of additional recovery of fines the life of tailing dam would also get extended resulting in better environmental friendly mining activity.

Selection of process technology for production of pellets for use in BF

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There are several iron ore pelletization process technologies in the world. The two most popular ones are straight travelling grate and grate kiln technology. Difference in two process technology is in drying, preheating and firing system. In straight grate system, a series of grate cars move in straight line at the same speed during drying, firing and cooling. This is similar to sintering machine. In grate kiln technology, all these process steps are achieved through grate, rotary kiln (similar to sponge iron kilns) and cooler. Choice of technology depends on factors like chemical composition of iron ore, type of ore (hematite or magnetite), capacity of pellet plant, end use (Blast Furnace or DRI kiln) etc. Indian iron ores require beneficiation also. Process of beneficiation plays an important role in selection of process. Straight grate technology is more popular. Going by the statistics of built in capacity in world, 247 Mtpa capacities is by straight grate and 125Mtpa is by grate kiln. Generally, grate kiln process is more suitable to magnetite ore (60 %) compared to straight grate process (38%). For hematite ore these figures are straight grate (25%) and grate kiln (7%). Straight grate technology is more popular for producing pellets which are used in BFs. SAIL is going in a big way to install pelletization plant along with beneficiation plants. Pellets produced shall be largely used in Blast Furnace. At Gua mines a beneficiation and 4 Mtpa pellet plant is being installed for which order has been placed. At RSP a beneficiation and 2.0 Mtpa pelletization plant is coming up which is

under tendering stage. Both the plants will have straight grate technology. A beneficiation and 1.0 Mtpa pellet plant is coming up at Dalli mines of BSP. Paper deals with description of processes, special features of various processes, advantages and disadvantages, selection criteria, suitability of process for BF application, present status in world and status of same in SAIL.

Development of a phosphate precipitation method for the recovery of uranium from carbonate leach liquor of tummalapalle ore

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Carbonate host rock of Tummalapalle uranium ore in Kadapa basin of Andhra Pradesh is one of the largest uranium reserves in the world. A novel alkaline pressure leaching technique is adopted to process the ore to bring the uranium value in carbonate leach liqour. Due to higher content of carbonate (~85%) and complex mineralogy of the Tummalapalle ore the uranium content in leach liquor varies in the range of 0.7 to 1.1g/L U3O8 with high contents of carbonate (~20g/l), bicarbonate (~75g/L), sodium sulphate (~105g/L) and total dissolved solutes (TDS) ~160g/L. Recovery of uranium from such a complex solution is generally accomplished by sodium diuranate precipitation route with concentrated NaOH at a pH >12. However, due to low concentration of uranium in the leach solution complete recovery is still a challenge. In order to get complete recovery of uranium, an effective and efficient method based on uranyl hydrogen phosphate precipitation route in the presence of a precipitating reagent under controlled pH has been investigated in the present work. Effect of various process variables including quantity of sodium hydrogen phosphate, pH, digestion time, temperature, concentrations of uranium, sodium carbonate, sodium bicarbonate, and sodium sulphate has been studied to optimize the best suitable conditions for quantitative recovery of uranium. The experimental results yielded > 99.9% recovery of uranium in the pH range of 6.0-6.5 with >10% stoichiometric amount of sodium hydrogen phosphate at ambient temperature. The recovery of uranium was found to be independent with respect to the concentrations of carbonate, bicarbonate and sulphate upto 75, 75 and 120 g/L respectively. Phosphate precipitation route was effective even at a lower concentration of uranium in the aqueous solution (~50 mg/L U3O8). Settling characteristic of the uranyl phosphate precipitate was relatively faster and amenable for further purification of uranium either by peroxide precipitation or by solvent extraction using TBP.

Megascopic and microscopic study of BHJ sample

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In the present paper a detailed megascopic and microscopic study of Banded Hematite Jasper (BHJ) sample from Gua (Jharkhand) area has been carried out. Megascopic study of the samples of the area reveals that it mainly comprises of alternate layers of hematite and jasper with micro-folds and faulted features dissecting each other. Textural and structural analysis is very much helpful in beneficiation point of view as during liberation process the coarse grains will separate out considerably, whereas micro-bands get camouflaged between iron rich and iron poor fractions. Structurally bands are parallel in nature but the concentration of iron ore in the iron rich bands is more or less uniform. In silica rich band it is highly erratic. The ore body shows complex interlocking of hematite and jasper. So it needs fine grinding to liberate the iron ore particles and jasper. Liberation study shows -300+200µ is best suited for liberation.

Study also signifies the clay percentage is minimum with respect to other, while silica percentage is too much high.

IOP grade bentonite for use as binder in iron ore pelletization

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Geologically bentonite is defined as a clay or clay stone composing largely of smectite minerals. The material has the ability to absorb water accompanied by a large increase of volume. Smectites are a group of swelling clay minerals, montmorillonite being the predominant smectite in most bentonites. Bentonite is essentially highly plastic clay containing not less than 85% clay mineral, montmorillonite. There are two types of bentonites; namely, swelling type or sodium bentonite and non-swelling-type or calcium bentonite. Sodium bentonite is usually referred to as bentonite, whereas calcium bentonite is called Fuller's earth.Owing to high green strength, resulting from its property to absorb and then release moisture, bentonite is used in iron ore pelletization. Sodium-based bentonite of 75 micron size finds suitability in iron ore pelletization for bonding by user industries. Bentonite presents strong colloidal properties and its volume increases several times when coming into contact with water, creating a gelatinous and viscous fluid. Bentonite significantly improves physical properties, i.e. green and baked compressive strength. The increased viscosity of fluids between the mineral grains in the pellets results in the production of the well-rounded, plastic pellets that can be easily handled for sizing and transport in the plant.As well as acting as a binder, the bentonite is required to absorb excess water that is not removed from the ore during processing. For this reason, Na-bentonites having a high moisture adsorption capacity are preferred by iron ore pelletizing companies. Sodium bentonite expands when wet, absorbing as much as several times its dry mass in water. Due to its water absorption capacity and colloidal property, bentonite is used as a binding additive to convert iron ore fines into pellets. . Its level of hydration and swelling depends on the type of exchangeable ions contained, with different hydrophilic and solvating power. Bentonites with Na+ as the predominant exchangeable cation exhibit a high swelling capability in water. Sodium bentonite, with sodium cation prevalence (Na+) allows water to penetrate through the platelets, forcing them apart, thus leading to swelling. In case local supply of bentonite is not available, synthetic bentonite can be prepared from calcium bentonite, by treating it with anhydrous soda ash (Na_2CO_3).

National steel policy – Challenges before iron ore producers

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In view of changed economic environment, both globally as well as domestically, Ministry of Steel has initiated the process of drafting the New National Steel Policy in place of existing National Steel Policy 2005. National Steel Policy 2005 estimated domestic steel production of 110 MTPA by the year 2019-20. The revised National Steel Policy 2008, envisages domestic steel production of the country to be 180 MTPA by 2019-20. About 2.5 tonnes of ROM iron ore or 1.7 to 2.0 tonnes of processed iron ore required for one tonne of steel production. To meet the projected steel production of 180 MTPA by 2019-20, the iron ore requirement would have to be in the order of 500MTPA which includes long term projected export contracts of around 100MT. Raw materials are crucial in determining the competitive growth of steel

industry as this is an input-intensive extractive industry. The situation calls for proportionate development expansion in adequate raw material supply to meet the demand of Indian steel Industry. The ores and minerals are site specific, non-renewable and finite. It is a challenging task for iron ore producers to meet the demand. In order to meet the demand, the iron ore producers has to face challenges like increasing the resource base, increasing production and productivity, utilisation of low grade iron ores, beneficiation of low grade fines and slimes, overcoming the infrastructure bottlenecks like roads, railways, ports, power, capital and water, human resource, handling, storage and utilisation of slimes/tails, encouragement for R&D activities, adopting environmental friendly measures and land acquisition for setting up new plants. This paper deals with all the above problems and possible solutions along with SWOT analysis.

Prediction of lanthanum recovery from Indian red mud using artificial neural network

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The assay and recovery of rare earth elements (REEs) in the leaching process is being determined using inductively coupled plasma atomic emission spectroscopy (ICP-AES) and inductively coupled plasma mass spectroscopy (ICP-MS). A neural network model to predict the effect of operational variables on the recovery of lanthanum during the leaching of Indian Red Mud by sulphuric acid is presented here. The effect of leaching temperature (35°C to 95°C), pulp densities (5% to 50%), acid concentrations (0.2M to 7M), and pH (0-5), were investigated to optimize the recovery of lanthanum (La) in 1h leaching time and constant agitation rate(200 rpm). The obtained data in the laboratory optimization process were used for training and testing the neural network. The feed-forward artificial neural network with a 4-3-6-1 arrangement was capable of estimating the leaching recovery of REEs. The neural network predicted values were in good agreement with the experimental results. The high correlations of R=0.98 in training stages, and R=0.96 in testing stages were a result of good prediction of La recovery. It was shown that the proposed neural network model accurately reproduced all the effects of the operation variables by simulation.

Session 7:Nov 15, 2014:[11:00-13:00]

Density functional theory computations for design of salicylaldoxime derivatives based flotation collectors for beneficiation of complex sulfide ores

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The presence of oxide and oxidized minerals along with sulfides in mixed sulfide-oxide ores leads to losses in metal recovery during flotation with conventional thiol reagents. Chelating type reagents such as

salicylaldoxime (SALO) and its derivatives are promising as flotation collectors for these systems. We have earlier reported on the possibility of selective separation amongst copper, lead and zinc minerals using SALO derivatives with appropriate alkyl group substitution in the main chain (CM-SALO) or side chain (CS-SALO). The chain length of the substituent group as well as its position strongly affects the selective interactions of SALO derivatives with metal ions both in the bulk as well as on the surface. We have performed first principles density functional theory (DFT) computations to study the interactions of SALO derivatives with copper (Cu), zinc (Zn) and lead (Pb) ions. The aim is to understand the underlying mechanisms governing these interactions and exploit this knowledge in the design and development of better reagents. All computations were performed using plane wave ultrasoft pseudo-potentials and Perdew-Burke-Ernzerhof (PBE) generalized gradient approximation as implemented in the Plane-Wave Self -Consistent Field (PWscf) code.The relative order of selectivity as per the computed interaction energies is found to be: Cu > Zn > Pb, thus indicating that Cu minerals can be selectively separated from Zn and Pb minerals using SALO derivatives. CM-SALO derivatives exhibit stronger interactions as compared to the CS-SALO derivatives. Our findings are consistent with experimental results reported earlier. The results are explained by HOMO (highest occupied molecular orbital) - LUMO (lowest unoccupied molecular orbital) and Lowdin charge analyses thus highlighting the immense potential of such calculations in design and development of selective reagents for mineral processing applications.

An investigation on red ochre iron ore from Bhilwara region, Rajasthan

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With India emerging as the third largest steel producer in the world and depleting high grade Iron ore resources worldwide, major thrust is on usage of low grade iron ore in Blast Furnace and other iron making units. In this respect, a basic mineralogical and metallurgical study has been done on Red Ochre Iron ore from Bhilwara region of Rajasthan. The ore has been characterized for Chemical composition, Microstructure, Phase analysis and other room temperature and high temperature metallurgical properties required for utilization in various iron making units. The ore is highly goethitic in nature and by selection of appropriate beneficiation route, extensive usage potential is foreseen. Some of the results of this study are presented in this paper.

Characterization of Sukinda chromite ores

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Chromite (FeO.Cr2O3) is the principal ore of the element chromium. Chromium (in the form of ferrochrome) is largely used as alloying elements in steels for production of stainless steels. The Sukinda area of Odisha has the largest chromite ore reserve of India. The supplied chromite ore was crushed and sieved. Sieve analysis was done to find the amount of chromite being liberated from the raw ore. Ore particles of -150 mesh size was divided into three groups and subjected to characterization by chemical analysis, heat treatment and optical petrography. The first group, i.e. raw ore was further divided into two sets, one was beneficiated and the other was non-beneficiated. These two sets were divided into numerous samples for Chemical Analysis to find the composition of chromium and iron. There was

difference in the composition of the beneficiated and non-beneficiated ore. The second group was then heat-treated to observe the thermo-chemical changes occurring in the sample. Samples were heated in air and in oxygen at various temperatures for varying periods of time in a horizontal tube furnace. Then the heat treatment was limited to a range of temperature between 1000°C to 1150°C to closely observe the mass change in a particular time interval. The third group was subjected to Optical petrography to study about the ore characteristics after heating at 1400°C for one hour. The samples were polished for a specific period of time till its microstructure was visible under the microscope for micro-structural studies. Pictures were taken from top, middle and bottom part. Some samples of the sets were kept for other characterization techniques and further studies. The information obtained helps in mineral identification, phase identification and identification of phase changes due to temperature.

Synthesis of rare earth borides

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Rare earth borides are potential materials for many advanced applications. Due to high neutron absorption they are candidate for control rod material in nuclear reactors. LaB6 and CeB6 are used as electron emitter in electron beam accelerators and other devices such as electron microscope. These borides have high hardness, high melting point, high thermal conductivity and low thermal expansion. Processing of these materials is difficult and very little information is available in open literature. This paper presents results of investigations carried out on synthesis of rare earth boride powders. Rare earth borides (EuB6, LaB6, CeB6, SmB6, NdB6,) were synthesized by boron carbide reduction of respective oxides according to the following reaction.

$Re_2O_3 + B_4C \rightarrow ReB_6 + CO$..... [Re= Eu, La, Ce , Sm, Nd].....(1)

Rare earth oxides and boron carbide powder were mixed in motorized mortar and pestle. The mixed powder was pelletized and heated in induction furnace to high temperature in vacuum for 2 hour. The reacted pellets characterized by X-ray diffraction and chemical analysis. Reaction at temperatures less than 1400 oC resulted in incomplete reaction and the product was analysed to contain higher oxygen content and other phases such as rare earth borates. Reaction at 1500°C, resulted in the formation of single phase rare earth borides. By using these technique borides of Eu, La, Ce, Sm and Nd were synthesized.

Session 8:Nov 15, 2014:[14:00-16:00]

Recycling of iron slime as an additive for making bottom ash bricks

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The usability of Iron Slime (IS) waste as an additive material for making Bottom Ash (BA) bricks, were investigated. BA was collected from Thermal Power Plant, Jharkhand and IS from iron ore beneficiation plant located in Orissa, India. Collected raw materials were examined for chemical, size fraction, phase analysis etc. Bricks of 30mm×30mm×30mm size were manufactured using a laboratory scale hydraulic press (under 20 MPa pressure) with varying different fineness (1-0.2mm; 0.75-0 mm) of BA with different

amount of IS content (0-10 %) having surface area of 2200m2/Kg at optimum moisture level. After drying, bricks of different composition were fired for 2 hours at different temperature of 1000, 1100 and 1200 °C. The different properties of fired bricks were evaluated such as density, porosity, crushing strength etc. Phase and micro-structural analysis of the product were also examined by using XRD and SEM analysis.Results of Cold Crushing Strength and porosity of the fired bricks show increasing and decreasing behavior respectively with increasing firing temperature, fineness of BA and IS content. SEM micrograph shows pores structure for coarse BA bricks as well as presence of low melting constituents inside the bricks. XRD analysis of 1000°C fired bricks revealed that the formation of minor amount of calcium silicate, aluminium silicate and also Magnetite. With increasing firing temperature, the formation of iron silicate followed by iron aluminium silicate occurred which imparts the strength inside brick. Maximum Cold Crushing Strength of 165.29 Kg/cm2 was achieved for the bricks having Coarse BA: Fine BA =1:3 with 10% IS, fired at 1200 °C, whereas maximum porosity value was achieved up to 45.8 % having 0% IS, fired at 1000°C. However, the optimum strength having comparable porosity (33 to 32%) was achieved 56 to78 Kg/cm2 in the product made at 1200°C, having Coarse BA: Fine BA =1:1 ratio with 5-10% IS addition.

Coal and coal ash characteristics to understand mineral transformations of Talcher coalfields

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In India coal is a major fuel source which is used as main feedstock for power generation industry and petrochemical industry. Coal has played a significant role in the development of civilization. A large variation of properties and composition of coal has been observed when sampled are taken from different sources. The results of an AFT analysis consist of four temperatures, namely initial deformation temperature, softening temperature, hemispherical and flow temperature, which are intended to reflect phases of the 'melting' process. The fusibility of the ash as function of the content of the eight principal oxides frequently found in coal ash, i.e., SiO2, Al2O3, TiO2, Fe2O3, CaO, MgO, Na2O and K2O. The objective of this study is to determine mineral transformation and agglomeration or slag formation during combustion or gasification, volatile contain and carbon contain from the sample of coal taken from Talcher coalfields. Factsage is used for analysis of different phase and decomposition of different minerals present in coal sample. From the results obtained on the coal samples, it has been observed that the major mineral phases present in coal samples are quartz, cordierite, hematite, apatite and rutile. An abundance of silicon rich particles, which are probably quartz has observed. The results of proximate analysis for different depth of coal sample taken from talcher coal field shows different ash%, moisture% and VM (volatile material) %. From analysis it has been observed that melting point of mullite is high, while rutile fully melted near about 1250 0C.

Electro oxidation of complex sulphides

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Complex Sulphide Ores (Cu-Pb-Zn) are often found to be close mutual association with each other and with the nonmetallic gangue. The beneficiation experiments showed that it would be very difficult to separate these (Cu-Pb-Zn) minerals with traditional ore beneficiation methods. In the present work, leaching of complex Sulphide ores in sulphuric acid was investigated. The lab-scale experiments were conducted to investigate the influences of pulp-density, electrolyte concentration, particle size, current density, stirring, temperature and time on recovery of Cu and Zn. The leach liquor obtained after electrolysis was subjected to Atomic Absorption Spectroscopy analysis for the recovery of minerals. The experimental results show that under optimum conditions, recovery yield of copper and Zinc reaches 54.05% and 29.14% respectively.

JSW journey towards clean coke technology through CDQ

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JSW Steel Ltd. has embarked upon a new approach to conserve both heat energy and water resources and abate air and water pollution associated with the conventional wet quenching process during manufacture of metallurgical coke. Coke Dry Quenching (CDQ) is a system to quench the red hot coke and recover sensible heat of hot coke by using inert circulating gas. The implementation of the CDQ assists in reduction of carbon emissions, energy and water consumption and aids in power generation in the operational processes. This steam produced without any additional energy is used for the generation of electric power. The distinct advantages of the system include energy conservation, pollution reduction, coke quality improvement and full use of resources. Due to the concern of environment protection and energy crisis, it is important to develop a new CDQ technology which can protect the environment and save energy resources. Along with the conventional CDQ, additional power is generated through BF Gas injection to CDQ boilers which has been implemented for the first time in CDQ's as a cutting edge technology. This paper describes the journey of JSW towards clean coke technology and water conservation through CDQ technology.

Studies on effect of usage of inert-rich coal in coal blend on coke properties through pilot oven carbonization tests

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The usage levels of imported coking coal in SAIL plants mostly varying from 78% to 90% due to inferior quality and low availability of indigenous coking coals. Thus, a study was carried out with an indigenous coking coal from Tarmi having very high inert content for using in SAIL blends through pilot oven carbonization tests. The Tarmi OCP coal has high raw coal ash (33.1%), it yields moderate clean coal (49.6%) and has very poor coking properties (CSN 1, LTGK coke type F and no fluidity) due to very low content of vitrinite (15.1%). Coal was de-shelled at 1.70 specific gravity and clean coal samples were produced at ash contents of about 13%, 16% and 19% respectively. The properties of clean coal samples of

Tarmi showed very low vitrinite contents (16.1% to 18.7%) and very poor coking properties. Deep beneficiation to about 13% ash did not produce much improvement. It remained a source of organic inert. Pilot oven carbonization tests using Tarmi clean coal at 10% level replacing Kathara and at 13% level replacing Moonidih showed no deterioration in M10 index up to 16% ash clean coals and some improvement in M40 index. Stamp charging could use 20% Tarmi coal of 13% ash content with improvement in M10 index by about 4 units. The results demonstrated that use of organic inerts-rich coal at lower ash content can improve coke properties. Moreover, stamp charging process can accommodate larger inerts than top charging process.

Met coke moisture reduction below 2.5% by using coke oven based partial waste flue gas

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JSW Steel Limited (JSWSL), Salem Works, is an integrated steel plant, which operates a Coke oven plant, which produces 0.5 million tons per annum of metallurgical coke through three non-recovery batteries Comprising 120 ovens and generates 30 MW power. JSWSL also operates two blast furnaces with a combined production capacity of 1.0 million tons per annum of hot metal. The metallurgical grade coke produced from captive Coke oven plant is used in both the Blast furnaces for iron making. The coke was being produced at 5.5 to 6% moisture level. The Coke oven endeavors to maintain the coke moisture content as low as possible to facilitate the Blast Furnace to operate at improved efficiency of fuel rate. An innovative & novel idea emerged that utilization of the sensible heat of the hot waste flue gases generated in the coke oven process which was partly being let out through the chimney. The successful installation of Coke Drying unit at JSW coke oven premises is a unique and first time in Indian steel industry. New 75 TPH coke drying unit (250 M3) was commissioned on 29th Mar-2013. The present paper describes the safe & Successful operation of coke drier which played vital role to achieve coke moisture quality below 2.5% consistently.

The influence of coke porosity on the moisture adsorption capacity and hot strength property of metallurgical coke

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As blast furnace operates with constant coke base, steady and consistent coke moisture is paramount for smooth blast furnace operation. Wide fluctuation in coke moisture leads to undue variation in silicon and sulphur in hot metal. JSW Steel Limited, Salem Works (JSWSL) operates three non-recovery batteries comprising 120 coke ovens to produce 0.5 million tons of metallurgical coke, which uses wet quenching technology to cool red hot coke. During quenching, coke adsorbs moisture due to the presence of porosity. Porosity experiments revealed that, less porous coke exhibit better hot strength properties (CRI & CSR) than highly porous coke. Coke porosity depends on inherent characteristics of coals and the process of coke making. Due to non-availability of captive mines, coals are being sourced externally, but they are

found to have wide variations in properties. The present paper discusses the partial control of coke porosity by fine-tuning process parameters and the installation of a coke drying system to obtain coke with consistent moisture.

Experience on usage of higher percentage of imported coal in coke making at Bhilai Steel Plant (BSP)

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Coke making is the process to convert coking coal, into metallurgical coke through a series of operations. Out of these operations crushing and blending plays a vital role in coke making. Blending plays a vital role in producing good metallurgical coke. Blending is a process of mixing the different types of coal, in a predetermined ratio to reduce the ash percentage of the blend coal, keeping the other coking properties intact. In Bhilai steel plant coal blend is prepared from Indigenous coal named as Prime coking coal (PCC),Medium coking coal(MCC) and imported coal consisting of hard and soft coal. There are 3 types of hard coal i.e., Hard1, Hard2, Hard3 and a soft coal (S1) used at BSP. In Bhilai steel plant in general 78% imported and 22% indigenous coal was used. But in this year 2014-15 due to shortage of indigenous coal supply, it became difficult to keep the coal blend as usual. Therefore, imported coal in blend was increased from a level of 78-80% to 85, 89, 91 and 100%. This was a challenging task to increase the % of imported coal while maintaining the other operational parameter like shrinkage, pushing amperage at normal level. This paper deals with experience of different combination of imported hard and soft coal. As a result of increasing the imported coal percentage, coke ash reduced up to 12.1%, although as a side effect microfines in blend get increased and it was reflected as increasing trend of Quinoline insoluble % (Q.I) in tar. On other hand coke quality parameter like Micum index (M10) first increase then after certain % show decreasing trend while M40 show continuous increasing trend. Hot strength properties like CSR improved up to a certain % then decreased by increasing the imported coal percentage and then again it increases. However reduction in coke ash and improvement in coke strength result reduction in slag rate and coke rate in the Blast furnace.

NON DESTRUCTIVE TESTING

Invited talks

Session 1: Nov 13, 2014: [15.30 - 17.30]

Advanced NDE Methods for flaw and materials characterisation

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An overview of several Nondestructive Evaluation (NDE) modalities will be covered that ranges from very small size features through micro/nano-scale imaging to imaging of large structures. The imaging modalities that allow for hidden region imaging as well as non-contact ultrasonic imaging techniques will be described with examples from industrial application. While the use of the physics of diffraction allows for the improvement in sizing, the use of phased arrays provides improved speed of inspection. The generation and reception of different wave modes and their exploitation for improved imaging will also be discussed. Some newer techniques including wave visualisation techniques, air coupled ultrasound, Guided wave imaging, structural health monitoring, etc. will be described. Applications in the Oil and Gas, Aerospace, Nuclear, Defense, and manufacturing industries will be uses as examples of application of ultrasonic imaging techniques. In this presentation, the various applications in the field of NDT (composite inspection, adhesive bond inspection, corrosion detection, etc.), Materials Characterisation, Structural Health Monitoring, and Process Measurements will be discussed. The ultrasonic guided waves, unlike bulk wave modes like longitudinal and transverse, are a manifestation of geometrical confinement of acoustical waves by one or more boundaries [1, 2]. In many instances, these waves travel long distances, depending on the frequency and mode characteristics of the wave, and follow the contour of the structure in which they are propagating. Usually, these waves not only propagate along the length of the structure but also cover the entire thickness (for plates) and circumference (in the case of cylinders and rods). The use of guided wave modes is potentially a very attractive solution to the problem of inspecting the embedded portions of structures because they can be excited at one point on the structure propagated over considerable distances, and received at a remote point on the structure, in a pitch-catch mode, as schematically illustrated in Figure 1 for an elbow pipe. The received signal contains information about the integrity of the material that lies between the transmitting and receiving transducers. Alternate approaches, where the receiving and transmitter are co-located, similar to a pulse-echo method is also possible.

Inspection of pipelines in process industries is essential as they are prone to corrosion. In-service inspection can be conducted using traditional NDT methods such as radiography, ultrasonics, etc. More recent techniques such as the use of ultrasonic guided waves propagating in both axial and circumferential directions can lead to methods for inspecting such pipes with less cost and time. Additionally, inaccessible regions such as corrosion between pipes and sleepers can be addressed with circumferential guided waves.

In recent times nonlinear ultrasonics is being extensively used to explore its unique abilities in material characterization. In this technique, change in nonlinear ultrasonics (NLU) parameter measured from the harmonics produced during a finite amplitude ultrasonic wave propagation is used to identify the sub-structure changes in a material. The cause of these substructure changes is due to change in dislocation

density, nucleation and growth of precipitates and phase transformation. Conventional techniques like TEM, SEM and SAD used to study the substructure changes are restricted to study the changes on the surface or thinner sections of a material and mostly are found to be destructive in nature. In contrast, nonlinear ultrasonic technique can be used to study the bulk materials and it is non-destructive.

The use of Air Coupled Ultrasonics, particularly for industry applications, is fast becoming a reality largely due to the advent of highly efficient transducers and the selection of wave modes that have lower acoustic impedances. The coupling through air allows for a wider use of the ultrasonic NDT technologies when compared with the conventional couplant based methods. The increased speed of inspection, the portability of the systems (since couplants are not required), the sensitivity to defects, inspection of hidden regions, etc. are some of the key advantages of the air coupled ultrasonic techniques. The dis-advantages of the technique include the limitation of the frequency of operation and the requirement of high voltage for excitation. In this paper, the application of Longitudinal, Shear, and Lamb wave modes that are excited and received by air coupled ultrasound transducers will be discussed. The technique is applied to aerospace components, composite pipes, adhesive bonded components, among others. The defects considered include weld inspection, delamination characterisation in composite structures, interfacial weakness in bonded components, etc. that are made with metals and or composites.

Structural Health Monitoring of composites is performed using ultrasonic methods in a three prong approach. The strategy for the long term asset management of these mission critical components will depend on the type of component, the access for sensors and the requirement for health monitoring. New technologies developed at the Centre for Non-destructive Evaluation at the Indian Institute of Technology will be extensively discussed.

Contributory papers

Session 1: Nov 13, 2014: [15.30 - 17.30]

Qualitative and quantitative investigation of microstructures within porous minerals/rocks/coals by using very high resolution X-ray micro-CT imaging

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Today's high-resolution X-ray CT with its powerful tubes and great detail detectability lends itself naturally to geological and petrological applications. Those include the non-destructive interior examination and textural analysis of rocks and their permeability and porosity, the study of oil occurrences in reservoir lithologies, and the analysis of morphology and density distribution in sediments – to name only a few. Especially spatial distribution of pores, mineral phases and fractures are important for the evaluation of reservoir properties. The possibility to visualize a whole plug volume in a non-destructive way and to use the same plug for further analysis is undoubtedly the most valuable feature of this type of rock analysis and is a new area for routine application of high resolution X-ray CT. All presented geological CT volume evaluations were performed with GE's phoenix nanotom, a 180 kV/15 W nanofocus CT system tailored specifically for extremely high-resolution scans of samples weighing up to 3 kg with voxel-resolutions

down to < 300 nm. In our first sample we will show a typical reservoir rock scanned with 1 µm voxel size to characterize the pore space and to extract information about the distribution of mineral components. The segmented in-situ porosity could be easily used for fluid flow modelling purposes, to predict permeabilities and complex flow processes within these structures.Next, a very porous pyroclastic rock has been examined at a resolution of 5 µm. The data set has been analysed with the Avizo software tool XLab Hydro. The resulting velocity field can be visualized whereas the colour mapping visualizes the velocity's magnitude. The resulting volume data can as well be used to produce surface data for any CAD application and furthermore for FEM modelling for hydrogeological purposes.

Frictional heating model for lock–in-frequency modulated ultrasound thermography: an ext generation advanced thermal NDT technique

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The newly discovered Ultrasound lock-in thermography(ULT) technique circumvents the drawbacks of conventional NDT techniques, which include slowscanning systems like conventional ultrasonic inspection and techniques hampering safety regulations example laser radiation, x rays etc. This novel technique combines remotes canning with selective response to faults, for quick and efficient large area defect detection, from a safe distance, attesting concrete potential for direct industrial applications. Defects differ from their surroundings by their mechanical weakness and cause selective thermo elastic effect by friction, when subjected to ultrasound i.e.mechanical waves. When the amplitude of ultrasound is modulated (Lock-Inmode), the local temperature near the defect modulates coherently. Consequently, the defect becomes a transmitter of thermal wave, which further is detected via Lock-In Thermography at the surface of the inspected component, using a thermal camera. The novel research work carried out at BHELR & DCENTRE, Hyderabad (first to bring this technique in India), lead to successful detection of hard to detect vertical crack (deliberately created) in induction pressure welded boiler tube using U L T technique. To increase the efficiency of the detection process, the frequency and amplitude of the applied ultra sound wave was modulated. For better signal to noise ratio, Phase contrast mode was used to characterize the images, without false detections. As this technique is very recent and lacks full in-depth understanding, a first time theoretical model quantifying the ultrasound sound energy distribution inside the sample is proposed. The theoretical model, based on heat transfer and wave theory, reveals thermo-elastic effect of a bove energy distribution as the chief cause of detection. MATLAB program was coded to numerically model and corroborate theproposedquantification. Remarkably, the results obtained were in good agreement when compared with the actual experimental data obtained in software interface of the thermal Camera, used during the actual experiment.

Characterization of microstructural changes in a β-titanium alloy using ultrasonic NDE techniques

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The present study reports a comprehensive characterization of various microstructural features such as volume fraction of α and β phases, interfacial strain and texture in a β-titanium alloy (Ti-10V-4.5Fe-1.5AL) using linear and nonlinear ultrasonic measurements. The β-annealed specimens were heat treated in the temperature range of 823 K to 1173 K at an interval of 50 K for 1 hour, followed by water quenching. Ultrasonic longitudinal and shear wave velocities were found to increase with decreasing heat treatment temperature below the β transus temperature. The increase in ultrasonic longitudinal and shear wave velocities is attributed to the increase in the volume fraction of α-phase, which has a higher modulus compared to the β-phase in titanium alloys. Poisson`s ratio, calculated using the time of flights of ultrasonic longitudinal and shear waves, exhibited the opposite behavior i.e. decrease with increase in the α-phase volume fraction. Ultrasonic velocities and Poisson`s ratio exhibited linear correlations with the α phase volume fraction. These relationships can be exploited for quantitative estimation of α/β phases with an accuracy of ± 4 % in the heat treated β-titanium alloy specimens. Ultrasonic attenuation is found to decrease with the precipitation of α-phase. This is attributed to the decrease in ultrasonic scattering with precipitation of α-phase due to randomization of β-grain and decrease in β-phase volume fraction having higher damping. Further, acoustic nonlinearity is found to be essentially governed by the interfacial strain in the alloy. The present study demonstrates the quantitative correlations between various ultrasonic parameters and the volume fraction of α/β phases in the heat treated β-titanium alloy specimens and provides an in-depth understanding of the interaction of ultrasonic waves with microstructural features in a β-titanium alloy.

Non-destructive evaluation of nuclear fuels and structural components during post irradiation examination

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The fuel and structural components used in nuclear reactors are subjected to stringent quality control during fabrication. These components suffer degradation during reactor operation under very hostile atmosphere of fast neutron flux, high temperature and corrosion. To evaluate the condition, many of the reactor components are brought to the hot cells for a detailed post irradiation examination (PIE). NDE plays an important role in the PIE of these components, being the primary method by which the integrity of the components is assessed. The failed fuel bundle, which is highly radioactive, is brought to the hot cells for detailed investigation to find out the primary cause of failure. The first stage visual examination is done through wall periscope and windows to examine externally the general condition locating any gross abnormal feature and to ascertain the identity of the fuel assembly. A special remote laser cutting and dismantling unit is used to separate individual fuel pins for other NDT examinations. The liquid nitrogen-alcohol test is carried out on individual pins to segregate the leaky fuel pin. The region of interest is identified for non-destructive testing like profilometry, gamma scanning, neutron radiography, eddy

current and ultrasonic testing. The presentation will outline various NDE techniques with some specific examples of relevance to nuclear fuels and structural components.

High resolution industrial CT systems: advances and comparison with synchrotron-based CT

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Nowadays, X-ray tube-based high-resolution CT systems are widely used in scientific research and industrial applications. Compact XCT systems are available that can reach resolutions down to 1 µm and below. But the potential, convenience and economy of these lab systems is often underestimated. The present paper shows the comparison of sophisticated conventional µCT with synchrotron radiation-based µCT (SRµCT). The different aspects and characteristics of both approaches like spatial and density resolution, penetration depth, scanning time or sample size is described in detail. Beside this, the advances in technology of industrial high resolution CT systems are shown. The paper also presents recent advances in the area of industrial high resolution CT systems from phoenix product line of General Electric. All major parts are designed to make the system extremely stable during the data acquisition process. So, the system is equipped with granite base and very precise rotation unit. The unique nanofocus tube technology with build-in cooling system stabilizes the tube and at the same time the diamond based target allows high photon flux at very small focal spot sizes. The unique detector with excellent contrast resolution and SNR is also thermally stabilized. Also, the user friendliness is increased through the fully automated process chain starting with detector calibration and going through acquisition and data reconstruction process with automated volume data evaluation. The application results of this new technology show its high potentials for usage of the state of the art laboratory systems in the industrial and scientific application fields of material research, metrology, petro-industry, etc. To compare the potentials of laboratory based CT with synchrotron based CT, different samples were used: e.g. a low-carbon steel sample, and an aluminium multi-phase sample ($AlMg_5Si_7$) and some other. Concerning measurement costs, scanning volume, accessibility and user-friendliness sub-µXCT has significant advantages in comparison to synchrotron-XCT.

Use of thermoelectric power to characterise different materials

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Precise Non Destructive Testing techniques are indispensable for modern day applications when special emphasis has been given for the manufacturing technology. In this area high level of quality control is needed to deliver high end product. Various techniques like ultrasonic, electromagnetic waves are already in use as quality control tools. In this work, attempt has beenmade to use thermoelectric power as a tool to characterize materials. In this technique the two ends of the component are maintained a temperature

gradient and consequently a voltage generate at the two ends which is measured by a nanovoltmeter. Two thermocouples are used to measure the temperature at the two ends of the test piece. Two copper blocks are attached at the two ends to maintain the constant temperature at the hot and cold junctions. As a result the thermo-emfare generated across the test object with respect to the Cu and hence measured Seebeck coefficient or thermoelectric power (TEP) is the relative one with respect to Cu. The developed experimental set up has been calibrated for Iron, Ni and Al samples and the observed relative Seebeck coefficient wasfound close to the literature value. The experiment was extended for various types of steel like cold rolled 304SS where different volume fraction of martensite are formed due to stress induced deformation and also to various heat-treated duplex steel which contain varied percentage of ferrite and austenite. The preliminary results are very encouraging and it appears that the measurement of TEP can lead to a powerful tool for characterisation of materials in a non-invasive way.

NUCLEAR MATERIALS

Invited talks

Session 1: Nov 15, 2014: [14.00 - 16.00]

Advances in manufacturing technology for nuclear and strategic materials

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Nuclear Fuel Complex is involved in manufacture of reactor structural and cladding components in different shapes and size for Pressurized Heavy Water Reactors (PHWRs), Boiling Water Reactors (BWRs), Prototype Fast Breeder Reactors (PFBRs) and other similar applications. Fabrications of reactor core components are critical primarily due to type of material selected for nuclear reactors core components and at the same time requirement of stringent control in chemistry of alloy, dimensional tolerances, shapes, mechanical properties, metallurgical properties etc. In recent years advances in metal forming technology in NFC has led to production of new products such as production of extremely thin wall calandria tubes by seamless route, square channel by pilgering, hexacan by seamless route etc. Technological developments achieved hitherto has also made NFC capable of producing number of different shapes such as tube of circular shape in inside and hexagonal shape on outside and vice versa, square shape in inside and circular shape on outside and vice versa etc. There is a wide range of materials which have applications in nuclear and strategic sector. NFC has successfully dealt with Zirconium alloys to nuclear grade stainless steels, Special steels such as Maraging steels, Incloy-800. In the recent times NFC has successfully ushered into new era by tubular product developments of technologically challenging alloys such as superalloys, P/M Based Oxide Dispersion Strengthened (ODS), Nb-based alloy, extra long length tubes with U-bend, bi-metallic tubes etc. Development of these products as import substitute has brought NFC on the forefront of material processing in the country as well as world over.Major alloy tube and component developments include hot deformation by forging and extrusion followed by variety of cold working processes such as pilgering, drawing, rolling and swaging. Manufacturing processes also include various intermediate and final heat treatment processes in vacuum or controlled atmosphere which are specific to alloy under development and their mechanical and metallurgical property requirements.Bulk deformation stage such as forging and extrusion are the major steps for primary shaping of the material. For technologically challenging materials, a suitable methodology has been developed for hot deformation of the alloys. Alloys are processed in well established deformation window of temperature and strain rate. These deformation windows are established by exhaustive experimentations and study of plastic flow behaviour by uniaxial hot compression. Flow behaviour studies have been extended for FEM simulation before actual trials. This methodology provides greater yield by eliminating extensive shop floor trials and optimum microstructural condition of hot worked products further amenable for cold working. Using combination of different cold working operations, NFC has produced seamless tubes of different sizes and shapes such as extremely thin walled Calandria tubes, pressure tubes of optimised metallurgical and mechanical properties, hexcans of different sizes, square channels, extremely low OD and thin wall tubes for nuclear and strategic applications. These critical product developments in NFC are backed by state of art quality assurance NDT techniques.

Development of advanced core structural materials for Indian sodium cooled fast reactors

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Economic competitiveness of sodium cooled fast reactors (SFRs) largely depends on the performance of core structural materials, i.e., cladding and wrapper materials of the fuel subassembly, which are subjected to intense neutron irradiation at high temperature during service. These operating conditions lead to unique materials problems like void swelling, irradiation creep and helium embrittlement. The operating temperatures are high and stresses of sufficient magnitude are present, and hence, creep strength, tensile strength and tensile ductility are also important requirements for the core structural materials. In order to increase the burnup of the fuel and thereby reduce the fuel cycle cost, it is necessary to employ materials which have high resistance to void swelling as well as better high temperature mechanical properties. Indian sodium cooled fast reactor program began with the commissioning of the 40 MWt Fast Breeder Test Reactors (FBTR). Core structural material in the case of FBTR operating with mixed uranium-plutonium carbide fuel is 316 austenitic stainless steel. The steel has been imparted with 20% cold work to increase void swelling resistance, which also increases creep strength. For the 500 MWe Prototype Fast Breeder Reactor (PFBR) with mixed uranium-plutonium oxide as the fuel, which is in the advanced stage of construction, 20% cold worked alloy D9 (15Cr-15Ni-Ti austenitic steel) has been selected as the cladding and wrapper material. The alloy D9 is selected because of the fine intragranular Ti(C,N) particles along with forest dislocation introduced through cold work increase void swelling resistance as well as creep strength of the alloy D9 over that in the 316 steel. The target burnup for PFBR having alloy D9 as core material is 100 GWd/t with about 85 dpa neutron fluence. India has a long-term plan to develop fast reactor core structural materials for enhanced fuel burnup. In the first stage of development, the target burnup is enhanced to 130 GWd/t by developing modified version of the alloy D9 through optimization of Ti, Si and P contents for better swelling resistance and creep strength. The alloy D9 having optimized composition of 0.26 wt.% Ti, 0.75 wt.% Si, 0.04 wt.% is designated as Indian Fast Advanced Core-1 (IFAC-1) alloy. The alloy will be used in 20% cold worked condition. Ferritic steels are inherently resistant to neutron irradiation induced void swelling. However, they cannot be used for fuel cladding tubes because of their inadequate creep strength at the desired operating temperatures in reactors with oxide fuel. The use of 9Cr-1Mo ferritic steel is being considered as wrapper application, where creep strength is of secondary importance because of relatively lower service temperatures. However, irradiation induced embrittlement of the ferritic steel is a concern. The minimum content of silicon in 9Cr-1Mo ferritic steel has been arrived at around 0.4 wt.% for the higher initial toughness to reduce the irradiation embrittlement. In the second stage of development of mixed uranium-plutonium oxide fuel based fast reactor, the 9Cr-1Mo ferritic steel will be used for wrapper along with the IFAC-1 SS as cladding material to increase the burnup to more than 150 GWd/t. Creep resistance of ferritic steels has been improved through the dispersion of highly stable nano size oxide particles. The yttria dispersed 9Cr-ODS (oxide dispersion strengthened) ferritic/martensitic steel fuel cladding tubes have been developed through powder metallurgy route employing thermo-mechanical processing. The 9Cr-ODS steel has high temperature creep strength comparable to the alloy D9. Further, to minimise dissolution of ODS steel during spent fuel reprocessing, efforts are underway to develop high-chromium ferritic ODS steel. A complexity of developing the ferritic-ODS steel over that in the ferritic/martensitic-ODS involves the extent of deformation in each stage of pilgering of extruded tubes and the heat-treatment process to be followed to soften the cold-worked steel during the intermediate stages of cold pilgering process. In the third stage of development, mixed uranium-plutonium oxide fuel based fast reactor, with a view to increase the burnup to 200 GWd/t, higher ODS- steels will be used as cladding material along with 9Cr-1Mo ferritic

steel as wrapper material. Parallel activities have been initiated to develop mixed uranium-plutonium metallic fuel based fast reactors to enhance the breeding ratio. Since the operating temperature of metallic fueled reactor is lower than that of the oxide fuel, modified 9Cr-1Mo ferritic steel will be used as the cladding material with 9Cr-1Mo ferritic steel as the wrapper material. The presentation illustrates the challenges and the progress made towards development of core structural materials for India's sodium cooled fast reactor programme.

Microstructural refinements for better performance in demanding environments in the nuclear sector: Case studies of Zr and Nb based alloys

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Stringent specifications of microstructure and purity are required for high performance alloys in use in the nuclear sector. Besides better mechanical properties and corrosion behaviour for applications in nuclear reactors, the material has to be well proven in radiation environment. This calls for explicit structure property correlation through in depth understanding of evolution of microstructure and texture in alloys. In addition to alloy design in terms of composition, the final microstructure is dependent on the nature of heat treatments and deformations imparted during processing. The selection of these will be facilitated by the vast experience and knowledge in phase transformation in alloys, their deformation behaviour and textural development. The aforementioned technological developments are therefore synergistically related to the basic scientific studies involving different physical and mechanical metallurgy aspects. Besides experimental studies, a better understanding can be achieved by using modeling. The initial empirical approach has been replaced by an ab initio approach in many of the steps involved in the aforementioned efforts. With the availability of aberration corrected electron microscopes, high resolution EBSD and tools for studying deformation behaviour at different length scales, the country is very well equipped to achieve the aforementioned objectives. This presentation showcases results of some of these activities. In Zr based alloys besides microstructural characterization by these highend instruments, the evolution of texture, which is very important for these alloys, has been carried out by in-situ texture determination during deformation by doing neutron diffraction along with deformation. These studies have also been used for understanding the slip mechanism, the process of twinning and the effect of alloying additions on deformation mechanism through crystal plasticity modeling. Results of these investigations along with microstructural characterization have been presented in this study. In the case of Nb based alloys, the characterization of microstructure has been done by using the aforementioned tools to achieve a complete understanding of the structure of the phases at an atomic level. Results of irradiation damage by neutron irradiation and proxy ion irradiation has been included in the presentation. A flavor of the *ab-initio* studies has also been given in this presentation.

Contributory papers

Session 1: Nov 15, 2014: [14.00 - 16.00]

Effect of normalization temperatures on ductile-brittle transition temperature of a modified 9Cr-1Mo steel

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The 9Cr-1Mo steel used in fast breeder reactor is exposed to irradiation during service which severely affects the dynamic fracture resistance by increasing the ductile to brittle transition temperature (DBTT). Thus, even at room temperature, the steel can become brittle and prone to cracking. The microstructural features, especially prior austenite grain size (PAGS), martensitic lath and packet size originating from prior processing routes has an effect on the toughness properties of 9Cr-1Mo steel, which is yet to be understood fully. In the present investigation, material is subjected to three separate normalization treatments i.e., at 950°C, 1025°C and 1100°C from As-received (Normalized at 1050°C and tempered at 750°C) condition. After subsequent tempering treatment, the impact toughness in terms of upper shelf energy (USE) and ductile-to-brittle transition temperature (DBTT) has been evaluated using Charpy impact testing (with $10\ \text{mm} \times 10\ \text{mm} \times 55\ \text{mm}$ specimens) following ASTM E23 procedures. A substantial improvement in USE (~20 J) and reduction in DBTT (~20°C) has been noticed for the 1025°C treatment. The result has been analysed in terms of change in yield strength and ductility evaluated with tensile tests. Further, effects of microstructure, precipitate and texture on the change of DBTT have been studied with TEM and EBSD analysis. The results have been attributed to the smaller 'effective grain size', higher fraction of high-angle boundaries, presence of beneficial γ-fiber texture and dissolution of the pre-existing coarse precipitates in the reheated sample as compared to As-received material.

Fracture toughness evaluation of heat-treated Zr-2.5Nb alloy using load normalization and DCPD techniques

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Recent work showed that the heat-treated Zr-2.5Nb pressure tubes (quenched and aged) exhibited a lower rate of in-reactor deformation during reactor operation and also immune to impurities like chlorine and phosphorus as compared to those fabricated using the CWSR (cold work stress relieve) route. One of the important stages in the fabrication of heat treated pressure tube material is solution heat treatment (SHT), which governs the microstructure and hence, the mechanical properties of pressure tubes. Usually, SHT is carried out in two phase ($\alpha+\beta$) region at a temperature closer to the β transus. In present work fracture toughness of heat-treated Zr–2.5Nb pressure tube alloy was determined at 25 and 300 ^{o}C using load normalization technique (LNT) and a comparison between the crack initiation and the crack propagation toughness parameters is presented with the values obtained using direct current potential

drop (DCPD) technique. The dependence of error in J-parameters determined using LNT to that obtained using DCPD on temperature and final crack extensions was also investigated. For all temperatures the error exhibited weak dependence on final crack extensions. At all temperature, the error in initiation toughness ($J_{0.15}$ and J_Q) was about 30-40% whereas error in propagation toughness (J_{max} and $J_{1.5}$) was practically unaffected.

Ratcheting fatigue behavior of Zircaloy-2

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Ratcheting fatigue is a phenomenon which leads to reduction in fatigue life of structural components due to cycle-by-cycle accumulation of plastic strain. Zircaloy-2 is widely used as structural material in core components like pressure tubes and fuel cladding in pressurized light and heavy water nuclear reactors. They experience plastic strain cycles due to power fluctuations in the reactor which cause low cycle fatigue (LCF), a life limiting factor for them. Ratcheting fatigue is another important factor to consider in structural components of pressurized pipes in the reactors subjected to cyclic loading with mean stress. It was found in SS304 that ratcheting strain accumulation increases with the increase in mean stress and stress amplitude in the piping components subjected to asymmetric stress cyclic loading and finally results in ballooning and deformation of pipes. Earlier work by authors was focused on LCF behavior of Zircaloy-2, at strain rates from 10^{-2} to 10^{-4} s^{-1} at different strain amplitudes between ±0.50% and ±1.25% at room temperature. Fatigue life was decreased with lowering of strain rate from 10^{-2} to 10^{-4} s^{-1} at all the strain amplitudes studied. While there was cyclic softening at lower strain amplitudes, cyclic hardening was exhibited at higher strain amplitudes at all the strain rates. In present investigation, ratcheting fatigue in Zircaloy-2 is studied as a function of mean stress and stress amplitude. The deformation behavior is analyzed using electron microscopy.

Effect of tungsten and zirconium on microstructure and mechanical properties of niobium

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Niobium based alloys are being considered for high temperature structural aerospace application because of their high melting temperatures and excellent workability. However these alloys are susceptible to high temperature oxidation and exhibit poor long term creep. Though development activities of these Nb-based alloys were undertaken during 1950s, 1960s & early 1970s and several potential alloy compositions were identified, the information available in the literature regarding the structure-property correlation is scanty. Therefore in this research work the effect of W and Zr on structure and mechanical properties of Nb has been investigated. High purity Nb, Nb-10W and Nb-10W-2.5Zr alloys were prepared by electron beam drip melting. Consumable electrodes for EB melting were prepared from high purity Nb rods and W & Zr wires by TIG welding. EB drip melting was carried out in 60 mm dia water cooled copper mould. The 60 mm dia and 300 mm long EB melted ingots were tested for internal soundness using X-ray radiography and characterized with respect to macrostructure. These ingots were also examined using optical & scanning electron microscopy, electron probe microanalysis and X-ray diffraction to understand the

microstructure of these alloys. Hardness and room temperature compression & tensile tests were performed on all the alloys in as cast condition. The EB melted ingots were found to be radiographically sound and exhibited columnar structure. A single phase microstructure has been observed in the alloys. Addition of W and Zr resulted into significant increase in hardness and strength values with a marginal reduction in room temperature ductility. No significant change in lattice parameters has been observed on addition of W and Zr.

Sorption behaviour of IDAA SDVB resin for uranium in sulphuric acid solutions

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Nuclear power industry relies heavily on uninterrupted supply of uranium. Therefore, there is constantly growing interest in the recovery of uranium from primary ores and other secondary resources. Among the various processing routes of uranium bearing ores, leaching by sulfuric acid produces uranyl sulfate ions, existing as various anionic species in the leach liquor. Uranium recovery from these aqueous streams can be best accomplished with anion-exchange processes either through liquid-liquid extraction or ion-exchange resins. Recently, we have reported a novel resin, namely, IDAA SDVB resin, synthesized by grafting of imino diacetamide moiety on to styrene divinyl benzene resin. This resin consists of tertiary amine and amide groups linked through methylene bridge, the nitrogen atom of the tertiary moiety gets protonated in acidic medium and it can therefore be used as anion exchanger. In the present work we have evaluated IDAA SDVB resin for recovery of uranium from sulphuric acid solutions, representing the leach liquor of low grade uranium ores. Uranium sorption on to the resin was fast, the kinetic data fitted well in pseudo second order kinetics model. Sorption of uranium was found to increase with the increase in initial concentration of uranium in aqueous feed solution, the sorption isotherm data fitted more closely in to Langmuir isotherm model. The sorption of uranium is observed to decrease with the increase in initial feed acidity, thereby, allowing sorption and desorption at lower (0.1 M H_2SO_4) and higher (1.0 M H_2SO_4) acidities respectively.

Leaching kinetics of UO_2 and FeS_2 in co-existing system under oxidative alkaline conditions

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Iron sulphide minerals, particularly pyrite, are commonly found gangue in uranium ores. Smaller content of pyrite plays beneficial role in oxidative alkaline leaching of uranium. However, when the same is present in excess, the leachability of uranium oxides is affected as both oxygen and carbonate ions which are essential for UO_2 solubilisation are consumed by sulphide gangue. Understanding the simultaneous dissolution of pyrite and uranium oxides in co-existing systems is complex. Thus, it is of importance to study the kinetics of dissolution of iron sulfides and uranium minerals in delineating the leaching characteristics of uranium ores in carbonate host rocks with substantial content of iron sulphides. The

study reported here is an attempt to develop rate equations for dissolution of iron sulfide vis-a-vis that of uranium oxide in carbonate solutions and elicit the effect of pyrite on leaching behaviour of UO_2. Leaching experiments were carried out in a batch autoclave reactor on a synthetic mixture of pure uranium dioxide (0.2%), pyrite (varied from 1% to 6%), calcite (varied from 89% to 84%) and silica (9.8%) in the size class of 74 to 100 µm. The reaction temperature and pressure were set at 125^0 C and 7.5 atm. respectively. The lixiviant concentrations were 0.5 M of Na_2CO_3 and 0.5 M of $NaHCO_3$. Oxygen was used as oxidant and total reaction time was 2h. The kinetic curves were analysed using shrinking core model. Chemical reaction controlled leaching mechanism fitted well for both UO_2 and pyrite, with coefficient of determination (R^2 value)>0.9. Scanning Electron Micrographs of the feed and leach residue too confirmed the predominance of chemical reaction control in the dissolution process. Conversion of pyrite varied from 9% to 72%, while the dissolution of uranium dioxide varied from 8 to 60%. Order of pyrite leaching reaction was found to be fractional which is in accordance with the chemical reaction controlled mechanism. The rate of leaching of uranium increased with initial increase in percent pyrite in the feed and decreased beyond certain concentration.

Development of soft magnetic steel for nuclear applications

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Low carbon soft magnetic steel is being largely used in nuclear and atomic research resulting in its increased demand. The challenge in manufacturing of such steels lies in attaining desired magnetic properties like high permeability/low coercivity as well as required mechanical properties with a stringent chemistry range - low Si, C, Al etc. To accomplish this, extensive laboratory heat treatment simulation studies and microstructural analysis have been done for freezing precise process parameters, alloy chemistry and successful in achieving these properties in As-Rolled condition itself, completely eliminating the annealing process which otherwise is an additional process in manufacturing of such steels. By proper design of alloy composition and processing parameters, the microstructure containing coarse ferrite and lesser % of pearlite is developed yielding the desired product.

PHYSICAL METALLURGY

Invited talks

Session 1: Nov 13, 2014: [09.00 - 11.00]

Overview of recent developments in study of phase stability in Ti alloys

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The first part of the paper would give a brief introduction to the role of electronic structure in stabilizing the crystal structure in titanium alloys. The development of Ti-Ta-Nb alloy for the reprocessing applications in nuclear industry will be presented briefly. The major part of the presentation would explain the relation between the crystal stability, electronic structure and bonding nature leading eventually to the electronic design criteria for the development of low modulus alloys for biological applications of titanium alloys. The need for an intermediate phase of orthorhombicα'', with solute addition in titanium lattice is explained in terms of relative differences between the lattices of hcp α' , orthorhombic α'' and bcc β phases in titanium alloys. The charge density distribution studies using Rietveld analysis of X-ray diffraction results yield additional insight into the bonding nature and anisotropy in titanium-molybdenum alloys, in terms of changes in atomic radius, bond length, and delocalization of electrons and mid-bond density, which will be discussed in detail.

Session 2: Nov 13, 2014: [13.30 - 15.30]

Developing ultra-fine and bimodal grain structures in low-carbon steel by inter-critical deformation and annealing

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Effect of deformation of metastable austenite (below A_{e3}) or both austenite and ferrite in the two-phase inter-critical region (between A_{r3} and A_{r1}) on the microstructure, crystallographic texture and mechanical properties of low-carbon ferritic steel has been investigated. Heavy single-pass deformation in the inter-critical region has been found to develop ultra-fine ferrite grain structure with average grain size less than 2 μm. Light deformation, on the other hand, produced bimodal grain structures. Depending on the deformation temperature the ferrite grain refinement is dictated by the following mechanisms: (i) Dynamic strain-induced austenite to ferrite transformation followed by conventional dynamic recrystallization of ferrite at high temperatures (≥ 1073 K), (ii) extended recovery and continuous dynamic recrystallization of ferrite at intermediate temperatures (~1023 K) and (iii) simple dynamic recovery of ferrite at lower temperatures (≤ 923 K). Rapid inter-critical annealing of warm-rolled steel can also develop 'bimodal' ferrite grain structures having fine- and coarse-grain regions. Microstructural changes have been analyzed using dilatometric studies, prediction of ferrite grain size, and micro-texture measurements. Fine austenite grains (< 5 μm) developed during rapid annealing, transformed into fine-ferrite grains (2 to 4 μm)

after cooling. Coarse-ferrite grains (28 to 42 µm) resulted from the recrystallization and growth of deformed ferrite grains. The effect of heating rate on the microstructure formation during inter-critical annealing has also been studied. A slow rate of heating (30 K/s) developed a uniform distribution of fine-ferrite grains and austenitic islands, while rapid heating (300 K/s) generated coarse blocks of austenite in the ferrite matrix. As expected, bimodal ferrite grain structures or fine-scale ferrite-martensite dual-phase structures showed superior combination of tensile strength and ductility, compared to the fine and ultrafine-grained ferritic steels. Besides tensile properties the effect of ferrite grain structures on the impact toughness properties have also been studied.

Session 2: Nov 15, 2014: [15.30 - 17.30]

Microstructural basis of 'Building Factors' in electrical equipment

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Manufacturing the laminations of electrical steels, both CRGO (cold rolled grain oriented) and CRNO (cold rolled non-oriented), involve elastic-plastic deformation. The semi-processed grades of CRNO are also subjected to post-fabrication annealing. Such manufacturing practices may alter the magnetic performance. This is generalized, by the designers of electrical equipment, as 'building factor'. The aim of this talk is to seek the microstructural basis for the 'building factor: relating microstructural evolution with magnetic performance in electrical steels.

Laves phase precipitation in the multi component in Nb-Si-Ti-Al-Cr-X alloys

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Advanced intermetallic materials, in particular the refractory Nb-based silicides, possess a good combination of properties like high strength at elevated temperatures, high stiffness, low density, etc., which make them potential candidate materials for high temperature applications. A multi element approach, which produces these silicides in equilibrium with the soft matrix, offer optimum combination of the high temperature properties. The microstructural investigations on the alloys which were aged between 600-1000°C has shown the presence of nano-phase precipitates. Morphologies, crystallographic analysis and chemical composition of these phases have revealed presence of at least two type phases. Detailed transmission electron microscopic investigations of these phases have established that the low temperature phase is the O-phase which upon aging at high temperature converts into the Laves phases. This observation raises certain questions. For example, is the O-phase a precursor for the Laves phase, do these phases have any structural relationships? Morphological distribution, structure and chemical composition of these phases have been investigated to address these issues. Strong solute partitioning tendency among the various elements has also been noticed. The phase is observed to display B2 ordering. Dissolution and re-precipitation of the Laves phase, which has been encountered during various heat treatments, has been studied in detail and characterized in terms of elemental distribution in the phase and mechanistic aspects.

Contributory papers

Session 1: Nov 13, 2014: [09.00 - 11.00]

Developing X-70 linepipe steel by simulated thin slab casting direct rolling route

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Two low carbon (Nb-V and Ti-Nb-V) non-peritectic microalloyed grade laboratory heats were made to develop X-70 linepipe grade steel through simulated thin slab casting direct rolling route. A part of the heat was forged and used to study the continuous cooling transformation and hot ductility behavior of the steels using a thermomechanical simulator Gleeble 1500 D. The other part of the heat was used to simulate the thin slab casting direct rolling processing using a Gleeble 3500 thermomechanical simulator. Two types of deformation patterns were used. After the deformation sequence the samples were cooled at certain cooling rates to the specified temperatures, 580°C and 610°C, followed by air cooling for coiling simulation. The simulated samples were wire EDM cut and precisely machined to obtain tensile samples that were subsequently tested using a mini-tensile tester. The microstructures of the samples with desired properties consisted of ferrite-bainite along with microalloyed precipitates. The tensile properties attained were: YS-500-532, MPa, UTS-659-739 MPa, YR-0.68-0.77 and Hardness-215-260 VHN. The marginal presence of fracture prone {100} planes with desirable tensile properties qualified the developed steels for satisfactory toughness.

Study of phase transformation temperature, ductility & mechanical properties for Cu-Al-Mn shape memory alloy synthesized by liquid metallurgy route

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Polycrystalline Cu-8Al-10Mn was prepared using the liquid metallurgy route; from metallic raw materials and was cast into fingers and plates using metallic moulds. In order to precipitate the martensite phase, samples were heated at 920ºC held for 2 hours and quenched in iced water; the samples were left in water overnight and removed only after they were completely cooled to room temperature. The microstructure observed in the cast and quenched conditions show a uniform grain structure with an average grain size of 150-200 µm, and with α+β phases in the cast alloys; which indicates the alloys have a potential for exhibiting the shape memory behaviour. The martensite phase in the quenched condition exhibits a needle shaped structure. The samples have been characterised by XRD, DSC and tensile tests and results establish its potential to form shape memory alloys with high transformation temperatures.

Optimising the cooling rate for maximum precipitation strengthening of naturally cooled V-microalloyed steels

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HSLA steels containing the microalloying elements such as Nb, Ti and V are widely used for construction, linepipe, automobile, pressure vessel naval and defense applications. V has long been used in the high-strength steel plate / strip primarily for the purpose of precipitation hardening. V-addition gives better hot ductility and may improve the earthquake resistance of the construction grades by combining with C and N. In spite of several studies on V containing steels, the beneficial effect of V addition has yet to be fully explored, especially in terms of the effect of section size on V(C, N) precipitation. The present study, therefore, aims to investigate the effect of different cooling rates on V precipitation (carbide, nitride or carbo-nitride) of naturally (air) cooled blocks after soaking at 1100°C for 20 minutes. Two different compositions with same vanadium (0.05 wt.%) and different carbon (0.055 and 0.22.wt %) and nitrogen (137 and 80 ppm) content have been chosen to investigate the effect of C and N on precipitation strengthening of these steels. Soaking temperature was selected on the basis of thermodynamic calculations and block sizes were varied from 30 mm to 5 mm in order to obtain different cooling rates by air cooling. One sample was furnace cooled to obtain very slow cooling. Involvement of second phases like prealite/bainite/acicular ferrite was more in high carbon compared to low carbon composition. Macrohardness of all the cooled samples, micro and nano indentations from the ferrite matrix have been taken to understand the optimum cooling rate for ferrite matrix strengthening. Micro and nanohardness values show increasing trend with increasing cooling rate. TEM studies have also been carried out to evaluate the precipitation strengthening from the hardness values after quantifying the precipitates in terms of number density and mean radius.

Aging response of two secondary hardening ultra high strength steels containing varying levels of chromium and molybdenum

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High Co-Ni containing secondary hardening ultra high strength (SHUHS) steels possess an excellent combination of strength, fracture toughness and stress corrosion cracking resistance which is realized by careful control of aging treatment during which a variety of carbides of type M_xC_y (M = Fe, Cr, Mo) form. Aging results in complex microstructural changes. At low temperature such as 400-425°C, formation of Fe_3C precipitates is observed. At higher aging temperatures such as 450°C, coarsening and ultimate dissolution of Fe_3C is observed. When the aging temperature is in the range of 450-600°C, formation of extremely fine M_2C carbides is observed. In addition to the M_2C carbides, recent 3D atom probe studies reveal the presence of sub-nanometric carbon-lean clusters containing Cr, Mo, Fe and C. In this study the effect of relative Cr and Mo contents on the isochronal aging response of two SHUHS steels: one containing 2wt.% Cr and 1wt.% Mo designated 2Cr-1Mo and the other containing 2wt.% Cr and 3wt.% Mo designated 2Cr-3Mo, with all other alloying elements such as Ni, Co and C maintained at the same level in the two alloys. The aging response was monitored by Vicker's hardness measurements and compression

testing. Both steels exhibit a similar isochronal aging response with a peak hardening at 450°C. However, the 2Cr-3Mo steel exhibits higher hardness and compressive strength compared to 2Cr-1Mo steel at all aging temperatures, with a maximum difference in hardness of 90 VHN at the peak aging temperature. Optical and electron microscopy studies reveal a similar tempered lath martensitic microstructure in both steels, while 3DAP studies revealed the presence of carbon-rich and carbon-deficient clusters in as-quenched as well as aged conditions of both steels. Further, it has been observed that 2Cr-3Mo steel contained higher fraction of carbon-deficient clusters with a concomitant decrease in carbon-rich clusters compared to 2Cr-1Mo steel. The observed microstructures are consistent with ThermoCalc predictions which suggest that the 2Cr-3Mo steel would contain a significantly higher fraction of M_2C carbides over the range of aging temperatures.

Microstructural evolution of the interaction zone between U–9 wt.% Mo fuel alloy and Zr–1 wt.% Nb cladding alloy

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A few very recent studies have identified the ability of Zr in acting as a diffusion barrier to reduce the deleterious fuel–clad chemical interaction (FCCI), which restricts nuclear fuel designers around the world to successfully utilize the potential of the γ-phase stabilized U–Mo alloys as reduced enrichment fuels in research and test reactor. Further investigations pertaining to metallurgical interaction between U-Mo and Zr are essential not only to establish Zr as a diffusion barrier in U-Mo fuel but also to envisage Zr-base alloys as cladding as against the currently used Al-alloys. In this work, metallurgical interaction between U–9 wt.% Mo metallic fuel alloy and Zr–1 wt.% Nb clad material has been assessed through scanning electron microscopy (SEM), electron probe microanalysis (EPMA) and transmission electron microscopy (TEM). Interdiffusion of constituent elements across the fuel–clad interface, together with the phase reactions occurring at high temperature and during subsequent cooling, resulted in development of a layered interaction zone where coexistence of a bcc solid solution phase with varying compositions, along with α-U, α-Zr and Mo_2Zr phases could be noticed. The instability in the γ-U(Mo,Zr) matrix leading to phase separation into α-U and α-Zr phases and the orientation relationships amongst them were established through microdiffraction and composite selected area electron diffraction (SAED) patterns, respectively. The present study is an endeavor to rationalize these observations, which remain unexplained in literature.

Effect of solutionizing temperature on age hardening behavior of cast Al-Si-Cu-Mg alloys

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A354 based cast Aluminium alloys with modified Cu-content were subjected to solution treatment at various temperatures between 490 to 520°C. To avoid the incipient melting during solution treatment, two stage heat treatments were employed for solution temperatures greater than 500°C. The solution treated alloys were then subjected to ageing at different temperatures (viz. 170 and 220°C) for various times. It is found that the peak hardness increases with increasing Cu-content. Although solution treatment at 500°C

was found to be giving good peak hardness, the solution temperatures of 510 and 520°C were found to be best in terms of slow coarsening behavior for ageing temperatures of 220 and 170°C respectively. The results will be discussed with the emphasis on its importance through the application point of view. Some phase equilibria simulations based on the thermodynamic program, Thermocalc, will also be presented in order to explain the observed age hardening behavior.

Aging behaviour of low carbon copper containing high strength low alloy (HSLA) steel

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This paper will present and discuss the results of our work on the investigation of the microstructure and mechanical properties of 0.08 wt% C HSLA steel, containing ~ 4.5 wt% Ni and 1.8 wt% Cu. The focus of the study was the effect of tempering/aging temperature on the structure as well as mechanical properties in the quenched and tempered/aged condition. The steel was melted in air induction furnace, homogenized, forged and hot rolled to 15 mm thick plate. The rolled plate was cut into pieces which were all subjected to austenitization at 900°C for 1 hour, followed by quenching in water and subsequently tempered/aged at different temperatures in the range of 500-700°C for 90 minutes, followed by water quenching. Microstructural observations, supported by ThermoCalc™ calculations, suggest that the upper end of the aging range is in the two phase (ferrite+austenite) region. The sample tempered at 500°C exhibited the highest hardness and yield strength. TEM of this sample showed profusion of fine (likely copper) precipitates. However, the same sample also had an impact toughness of only about 33 J at -30°C, whereas samples aged between 575°C and 650°C had impact toughness at -30°C in excess of 100J. While the impact toughness decreased at higher aging temperatures, average impact energy of 74J was still obtained upon aging at 700°C. Fractography of the different samples revealed the presence of mixed quasi-cleavage and ductile (void nucleation and growth, characterized by dimples) features in the sample aged at 500°C, while the other samples essentially showed the ductile features. For tempering/aging temperatures higher than 500°C, the hardness and strength values dropped continuously, reaching a minimum at 625°C (UTS) or 650°C (YS), due to the partial recovery of the matrix and coarsening of Cu precipitates. These changes are also believed to be responsible for the gradual improvement in the toughness until 650°C. The increase in strength and hardness at higher aging temperatures may be attributed to the formation of freshly formed martensite from reverted austenite.

Evolution of microstructure in Al-Si alloys cast through RSF process

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RSF is a process in which particles have partial solid and partial liquid behavior. This process is based on enthalpy exchange between atleast two alloy systems with different enthalpies and having same

composition. The solid metal which is known as Enthalpy Exchange Material (EEM) is used as stirrer and after few seconds it dissolve in the melt and drops the temperature of the melt to give pre-decided solid fraction. In RSF process small particles come together and form the large particles. So coarsening is a process or mechanism in which small particles grow by reducing solid/liquid interface free energy. The reduction in interfacial energy is considered the driving force of this mechanism and it is known as Ostwald ripening which is given by the Lifshitz, Sloyozov, and Wagner and known as LSW equation. According to this equation as time increases the grain size of particles also increases. In LSW equation the value of coarsening rate constant is calculated theoretically and experimentally. Many investigators have observed that the experimental value of coarsening rate constant is greater than the theoretical value, so from this they conclude that coarsening is not occurs only due to Ostwald ripening but it also occurs due to two other phenomena known as coalescence and convective mass flow.

Effect of grain refinement on hot tearing in aluminium alloys

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Hot tearing is a common and severe defect encountered in alloy castings. It is identified as cracks, either on the surface or inside the casting. It lies at the intersection of heat flow, fluid flow and mass flow. Different factors have influences on its formation. These factors include alloy composition, its solidification and thermo-mechanical characteristics, melt treatment, casting and mold design, mold material, and process parameters etc. A fine grain structure and a controlled casting process reduce and limit hot tearing. In this study, Al-5Ti-B grain refiner is used to investigate the effect of grain refinement on hot tearing in Al-7Si and Al-4.5Cu alloys during permanent mold casting. The results suggest that Al-5Ti-B additions significantly reduced hot tearing in both alloys. Grain sizes were also seen to reduce significantly in both alloys with addition of Al-5Ti-B grain refiner.

Metallurgical evolution to achieve DWTT properties in high strength line pipe steels

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Over the past decade, requirements for high strength line pipe steel have turned to be stringent with respect to the strength and toughness requirements especially for drop weight (DWTT) properties. Development of high strength plates for transporting oil and gas under high pressure had always lead to challenge for satisfying the DWTT at sub-zero operating temperatures. Since toughness tends to decrease as strengths increase, structural integrity at low temperatures should be seriously considered in order to develop new high-strength, high-toughness pipeline steels. In meeting these demands, remarkable progress has been made in steel making and plate rolling technology. The alloy design and the rolling philosophy for the desired micro-structure have made it possible to improve toughness properties. This paper describes the metallurgical design philosophy to achieve DWTT properties at low temperature. Important elements of the metallurgical approach are low carbon chemistry with well-defined usage of micro-alloying elements, a reproducible high level of steel cleanliness and micro-structure homogeneity

with a sophisticated combination of rolling and cooling. Special emphasis has been made on the austenitic conditioning during roughing and finishing stage of thermo-mechanical rolling process. The basic theme is refinement of final ferritic grain size by proper choice of reduction schedules for thermo-mechanical rolling process. Special process conditions of industrial rolling of a X-70 garden in 1 inch thick through state of the art wider width plate mill at ESSAR Steel along with steel making practices is presented. It is demonstrated that a fine and uniform homogenous grain size in the final product is the key requirement for improved DWTT.

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Grain size modification of Aluminium foam

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In a world were safety and damage mitigation are increasingly becoming key issues – aluminium foams have immense potential. The high specific strength and stiffness achievable in aluminum foam makes it a competitive material for structural application where energy absorbability is premium. The typical stress – strain curve of the foam beyond the elastic region exhibits a plateau stress over an extended strain which is useful for high energy absorption at force levels commensurate with crash and blast energy absorption. In the present work, small amount of Scandium (0 to 1 wt.%) was added to the alloy to achieve grain refinement. The addition of Sc was found to reduce the grain size from 103 µm (without Sc addition) to 30 µm (with 1 wt.% Sc addition). The perceptible grain size reduction showed variation in the microhardness values of the cell wall material. The stress versus strain plot of foam material shows a typical yield point (much lower compared to bulk material) followed by an extended plateau region where the cell walls collapse layer by layer until full densification. The significant observation is that the foam yield point increases with increase in scandium addition and can be correlated to the grain size decrease according to the Hall-Petch equation, which is, $\sigma_y = \sigma_0 + k/\sqrt{d}$. The Hall-Petch equation is well established for bulk metal samples where increase in grain boundaries provides hindrance for dislocation motion. The implication of this result for metal foam will be discussed in the talk.

Microstructure and magnetic properties of Cu-Co alloy with the addition of Mn, Cr and Si prepared by ball milling and annealing

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Ball milling has been successfully employed in synthesis of supersaturated solid solution of immiscible metals. Appropriate annealing of the metastable solid solutions form dispersion of nanocrystalline phases. Such microstructures have been found to offer promising mechanical and magnetic properties. Similarly, thermal treatment of the supersaturated solid solutions of Cu-Co and Cu-Fe alloys leads to precipitation of

single domain magnetic phases like Fe and Co in the Cu matrix. The diamagnetic and conducting Cu-matrix reduces the repulsion between the precipitated magnetic phases and gives rise to interesting magnetic properties like Giant magnetic resonance. Therefore, improvement in the driven solubility of the immiscible systems appears to be effective in enhancing their technological potential. Mn has an effective role in improving the solubility. This has been attributed to the comparable Goldsmith radius and electronegativity value of Mn with those of Cu and negative enthalpy of mixing for Mn with each of the three transition metals. Present study investigates the effect of ternary addition of Mn, Cr and Si (10 wt.%) on the mechanically driven non-equilibrium solubility. It is found that addition of Mn has been most effective in enhancing the solubility and formation of complete solid solution between Co and Cu in short duration (30h) of ball milling. The microstructure of the ball milled Cu-Co-Mn alloy was found to stable after the isothermal annealing upto a temperature of 450°C for 1h. The magnetic properties *viz.,* magnetic saturation, coercivity and remanence of ball milled Cu-Co alloy in the presence of Mn altered significantly after annealing at the temperature range of 350-650°C for 1h. It is found that the best combination of magnetic properties of Cu-Co-Mn alloy has been found after annealing at 550°C for 1h.

Precipitation of γ′in alloy 693 and its effect on mechanical properties

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Alloy 693 is a newly developed precipitation hardened Ni-based alloy. It is a modified version of Alloy 690 with Al and Ti additions to promote the formation of strengthening γ′ phase in the austenite matrix. The alloy exhibits excellent mechanical strength, rupture strength and creep resistance at elevated temperatures. It also exhibits good surface stability, corrosion, sulfidation and oxidation resistance. Aim of this work is to study theγ′ precipitation, dissolution and its effect on mechanical properties of the alloy. The γ′ precipitation and dissolution reactions have been studied employing dilatometry experiments. The mechanical properties have been studied by measuring hardness of the alloys aged at different temperatures (800°C- 950°C) for a series of times (0.5- 100 hrs).It has been found that ageing treatment upto 900°C resulted into monotonic increase in hardness with time while at 925°C and 950°C hardness decreased initially upto 5 hrs followed by a sharp increase during later stages ageing. Microscopic examination of aged samples revealed the presence of polygonal shaped matrix grains of 100 µm size order and also the primary carbides of about 1-10 um size distributed uniformly throughout the matrix, as well as on grain boundaries. In this paper, time-temperature transformation curve for γ′ precipitation as well a detailed examination of microstructure as studied by XRD and electron microscopy will be discussed.

Microstructure of Nb-Cr-Si ternary alloys

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Investigations on Nb-Si-X ternary alloys are commonly aimed to improve the mechanical properties, oxidation resistance, creep strength etc. of the Nb-Si binary alloys by suitably selecting X as Mo, Al, Hf, Zr, W, V, Ga, Mg, Ti, Cr etc. Chromium is known to improve oxidation resistance, but scattered and

contradictory information are available on the effect of Cr on the evolution of microstructural features in Nb–Si–Cr alloys. For example, one group of researchers has reported the presence of the ternary phase $Nb_9(Cr,Si)_5$, unlike the observation by another group of researchers in a similar alloy. Additionally, information related to the morphology of the eutectic constituents in Nb-Si-Cr systems has not been explored so far. The aim of this report is to achieve in-depth understanding of microstructural evolutions in five arc melted Nb-Si-Cr cast alloys – Nb-16Si-32Cr (CrA), Nb-13Si-22Cr (CrB), Nb-16Si-9Cr (CrC), Nb-18Si-23Cr (CrD), and Nb-9Si-7Cr (CrE) The microstructural examinations were carried out by optical, scanning and transmission electron microscopy, X-Ray diffraction, and microhardness tests. The obtained results infer that: (i) the microstructure of the as cast ternary alloys CrA, CrB and CrC, exhibits primarily niobium solid solution [$(Nb,Cr,Si)_{ss}$], niobium silicide [$Nb_5(Cr,Si)_3$], and laves phase [$Nb(Cr,Si)_2$] along with some eutectic in the interdendritic spacing. (ii) the ternary alloys CrA, CrB and CrC can be considered as hypereutectic whereas alloys CrD and CrE as hypoeutectic with reference to the Nb-Si binary equilibrium diagram; the former alloys exhibit the laves phase unlike the latter ones, (iii) none of the investigated Nb-Si-Cr alloys show the presence of any ternary phase like $Nb_9(Cr,Si)_5$, (iv) The observed eutectic domains in hypereutectic alloys are coarse, degenerated in nature and consist of $(Nb,Cr,Si)_{ss}$, $Nb_5(Cr,Si)_3$ and $Nb(Cr,Si)_2$; the eutectic domains in hypoeutectic side are fine and consists of $(Nb,Cr,Si)_{ss}$ and $Nb_5(Cr,Si)_3$.

A study of Mg solubility in Al-Mg systems by novel X-ray diffraction technique

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Mechanical alloying (MA) has been successfully used for the synthesis of supersaturated solid solutions in Al–Mg system with Mg content (10-40 at. %). The equilibrium solid solubility of Mg in Al at room temperature is only about 1 at. %. In the present investigation, the starting materials for high-energy ball milling were Al-Mg powders with proportion of 60:40 at. %. MA has been done up to 100 hours in a Fritsch Pulverisette P6 planetary mill at a speed of 300 rpm with a ball-to-powder ratio of 10:1. The detailed phases evaluation were characterized by XRD pattern of the different milling hours using Philips PANalytical X-Ray diffractometer equipped with the position-sensitive detector and Ni-filtered Cu-K_α (λ= 0.154 nm) radiation generated at 40kV/40mA with a slow scan speed of 0.5s for steps of 0.02 for the angular range (2θ) of 25° to 90°. The details study of linear fit of Nelson-Riley (N-R) parameter showed that as the milling time progress the precise lattice parameter was increased i.e. the solubility of Mg in Al increases. The variation of stacking fault probability and dislocation density signifies the above regulation. Crystallite size was measured by Williamson-Hall (W-H) methods which show decreases in crystallite size at higher milling time. The monotonic variation of crystallite size and lattice parameter signifies elemental powder transform to nano-crystalline and the continuous formation of FCC solid solutions. Transmission electron microscopy was carried out to investigate the sequential microstructural evolution by Philips CM-12 operating at 200 kV. The grain size obtained from TEM analysis is in close agreement with the results obtained from W-H method. The minimum crystallite size was found to be around ~ 33 nm after 100 hours of milling. The solubility of Mg in Al was measured and explained by thermodynamic modeling using the Miedema model.

Designing thermo-mechanical process for superior creep resistance through directional solidification

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This work deals with designing a thermo-mechanical process for sheet metals with the aim of imparting superior creep resistance without compromising on strength. Directionally solidified stocks are subjected to cold rolling along the solidification direction followed by recrystallization annealing to produce grains with a high aspect ratio. Microstructural details including grain aspect ratio, grain-size, texture, and precipitation in the recrystallized sheet metal are analyzed using light microscopy, SEM-EDS, XRD, and EBSD. Superior creep resistance of the processed sheet metals is demonstrated for Al and Al-alloys by mechanical, indentation creep, and creep-sag testing.

The study of creep behaviour in gamma titanium aluminides

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The creep behaviour of γ-TiAl alloys is complicated due to its complex structure. The rate controlling mechanisms are still not fully understood. One major disadvantages of alloy is brittleness which limits their industrial applications. Brittleness in γ-TiAl alloys stem from strong bonding between unlike atoms as well as long range order which give rise to high Peierls stresses. Other factor such as intrinsically weak grains boundaries, limited operative slip systems segregation of impurities at grain boundaries, high work hardening rate, planar slip and the presence of constitutional defects may contribute to the brittleness in γ-TiAl alloys. Many investigators have attempted to understand the creep mechanism in titanium aluminides over the last two decades. The creep behaviour of Gamma Titanium aluminides depend strongly on alloy composition and microstructure. It is now well established that the optimum microstructure for the highest creep resistance, the lowest minimum creep rate and the best primary creep behaviour stem from fully lamellar (FL) microstructure in two phase (γ-TiAl + α_2Ti_3Al) alloy. Many of fascinating views on creep mechanism of γ-TiAl based alloys stem from metallographic observations using advanced experimental techniques and analytical methods. However, it is a matter of debate and often controversy arises in establishing the rate controlling mechanisms over a range of temperatures and stresses in γ-TiAl alloys.

DWTT in thicker gauge hot rolled steels for line pipe application

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Ductile failure in DWTT (Drop weight Tear Test) at subzero temperatures is one of the key requirement for line pipe steel segment. As the temperature drops or thickness increases, the ductile failure mode is difficult to achieve in DWTT. JSW Steel has successfully developed the new generation Nb micro-alloyed line pipe steels up to API X-80 grade for the first time in India through hot strip mill. It has been

understood that, to achieve ductile DWTT failure, the crack propagation of brittle Griffith crack need to be arrested by high angle boundaries placed with the interspacing lesser then the critical Griffith crack length. It is further understood that prior austenite grain boundaries act as a major Griffith crack arrester. Hence refining the prior austenite grain size will lead to improved DWTT. Refining austenite grain size requires multiple dynamic and static recrystallisation coupled with reduced grain growth rate at rolling temperatures.There are multiple mill limitations to roll thicker gauge line pipe steel especially high strength (API X70/80) with austenite grain size refining. This paper describes the chemistry design aspects and mainly the process optimization to achieve subzero temperature DWTT in thicker gauge line pipe steels. Multiple online trials have been conducted at JSW steel limited for stabilization of DWTT characteristic. The effect of chemistry and process variables are well connected with end properties, end microstructure and transfer bar microstructure (taken from crop ends). The end passes of rough rolling and its temperature plays the vital role in defining the end DWTT performance. Further to it, the partial recrystallisation in finish rolling, need to be avoided. The optimized rolling windows are identified based on grade and thickness to achieve superior DWTT in line pipe steels.

Study on the low-carbon ultra-fine bainitic steels with improved strength and toughness produced through conventional hot-rolling and air-cooling

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Low-carbon bainitic steels have created enormous interests among the scientists across the world in past few decades, because their high strength, toughness and weldability replacing the conventional quenched and tempered medium carbon steels. These steels are usually micro-alloyed with niobium, vanadium, titanium and copper. To add further strength and toughness chromium, molybdenum, boron and nickel may also be added to it. Three experimental steels were made in 100 kg laboratory induction furnace with various combinations of the above alloying elements and cast into 100 mm diameter cylindrical ingots. Subsequently, these ingots were hot-rolled to 6 mm plates in an experimental rolling mill with optimum thermo-mechanical parameters through both air-cooling and water-quenching routes. Steels processed trough both these routes provided ultra-fine low-carbon bainitic microstructures, Maximum YS and UTS achieved in air-cooled steels were 575 and 705 MPa and those were 770 and 896 MPa in water-quenched steels. The elongation percent in those steels were 20.07 and 16.19 respectively. The Charpy impact toughness of both the air-cooled and water-quenched steels was excellent and at -20 °C, the CVN energies varied from 113 to 169 Joules and 72 to 142 Joules respectively in these steels. Cu-B added steel was found to give optimum combination of strength (YS-575 MPa) and toughness (113 Joules at -20 °C) in air-cooled steels, whereas, the best combinations of strength (770 MPa) and toughness (137 Joules at -20 °C) could be achieved in Cu added steel in water-quenched conditions. Thus, low-carbon steels could be developed with proper combination of alloying elements and thermo-mechanical parameters even in air-cooled condition. The present steel will have several industrial applications and can be produced by simple industrial processing of multi-pass rolling and air-cooling without the necessity of going into traditional and costly quenching and tempering.

Accelerated cooling of steel plates after hot rolling to improve mechanical properties

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Thermo-mechanical controlled rolling is a technique designed to improve the mechanical properties of steels by combining controlled hot deformation process and accelerated cooling. The project goal is to achieve the fine and uniform ferrite-pearlite structure of hot rolled products. Due to this structure steel shows good combination of strength and toughness with improved weldability and thus avoiding the need for heat treatment process like normalizing. A study was done with low-carbon steel of two different samples. Sample A was prepared with 0.13%C, 0.77%Mn, .02%P, .02%S, 0.22%Si, 0.21%Al and Sample B was prepared with 0.15%C, 0.81%Mn, 0.02%P, 0.02%S, 0.25%Si, 0.21%Al. The process was carried out with slabs of different thickness in a controlled schedule of heating, soaking, rolling and cooling. The slabs were heated in the walking beam furnace within the temperature range of 1050°C to 1200°C which result in austenitization, then soaked at 1 hour period and accelerated cooling of plates was applied between 800 to 450°C under rates of 1 to 20°C/s, then followed by air cooling.The result of the above treatment showed that, plates have fine grains of ferrite-pearlite structure, uniformly distributed along the entire cross section with minimum amount of transformation products of austenite in bainite-martensite zone. The microstructure of both samples studied under different finish rolling and cooling conditions, consistedmainly of ferrite and pearlite, as revealed by optical and SEM microscopy. Controlled cooled plates have resulted inconsiderable refinement of ferrite grain size and pearlite inter lamellar spacing, and increase in volume percent of pearlite compared to normalized plates. Accelerated cooling of hot rolled product resulted in a decrease in grain size from ASTM grain size no. 8-9 to 10-11.

Session 3: Nov 15, 2014: [15.30 - 17.30]

Synthesis and preliminary study on iron-based shape memory alloys

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An Fe-Mn-Si-Cr shape memory alloy was produced from elemental metal powders using mechanical alloying process followed by sintering.. Mechanical alloying involves solid state reactions among powders due to high energy collisions and offers several advantages like reduced grain size, formation of intermetallic compounds and capability of producing nano-sized structures. The α phase transformed into the γ′ phase during MA. MA plays an essential role in stabilizing the γ′ phase, which is associated with the shape memory effect in this alloy system. XRD peaks shows the presence of ε phase after 30h of milling The alloyed powder was compacted and sintered at 850-900°C. The samples were then annealed under vacuum condition to relieve the resulting internal stresses. They are then subjected to hot rolling to thick strips. After deformation, γ′ → ε stress induced martensitic phase transformation occurred. Shape recovery was observed after subsequent heating with a ε→ γ′ reverse martensitic transformation. The shape memory effect was investigated after six training cycles.

Effect of differential warming on microstructure and wear behaviour of cryotreated HSS tool steels

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Cryogenic treatment enhances tribological properties of material to a large extent. For determining the effect of differential warming up on cryotreated M2 and M35 tool steel, the specimens were hardened (1200^0C) followed by cryogenic treatment (-185^0C,16 h) and then immediately the specimens were taken up to room temperature at different warming up rates. In this, one set of specimens was warmed up by directly dipping into water (Water warmed) which was maintained at room temperature and another set of specimens was warmed up by holding into an insulated box (Naturally warmed) then both these differentially warmed up tool steels were followed by multiple tempering (400^0C, 2 h each per cycle). These treated tool steels were examined for wear rate, hardness and carbide counts. A trend was established for wear rate and hardness as a function of differential warming up. Cobalt bearing M35 tool steel shows improved wear resistance than that of M2 tool steel in single tempered condition (HCT) especially in naturally warmed condition whereas reverse trend is seen in HCTTT as compared to water warmed condition.

Development of new bainitic steels for rail applications

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The major objective of development of new Bainitic rail steel is improved wear resistance, toughness, corrosion resistance and tensile strength. Apart from above properties castability and machinability are other important properties to be considered. The pearlitic rail steels used have good ductility and wear resistance but tensile strength and hardenss was poor. Bainitic structure has generally higher wear than pearlitic steels because Bainitic structure consists of carbide particles finely spread over the matrix of fine ferritic structure. Carbide causes particle shelling away from ferritic matrix during run over Bainitic rails. So, new Bainitic rail steel having distribution of fine pearlite and upper bainite in ferritic matrix was developedby the modified austempering technique which includes austenitising followed by controlled air cooling and quenching. Wear, tensile and hardness test were performed which showed improved wear resistance and good combination of tensile strength and elongation than previous pearlitic or Bainitic rail steel.

Development of medium carbon niobium-microalloyed steels withchromium and nickel addition

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High strength along with good ductility is required for structural parts, automobile components, military applications and underground pipes for petroleum products. Hence many research works are going on in

microalloyed steels all over the world. Medium carbon microalloyed forged steels exhibit enhanced mechanical properties as compared to the quenched and tempered grades. Two medium carbon niobium-microalloyed steels with slightly higher nickel and chromium contents were developed. These medium carbon steels contained 0.05-0.06% Nb, 1.8-2% Ni and 1.2% Cr. The steels were produced in a laboratory scale induction furnace by adding required alloying elements in low carbon steel scrap. After superheating, the molten metal was poured in a preheated metallic mould and produced rod shaped castings. These castings were hot forged as per the specially designed cycle and given 25% reduction in the cross section. These samples were subjected to various tests such as tensile test, hardness test microstructure observation, grain size determination etc. High tensile strength along with good ductility was obtained in the microalloyed steels. The high strength obtained was due to the combined effect of 1) precipitation strengthening by Nb carbide and/or carbonitrides, 2) grain refinement of ferrite (during hot forging) by pinning and/or recrystallization retardation effect of Nb and 3) solid solution strengthening due to Ni and Cr addition.

Development of multiphase high strength and ductile construction steel

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High strength and ductile steels are needed in construction and automotive industry. The possible upper limits of strength and ductility of pearlitic and martensitic steels have already reached. This prompts the scientific community to go for multiphase steels consisting of optimum combination of bainite, ferrite, pearlite and tempered martensite which can give very high strength along with good ductility. This research aims to develop a multiphase steel combining nano-spheroidal cementite and bainite in ductile ferrite matrix, which is expected to possess an optimum balance of strength and ductility. A novel heat treatment cycle has been designed to obtain this multiphase structure consisting of bainite, spheroidised cementite, ferrite and retained austenite. For this purpose, typical medium carbon high silicon EN45 steel was austenitized and isothermally annealed in the intercritical temperature zone followed by quenching to room temperature. The samples were then strained to 10% to 30% by cold rolling. To obtain a spheroidized microstructure after cold working, the steel samples were held at temperature below Ac1 for around 3 hrs. Then the samples were further held in intercritical range at 800°C temperature for different durations ranging from 10 to 30 minutes. After that to attain a bainitic microstructure, the samples were quenched in a salt bath kept at an isothermal transformation temperature of 350°C for various time intervals varying from 10 to 120 minutes followed by water quenching. Samples were then characterized with the help of optical microscopy, X-Ray diffraction and scanning electron microscopy. Standard tensile specimens were made to study variation of mechanical properties with the microstructure.

Development of ultrafine- grained ferritic stainless steel

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Ferritic stainless steel of grade (430) was deformed upto equivalent strain of 1.2 by Equal Channel Angular Pressing (ECAP) adopting route B_C.Material was also cold rolled for 87% reduction in area. ECAPed and cold rolled samples were flash annealed at temperatures from 650 to 750^{0}C.Microstructure was characterized by optical microscopy and transmission electron microscopy. Mechanical property was determined by hardness measurement.Grains of ~28µm in undeformed material got refined to ~18µm (atequivalent strain of 1.2). Elongated grains consist of width size 0.243µmafter ECAP. Flash annealing of ECAPed sample produced recrystallize grain structure of size 0.444µm. However flash annealing convert sub-grains into grains without significant grain growth. Cold rolling produced elongated grains with average width of 0.375µm. Flash annealing of cold rolled sample at 750^{0}C produced grain size of 0.667µm. Hardness value increases in both ECAPed and cold rolled sample due to deformation whereas decreases slightly when flash annealed at different temperatures due to recrystallization. Now ECAP and cold rolling followed by flash annealing could be utilized to produce bulk ultrafine grains.

Synthesis and optical properties of undoped zinc sulfide nanocrystal embeded in aerogel matrix

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Combining sol-gel chemistry, which is a widely used technique for creation of glasses and ceramic materials, and semiconducting nanocrystals (NCs) could lead to the development of new materials to replace environmentally unsafe solid-state lighting components and also various applications of optical materials. ZnS semiconductor nanocrystals were synthesized by sol-gel technique in room temperature and embedded in aerogel. Silica aerogel was used because it possesses pores of all sizes and have maximum possible amount of surface area enabling embedded nanocrystals to sense the environment independently. Synthesized sample are characterized by UV-Visible spectrophotometer, Scanning Electron Microscope, Transmission Electron Microscope and Photoluminescence spectroflourimeter. The UV-Vis absorption spectra revealed the presences of absorption peak in ZnS at 332 nm which is red shifted to 389 nm for ZnS with aerogel samples. From the morphology of the sample, measured by TEM and SEM, it is found that ~ 5nm.The samplesare annealed at 100^{0}C, 200^{0}C and 300^{0}C temperatures for 30 min., to study the morphology and size variation of the prepared samples. PL spectra also showed a red shift for ZnS and ZnS with aerogel sample for increasing particle size. But after annealing, PL spectra showed red shift for ZnS and blue shift for the ZnS with aerogel sample.

Grain refinement of magnesium alloys through vibrations

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AZ91 Mg alloys are extensively being used in automotive, aerospace, electric and communication industries due to their light weight, improved damping property, electromagnetic shielding capacity, excellent machinability and good castability. Mg- Al Alloys have large freezing range, hence are susceptible to the formation of coarse grained structure. Refinement of microstructure can be done either by chemical method in which grain refiners are added to refine the microstructure or by vibrations, where vibrations are imposed during solidification. The various techniques to impose vibrations are; mechanical vibrations, electromagnetic vibrations and ultrasonic vibrations. Vibrations have strong effect on microstructure and mechanical properties. The changes in properties remain consistent with fine uniform non- dendritic grains. In the present paper mechanical vibrations at constant amplitude of 1mm and different frequencies of 25, 50 and 75 Hz have been imposed during the solidification of alloy to observe the grain refinement in the alloy and variations in the mechanical properties.

Phase transformations in chromite ore during thermal treatment

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Chromite ores used in the production of ferro-chrome contain the chromite spinel where the Cr^{3+} ions are present in the trivalent sub-lattice of the structure. In the simple iron chromite mineral, the divalent sub-lattice is occupied by the Fe^{2+} ions. However, the spinels are usually complex. Mg^{2+} ions are present along with Fe^{2+} ions in the divalent sub-lattice. Similarly, Fe^{3+} and Al^{3+} ions are present in the trivalent sub-lattice in many cases. It has become a common practice among Indian ferro alloy manufacturers to source chromite ores from different parts of the world. Hence, the ores used in the process show a large variation in chemical properties. The reducibility of the ores is affected by the thermal regimes encountered by the ore during the passage through submerged-arc furnace because of various phase transformations occurring during the descent through different regimes. The phase transformations occurring in different chromite ores used in the manufacture of ferro-chrome were studied. The results are discussed in this communication.

Characteristic study of graphite nanoparticle diffusion in bcc iron

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The diffusion phenomenon is greatly dependant on the size of initial diffusing particle. As per our study it is proved that, if the initial size of the diffusive particle is at nanoscale, diffusion occurs at faster rate

compared to initial bulk diffusive particle. As the size of the nanoparticle decreases, the diffusion activation energy of atoms decreases and the corresponding diffusion coefficient strongly increases due to the Arrhenius relationship between them, which leads to evident diffusion at lower temperature. The model prediction is in agreement with the experimental diffusion results of graphite (C) into bcc Fe substrate.

Thermal analysis and subsequent phase analysis of quaternary and ternary high entropy alloys synthesized by mechanical alloying

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High entropy alloys are a new class of alloys that are distinctly different from other conventional alloys, consist of more number of elements in equi-atomic ratio. These alloys exhibit outstanding properties such as strength retention at higher temperatures, good corrosion and oxidation resistance. Since the property of any materials depends on the structure of the material, it's important to understand the influence of elements, processing route on the phase evolutions. By having a proper knowledge on the factors influencing the phase evolution, it will be easier to synthesize a material for a desired application. Thus, in the present study, to choose an alloy for high temperature applications, various quaternary and ternary high entropy alloys weresynthesized by mechanical alloying, and their phase evolution and thermal analysis were investigated. From the phase evolution studies after mechanical alloying, it was observed that alloy that contains Ni tends to form major FCC phase whereas alloy without Ni tends to form major BCC phase. The BCC phase which forms after mechanical alloying was a metastable phase, and when annealed at higher temperature:(a) either dissolves into FCC phase, or forms chromium carbide if FCC is the major phase, and(b)it transforms to FCC phase if BCC is the major phase. During annealing, carbide phases were observed to form only in the Cr containing alloys. In alloys that contain both Ni and Co, Cr_7C_3 carbide was observed to be stable till 1000°C, whereas in other alloys, Cr_7C_3 transforms to $Cr_{23}C_6$ carbide which observed to be stable till 1000°C. These phase changes after annealing were correlated with the transition peaks observed in the DSC studies of the mechanically alloyed powder.

POWDER METALLURGY

Invited talks

Session 1: Nov 15, 2014: [11.00 - 13.00]

Power of powder –Inspire industry to make more with less

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Among metallurgical technologies, powder metallurgy (PM) alone can claim that it is a technology where an engineer can design the process to minimise the use of materials and not produce the part and machine (and thus produce scrap) to final dimensional requirements. In Ferrous PM, from the time Höganäs invented (100 years ago) the Höganäs Process and developed the first application for welding electrodes and later, self-lubricating bearings along with GM, Höganäs has been in the forefront of developments for Ferrous PM applications. These developments along with the new exciting research to develop PM transmission gears for automotive applications will be presented. MIM is an area of PM which is growing rapidly but from a small base as it is recent PM technology. The growth of this powder is hampered by high cost of fine powders required to produce parts using this technology. The MIM parts are also limited in size because the process limitations of the debinding process limitations. Höganäs is working on solving both these problems so that MIM use will increase rapidly. Höganäs through its own Digital Metals is taking the technology to the next level where additive technologies are being developed to produce 3-D parts which are mind boggling in complexity and shape. Some research which Höganäs is conducting in this area will also be described.

Session 2: Nov 15, 2014: [14.00 - 16.00]

Sintering time compression strategies for high performance P/M products

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Among the various material processing technologies, powder metallurgy (P/M) is the most diverse manufacturing approach. One attraction of powder metallurgy (P/M) is the ability to fabricate high quality, complex parts to close tolerances in an economical manner. The process effectively uses automated operations with low relative energy consumption and low capital cost. Furthermore, it is a flexible manufacturing process capable of delivering a wide range of new materials, microstructure and properties. This creates several unique niche applications for P/M. This talk describes the recent advances in consolidation of high performance P/M alloys and composites through sintering time compression. This talk will also focus on the processing a range of metallic systems and metal-ceramic composites through microwave sintering. One of the key characteristics involving microwave sintering is the inverse thermal gradient being observed as compared to conventional heating. While microwave heating of ceramics is well- understood and modeled, there is still an uncertainty about the exact mechanism and mode of

microwave heating in particulate metals. This talk will attempt to elucidate the dynamics of microwave-metal interaction during the sintering operation and its application to a range of ferrous and non-ferrous metallic systems and their composites.

Contributory papers

Session 1: Nov 15, 2014: [11.00 - 13.00]

Development of nano-oxide dispersed austenitic stainless steels by mechanical alloying

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The present study aims at synthesis of nanostructured alloys with nominal compositions of Fe-19.00Cr-11.00Ni (alloy M), Fe-19.00Cr-11.00Ni -1.0 Y_2O_3 (alloy N) and Fe-19.00Cr-11.00Ni-1.0 TiO_2 (alloy P) all in wt. %, through mechanical alloying and spark plasma sintering (SPS). The phase analysis and microstructural characterization of milled powders and sintered products were examined by X-ray diffraction study (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and energy dispersive spectroscopy (EDS).A gradual refinement of crystallite size with increasing the milling time is revealed by XRD analysis. Physical (density and porosity) and mechanical (hardness and wear resistance) properties of the sintered pellets were evaluated. Vickers microhardness value is highest for alloy P (1.0 wt. % TiO_2) (4.2 GPa) as compared to that of alloy M (2.6 GPa) and alloy N (1.0 % Y_2O_3) (4.0 GPa). Similar kind of trend is found in wear resistance property of the sintered pellets. The present oxide dispersed austenitic alloys record extraordinary levels of hardness and measures 1.5-2.0 times higher than of other austenitic steels. The novelty of the present consolidation route lies in the unique microstructure comprising uniform distribution of 30-50 nm Y_2O_3 or TiO_2 particles in the austenitic matrix recommended for grain boundary pinning and creep resistance.

Grain growth and densification during SPS of nano NiO: Coalescence of nano crystals

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In the current work densification mechanisms and microstructural evolution of nano Nickel Oxide (NiO) during Spark Plasma Sintering (SPS) was investigated with emphasis on the role of pressure and electric field. For this, consolidation was carried out in three different modes: pressure-less SPS, pressure-less SPS with NiO sandwiched in-between BN layers at the top and bottom, and conventional SPS with 50MPa sintering pressure in the temperature range of 900-1300 °C.Novel grain growth and densification behavior was observed for the pressure less SPS NiO in the temperature range of 1000-1200 °C, with ordered

coalescence of faceted nanocrystals being the primary mechanism. Enormous grain growth occurred in a narrow temperature window and at higher temperatures porosity was observed as perfectly formed cuboidal structures, hinting about the rapid diffusional pathways active during SPS. Further, annealing of the raw powders also revealed similar faceting of the irregular shaped starting powders. Metallic nickel was observed to have formed on the exposed sintered surface due to in-situ reduction during annealing. The grain size, hardness and relative density were evaluated and compared for all the three modes and the microstructure was characterized using FEG-SEM equipped with EDS along with XRD and TEM.

Spark plasma sintering of titania - structural characterization and properties

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Titania (TiO_2) has a wide range of applications. It is valued for its high thermal and chemical stability. Due to its brightness and high refractive index it is used as a pigment and also for optical coatings. It has also a preferred photocatalyst due to which it offers great potential in the detoxification of waste water. Recently Titania crystals were found to exhibit switching between two states of light which may lead to using it in electronic storage media. Researchers till date have focused on the various parameters of Titania doped materials. However the effect of sintering parameters on the structural and functional properties of pure Titania has never been explored.In this work Titania (TiO_2) powders were sintered at 900^0C, 1000^0C and 1100^0C using spark plasma sintering at with a holding time of 5 minutes and at a pressure of 30 MPa in vacuum. On sintering the samples were characterized by X-ray diffraction (XRD), their densities were measured and their micrographs were taken through a scanning electron microscopy (SEM).The sintered samples were mirror polished and thermally etched in order to reveal the microstructure. The samples were also subjected to micro- and nanoindentation to determine the mechanical properties of the sintered samples. This microstructure was fed into OOF2 a finite elemental microstructure modelling software and the structural and thermal properties of the material was estimated through finite elemental analysis considering practical boundary conditions. The results obtained were compared to the experimental results.

Effect of milling time and amount of Y_2O_3 on the properties of ODS ferritic steels for fast breeder reactors

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Ferritic steel powder having a nominal chemical composition Fe-9Cr-2.24W-0.2Ti-0.027Mn (wt. %) was introduced in the dual drive ball mill and milled with varying nano Y2O3 composition (0, 0.35 and 0.5 % (wt.%) to produce oxide dispersion strengthened (ODS) ferritic steel. Reduction in particle size and crystallite size were studied with the effect of milling time and varying Y2O3 percentage. The resulted milled powder was characterized by using particle size analyzer (PSA), X-ray diffraction (XRD) and field-emission scanning electron microscope (FE-SEM) to approximate the particle size, crystallite size and morphology of the particle respectively. X-ray peak broadening of ferritic steel was investigated by

Williamson-Hall (W-H) analysis. Crystallite size, lattice strain and deformation stress was estimated by using W-H analysis. Better Pearson's coefficient was observed for uniform stress deformation model (USDM) (0.988) in comparison to uniform deformation model (UDM) (0.64) which shows better estimation of lattice parameter. With milling, dislocation character was observed to be changing, from near edge to mixed type. Starting ferritic steel powder was having a mean diameter of 52 µm and d_{50} as 46 µm and after the milling it was observed as low as 13 µm and 12 µm respectively. 0.35%Y2O3 shows higher densification (99%) with respect to 0.50% Y2O3 (97.5%). At the optimized condition, ferritic steel powder together with yttria was milled in the ball mill to produce ODS ferritic steel powder suitable to be used as structural material in fast breeder reactors at 1.0 kg batch scale. Lattice parameters of the milled powders were also estimated. Uniform milling with convoluted particle shape and homogeneous distribution of Y_2O_3 throughout the matrix was observed by using electron microscope.

Synthesis of p and n-type β-$FeSi_2$ thermoelectric material by mechanical alloying

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Development of high temperature thermoelectric materials like β-$FeSi_2$ is the need of 21st century to harvest waste heat energy and to convert it into a more user friendly form like the electrical energy. Temperature difference is the only driving mechanism required for this thermoelectric material. β-$FeSi_2$ has been found to exhibit advantages like high temperature (≈900°C) thermoelectric conversion properties, large Seebeck coefficient, chemical stability, non-toxicity and relatively low cost of production. It is an intermetallic phase in the iron-silicon system with 53.4 – 58.2 wt% Si. In the present context, the elemental powders of iron (99.9% purity, 400 mesh), silicon (98.5% purity, 200 mesh) and manganese (99.0% purity, for *p*– type semiconductor) or cobalt (99.5% purity, for *n*– type semiconductor) have been mechanically alloyed for 6h using high-energy Attritor ball-mill (400 rpm, BPR= 10:1, Ar atmosphere). These mechanically alloyed powders were then compacted into pellets (16 mm diameter, 3mm thickness) at compaction pressure of 700 MPa. The pellets were subsequently sintered according to an innovative sintering cycle. The sintered pellets were then characterized using XRD and SEM. Thus the resultant matrix substantially consists of β-$FeSi_2$ with traces of Si and no distinct peak of Fe depicting complete dissolution of Fe in compound formation.

Age hardening behaviour of Al 2014-TiB_2 composite prepared by powder metallurgy route

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This work deals with a comparative study based on the age hardening behaviour of Al 2014 metal matrix composite reinforced with TiB_2 particulates in different volume percentage. Al 2014–*x*TiB_2 (*x* = 5, 10 and 15 vol.%) ex situ composites have been successfully prepared by P/M route. All the samples were solutionised; quenched in air and aged in a Tubular furnace. Field emission scanning electron micrographs at low magnification show uniformly distributed Tib2 particles throughout the matrix at 5% volume. Field emission scanning electron micrographs at higher magnification shows the presence of uniformly distributed secondary phase in the alloy matrix indicating a strong continuous bond between TiB_2 particle

and the alloy matrix. TiB_2 particles enhance the aging kinetics and the time to peak aging at the aging temperature of 160 ◦C comes down from 6 to 5 h when the TiB_2 amount is increased from 0 to 5%volume.

Synthesis of Ni-Ti-Zr shape memory alloys through powder metallurgy using spark plasma sintering process

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Shape Memory Alloys (SMAs) are a unique class of alloys, which exhibit shape change when subjected to an appropriate thermal, mechanical or thermo-mechanical procedure. While equiatomic binary NiTi has been the most popular among SMAs, over the years a lot of research has gone into the development of Ni-Ti SMAs with ternary additions to meet the need for high temperature shape memory alloys (HTSMAs) with high transformation temperatures. Amongst the potential alloy systems based on Pd, Pt, Au, Hf, Zr, it is alloys based on the last two which are economically viable. NiTiZr alloys produced through the conventional casting technique tend to be brittle and are difficult to process.Powder metallurgy has, therefore, been used to produce fine-grained alloys so as to avoid secondary processing. Conventionally compacted and sintered specimens have problems, such as porosity, large volume fraction of secondary phases and oxide formation owing to prolonged holding times at high temperatures.Mechanical alloying offers the advantage of producing a homogenous microstructure, while spark plasma sintering leads to extremely dense specimens at shorter sintering times.In the present work, nickel and titanium powders were milled in a high energy ball mill at 300 rpm using tungsten carbide balls and vials with toluene as the medium for a period of 10 h. A milling time of 5 hours was selected as the optimum duration. The same parameters were adapted to mill powders of nickel, titanium and zirconium. The as-milled powders were then sintered in a spark plasma sintering setup at a temperature of 800°C and 900°C, respectively. The as-sintered samples were homogenized at temperatures between 900°C and 1200°C. The relative density of the as-sintered samples was determined using the Archimedes principle. XRD, Optical Microscopy, SEM and EDX were used to confirm mechanical alloying in the as-milled powders as function of the milling time. These techniques were also used to trace the phase evolution in the sintered alloys as a function of the sintering parameters.

Synthesis, characterization and mechanical properties study of spark plasma sintered FeCoMnW and FeCoMnCuW high entropy alloys

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Multi component alloys with equi atomic compositions called as high entropy alloys have attracted researchers in recent years. These alloys possess high configurational entropy which aides in the formation of simple solid solution ssphases and excellent mechanical properties even at high temperatures. Fe-Co-W alloys have been studied for tool steel applications. In this study, FeCoMnW and FeCoMnCuW high entropy alloys were synthesized by high energy ball milling for 20 h at 300 RPM with Toluene as medium. X-Ray diffraction (XRD) patterns were taken at regular intervals during the milling and peaks corresponding

to a single phase BCC solid solution were observed in FeCoMnW alloy. XRD Peaks corresponding to BCC phase and Cu were identified in FeCoMnCuW milled powders. Scanning electron microscopy (SEM) and EDS analysis of the particle cross section showed uniform distribution of the elements in the milled partciles. DSC analysis was used to fix the sintering temperature at 1000 ^{o}C. Spark Plasma Sintering (SPS) was done at a temperature of 1000^{0}C and a pressure of 50 MPa for 10 minutes. Density measurements were done using Archimedes' principle and obtained good densification of the alloys, which is confirmed by the SEM images. Sintered samples were characterized using XRD and SEM. Microhardness and compression results of compacted FeCoMnW and FeCoMnCuW alloys have been studied.

Powder metallurgy technology - "A cost effective solution"

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Powder Metallurgy is Net Shape Technology. Main Characteristics is Good Dimensional Control & Stability. This makes PM Cost Effective in comparison with traditional manufacturing methods like Forging/Broaching/Machining/Casting. PM Components are created by mixing, bonded mixing, diffusion bonding or alloy powders and compacting the mixture in a die/tool set, further these parts heated or "sintered" in a controlled atmosphere of Hydrogen & nitrogen furnace to bond the particles metallurgical. The high precision forming capability of PM generates components with near net shape, with features of good dimensional control. Parts get finished without the need of secondary machining. With controlling porosity parts gets capability of self-lubrication. As Theoretical Density is not possible to achieve through PM, parts are in less weight as compare with Forged/Cast or machined parts. This Light Weight is advantageous in automotive sector, as it reduces engine weight & better performance is achieved.

Microwave properties of MgO coated iron soft magnetic composites (M-SMC)

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Soft magnetic materials are extensively used for power electronics applications, electric motors because of their lower losses. Soft magnetic composites (SMC) are the second generation soft magnetic materials. In the present work, MgO coating was done on electrolytic iron powder and was compacted in the form of toroid and pellets. Pellets of varying percentage of MgO coated Fe were also compacted. The scattering parameters (S11, S12, S21, S22), complex permeability ($\mu = \mu' - j\mu''$) and complex permittivity ($\varepsilon = \varepsilon' - j\varepsilon''$) were measured using a flanged coaxial sample holder with two port vector network analyzer up to 8 GHz. The properties were measured using transmission line method. The microwave properties were studied as the function of frequency. The effect of thickness of the sample on the microwave properties was also studied.

Effects of the temperature of hot isostatic pressing (HIP) treatment on Cr–Si targets

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Commercial as-hp (hot pressing) treated Cr–Si targets are used throughout this study, with three different compositions: Cr20–Si80, Cr35–Si65 and Cr50–Si50. To evaluate the effects of microstructure and properties of as-hp treated Cr–Si targets by hot isostatic pressing (HIP) SEM, XRD and porosity inspections were performed. The experimental results showed that the 1373 K, 1750 MPa, 4 h HIP treated with three different Cr–Si targets had suppressed porosities successfully. The most efficient was Cr50–Si50 target subjected to HIP treatment. Porosity decreased about 60% after HIP treatment, and both the nitrogen and oxygen concentrations of the targets were slightly increased after HIP treatment. This was especially true for the single silicon in Cr–Si targets such as Cr20–Si80 and Cr35–Si65. The aim of this paper is to discuss these methods and finding suitable temperatures for the HIP for Cr–Si targets.

Microstructural examination and mechanical properties of Cu-Al-Ni shape memory alloys developed through an optimized mechanical alloying and P/M route

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Shape Memory Alloys (SMAs) are the class of generic alloys which can retain their predetermined shape upon heating to a particular temperature. The present work is devoted towards the development of Cu-Al-Ni SMA with Ti as grain refiner through an optimized P/M process involving mechanical alloying, homogenization sintering, secondary densification processing and quenching. For optimization of the mechanical alloying, several parameters such as milling duration, speed and ball-to-powder ratio have been varied in order to obtain fine grain size and homogeneous solid solution of the alloy. The SEM, XRD and EDX analyses indicate that a fine homogeneous pre-alloy have been obtained after milling for 40 hrs using 40:1 BPR at 200 RPM. The proper alloying did not occur when milling was done at lower BPR and RPM, This has been confirmed through the observation of undiffused Al rich islands in the SEM images. On the other hand, milling at higher BPR and RPM led to oxidization of Al and reduce the refinement rate of the crystallite size. In order to evaluate the mechanical properties of the sintered and quenched samples, Vickers Hardness and three point flexural tests have also been performed. The Vickers hardness measurements show a significant reduction in the hardness of the sample milled at 200 RPM and 40:1 BPR for 40 hrs. This could be due to the formation of martensite phases in the sample. In all other samples, the hardness values remained the same as that of the sintered samples before quenching. The flexural strength of the hot isostatically pressed sample prepared using the optimized milling condition has also been very high as compared to that of other samples. The XRD and SEM analyses of the quenched samples confirms the formation of γ' martensitic phase in the samples milled using the optimized conditions.

Session 2: Nov 15, 2014: [14.00 - 14.30]

Microstructure and properties of spark plasma sintered $[(Fe_{0.5}Co_{0.5})_{0.75}B_{0.2}Si_{0.05}]_{96}Nb_4$ bulk metallic glass

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Bulk metallic glasses (BMGs) are promising materials for structural application as they exhibit high mechanical strength, high hardness, good fracture toughness, superior corrosion resistance. Recently, BMG rods of $[(Fe_{0.5}Co_{0.5})_{0.75}B_{0.2}Si_{0.05}]_{96}Nb_4$ alloy with diameter up to 5 mm have been produced using Cu mold casting technique.The actual utilization of these amorphous materials finds limited practical applications due to the difficulties in fabricating large sized components. The present investigation deals with the sintering of $(Fe_{0.5}Co_{0.5})_{0.75}B_{0.2}Si_{0.05}]_{96}Nb_4$ alloy in the form amorphous compact discs of 10-20 mm diameter using spark plasma sintering (SPS) equipment. In this case, the dimensions are not restricted by the glass forming ability of the alloy. The process involved the preparation of amorphous ribbons using a vacuum melt spinner followed by high energy ball milling to crush the ribbons into very fine particles. The powder was then compacted in the form of pellets using SPS process within the supercooled liquid regime of the amorphous phase. The structure and thermal stability of the sintered specimens as well as the interface characteristics between powder particles were investigated.The relative density of the sintered compacts, processed at a sintering temperature of 530°C with a uniaxial loading pressure of 450MPa was found to be 98%. Microstructures of sintered compacts indicated that the density and degree of crystallinity can be effectively controlled by optimizing SPS parameters. A detailed analysis was carried out to understand the densification behaviour of the amorphous powder during SPS from the variation of temperature and punch displacement vs time diagram. Transmission electron microscopy (bright field) micrographs reaveled flow lines between the particles as a result of deformation and joining between particles during sintering. The structure of interface is nano crystalline where as the particles are completely amorphous. Quasistatic compressive properties of the SPS sintered specimens were studied at different strain rates. All the results suggest that the compacts of BMGs by SPS process opens a new possibilitiy of fabricating large sized components for structural applications.

Sol-gel coated hydroxyapatite on magnesium-zinc alloy for biomedical applications

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Metallic materials are the most favored ones in orthopedic application, due to their excellent load bearing capability, strength and toughness as compared to polymers and ceramics. Currently stainless steel, titanium and cobalt-chromium based alloys are commonly used in orthopedic implants. But these alloys have some limitations, like, release of toxic ions due to wear and much higher elastic moduli than natural bone, resulting in stress shielding.

Magnesium alloys can be a better option for orthopedic application. The advantages with magnesium alloys include its low density of 1.74g /cm^3, similar to bone tissue. The elastic modulus and compressive yield strength of Mg are also closer to natural bone, which reduces the stress shielding effect. Mg is biocompatible and biodegradable to human body. However, high corrosion rate of Mg results in loss of mechanical strength in chloride environment, limiting its application in orthopedic implant. Protective coatings of a suitable material system can tailor the corrosion rate of Mg based alloys. Hydroxyapatite (HA) can be the best choice for such coatings for following reasons. HA is an important inorganic biomaterial in orthopedic, due to its similar chemical composition and crystal structure as the apatite in the human skeletal system. Further, being a ceramic, it is inert in nature and resistant to corrosion. Aim of the present work is to develop a bioactive HA coating on magnesium alloy (Mg-Zn) through sol- gel route. The preliminary characterization of the coating was done for morphological and structural information, which is further followed by the detailed investigation of mechanical behavior of the coating (like, hardness, elastic modulus, adhesion strength to substrate etc.). Corrosion behavior of coated samples is investigated by electrochemical polarization test. This study explores the potential of HA coating on Mg surface to improve its performance in orthopedic application.

Mechanical and corrosion behavior of mechanically alloyed super saturated Fe-Cu system

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Extended solid solubility limits give us many advantages e.g., new phases can be formed thus giving rise to possibility of formation of new materials, fine grain size can be obtained giving greater strength, strength can further be increased as hindrance to dislocation movement is there, microstructure which are not possible from methods can be produced. Thus a desired combination of mechanical, electrical and magnetic properties can be achieved. Various techniques in use for extending solid solubility limit are; mechanical alloying, rapid solidification, laser processing, thermal plasma processing, ion mixing.It is found that solid solution can be readily formed in those systems containing elements with similar atomic radii and electrochemical properties. Fe- Cu system however is an exception. Although their atomic radii are virtually equal and affinities or other chemical properties are similar, the mutual solid solubility of the system is nearly zero at the temperature up to 3000C. Hence in the present investigation, it was planned to produce a Fe-10%wt Cu supersaturated solution by mechanical technique using high energy attrition mill. Solubility of Cu in Fe was confirmed by X-ray diffraction studies. The MA powder was then degassed and consolidated by hot pressing. Mechanical and corrosion behavior of the alloy was evaluated in comparison of pure Fe sample. The alloy was found to have hardness value of 454 VHN while the UTS was found to be 41.7 MPa. The corrosion resistance of the alloy was found to be three times better than the iron.

Effect of manganese addition on the mechanical properties of sinter-forged Fe-Cu-C alloys

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In the present study, the effect of Mn addition on the mechanical properties of Fe-Cu-C alloys under forged conditions was carried out. Fe-2Cu-0.7C and Ferro-Manganese (0, 0.5, 1.0, 2.0 and 3.0 wt %) powder were mixed in a double conned blender for 4 hours. The mixed powder was filled in a mild steel can of 62 mm diameter and 80 mm length and was sintered in a tubular furnace. Sintering was carried out at 1150°C with a holding time of 40 mins in N_2-10%H_2 atmosphere. Further, the sintered can (at 1150°C) was immediately forged in a channel die using a friction screw forging press of 100 ton capacity. The forged density was measured and the slab was further reforged at the same temperature to ensure almost full density. The homogenization of the forged slab was carried out at 1200°C for 2 hrs in muffle furnace. Tensile samples were prepared from the homogenized slab and were subjected to different heat treatments i.e. air cooling, furnace cooling, quenching and quenching plus tempering. Mechanical properties of the various specimens were determined using tensile and hardness tests. Micro-structural characterization of the samples was carried out with the help of optical microscopy and SEM. The effects of Mn addition on the tensile, hardness and microstructures under different heat treated conditions were studied and have been reported and discussed.

Effect of rolling on the microstructure of liquid phase sintered tungsten heavy alloy

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This study investigates the effect of hot and cold rolling on the microstructural properties of the liquid phase sintered tungsten heavy alloy (90W-7Ni-3Fe). Tungsten, nickel and water atomized iron powders were mixed and compacted at 200 MPa. The as-pressed pellets were sintered at 1500°C in reducing atmosphere. Microstructural analysis of the sintered pellets showed good interfacial bonding between the tungsten grains and Ni-Fe matrix. The as-sintered pellets were cold-rolled and hot-rolled to various levels (20%, 40%, 60%, 80% reduction in thickness). Microstructural investigation of the rolled sample indicates the increase in connectivity and contiguity of tungsten grains. XRD investigation revealed a decrease in crystalline size with increasing deformation.

Microstructure and corrosion study of spark plasma sintered duplex and ferritic stainless steel

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In the present work, high energy ball milled nano-structured duplex (Fe–18Cr–13Ni) and ferritic (Fe–17Cr–1Ni) stainless steel powders were prepared by planetary milling and consolidated by spark plasma sintering (SPS) at a temperature of 1000°C. Duplex stainless steel shows high hardness value of 765HV, whereas ferritic stainless steel shows 637HV. These values are quite high when compared with the normal hardness value for duplex (220HV) and ferritic (180HV) stainless steel respectively. Further, the hardness measurements were carried out at different loads such as 10, 25 and 50gf to study the effect of load on hardness. In both the type of stainless steels, the hardness value decreases with increase in load. Density measurement was performed using Archimedes principle and achieved around 95% density in both the cases. XRD spectra shows broadened ferritic and austenitic peaks for duplex stainless steel and only ferritic

peaks for ferritic stainless steel respectively. However, peak width slightly decreases after SPS in both the type of stainless steel due to the marginal grain growth and strain release. Linear sweep voltammetry (LSV) tests were employed to evaluate corrosion resistance of the samples. Corrosion studies were carried out in 0.5, 1 and 2M concentration of NaCl and H_2SO_4 solutions at different quiet time of 2, 4, 6, 8 and 10 seconds. Duplex stainless steel samples show more resistance to corrosion than ferritic stainless steel samples in both the electrolytes. Maximum pitting potential (E_P) of duplex and ferrite at 0.5M NaCl and H_2SO_4 are 0.63V, 0.57V and 0.18V and 0.14V respectively. Corroded samples were then characterized by FESEM, optical microscope.

Effect of milling condition on β-$FeSi_2$ phase formation

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Thermal management and energy crisis have been two major problems in this 21st century. Thermoelectric materials, which convert heat into electrical energy, are the energy resources of the future. Amongst the thermoelectric materials, β-$FeSi_2$ is emerging as an alternative promising high temperature semiconductor useful for temperature from 600-900˚C. This work focuses on synthesis of β-$FeSi_2$ semiconductor through powder metallurgical route. The powders of pure electrolytic iron (99.5%) and silicon (99.9%) were blended in an Attritor mill (AM) and Cryo mill (CM) for 4- 10 hr. Then it was compacted at 700 MPa and then subjected to heat treatment cycle. Phase transformations during the process were investigated using DTA, XRD, SEM and EDS. It was established that milling period, particle size, sintering temperature and sintering period has significant effect on β-$FeSi_2$ phase formation.

Optical properties of transition metal doped zinc oxide

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Zinc oxide (ZnO) is a wide band gap semiconductor with a large exciton binding energy. The optical properties can be tuned by suitable doping of other elements leading to potential applications in optoelectronic devices. In this study, we investigated the manganese (Mn) doped ZnO system with a general formula $Zn_{1-x}Mn_xO$ (x=0–0.25). The large solubility of Mn in ZnO coupled absorption in the visible region makes it ideal for optoelectronic device applications. Powders were synthesized by solid state sintering and the influence of Mn dopant on structural and optical properties was investigated. X-Ray diffraction measurements were used to optimize the sintering conditions. Scanning electron microscopy with energy dispersive spectroscopy was used to measure particle sizes and elemental distribution. Optical gap was measured by Diffuse Reflectance Spectroscopy and found to decrease with Mn concentration. These results show that Mn doped ZnO can be used in optoelectronic devices with precisely tuned optical band gap.

Advanced cermet systems for improved tribological performance

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TiCN-based cermets are considered as candidate materials for cutting tool inserts. Research has been focussed in designing the composition to improve the performance of cermets in complex wear conditions. In the present work, effects of TaC ternary carbide and Co binder addition on the wear behaviour of TiCN-Ni-WC cermets are studied. The powder mixtures with TiCN-20Ni-5WC, TiCN-20Ni-5WC-5TaC, TiCN-10Ni-10Co-5WC, TiCN-10Ni-10Co-5WC-5TaC compositions (all in wt %) are sintered to high density (>97%) by pressureless sintering at 1550°C for 2h in Ar atmosphere. Microstructures of all sintered specimens reveal typical core-rim morphology with variation s in size of core and rim phases. The sliding wear behaviour of sintered cermets against bearing grade steel ball was studied in unlubricated conditions at 5N load and 500 rpm speed. While the coefficient of friction varied between 0.4 and 1.1, the wear rate changed from 4.5 X 10^{-3} mm^3/Nm to 7.1X 10^{-3} mm^3/Nm with change in cermet composition. Minimum friction and wear rate obtained with the addition of TaC and Co in TiCN-Ni-WC cermet. The surface analysis of worn cermets indicates that material removed mainly by abrasion in the investigated sliding conditions. The wear behaviour is explained on the basis of core and rim phase sizes and mechanical properties of the sintered cermets.

Microstructural characterization of ZrO2 dispersoids during annealing in Fe-15wt.% ZrO2 model ODS alloy

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Oxide Dispersion Strengthened ferritic steels, which are known for their superior high temperature mechanical properties are candidate materials for future Fast Breeder Reactors. Ultra-fine dispersoid size and narrow size distribution range are key for attaining the high mechanical strength here. Yttria-Titania complexes are conventionally used as dispersoids for their better high temperature properties and they are well studied. Yttria being a cI80 structure is known to amorphize during mechanical milling, but reappears during annealing, growing up to micron sizes. Titania addition to the matrix can refine the size by forming Yttria-Titania complexes, but the size distribution is still quite broad. In this scenario, an alternative oxide with better dispersoid evolution mechanism needs to be investigated. In this respect, ZrO_2 is an excellent choice owing to its high temperature stability, low fast neutron absorption cross section and possibly most importantly lower number of atoms in the unit cell with mP12 structure. A composition of Fe-15wt%ZrO_2 was chosen for the experimentation because non dilute dispersoid content in the matrix helps in reliable XRD and TEM analysis. Dispersoid microstructure evolution with different milling times was studied and as predicted, ZrO_2 does retain its crystallinity under the same conditions where yttria amorphises. Also, majority of the dispersoids are skewed within the narrow size distribution range of 5-10nm. This has motivated us to study the behavior of ZrO_2 dispersoids at high temperatures about which very little is known. The 100h mechanically milled powders were pelletized, vacuum sealed in a quartz tube and annealed at different temperatures for various time durations in order to get insight

about recrystallization kinetics of the dispersoids. Characterization using SEM, TEM and XRD techniques are under way in order to understand the optimum time-temperature combination at which the dispersoids remains stable. Details of this research will be discussed in the paper.

Effect of alloying addition and microstructure parameters on mechanical properties of 93% tungsten heavy alloys

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Liquid phase sintering, heat treatment and swaging studies on three tungsten heavy alloys, 93W–4.9Ni–1.4Fe (Wt.%),93W-4.2Ni-2.1Fe-1.6Co (Wt.%) and 93W-4.9Ni-1.9Fe-0.2Re (Wt.%) were investigated in detail with respect to microstructure, tensile and impact properties. All the alloys were liquid phase sintered, heat treated and swaged to 34% deformation. The results indicate that Re addition reduces the grain size and contiguity of the alloy compared to W-Ni-Fe and W-Ni-Fe-Co alloys. W-Ni-Fe-Re alloy shows superior tensile properties in heat treated condition as compared to W-Ni-Fe and W-Ni-Fe-Co alloys. SEM study of fractured surfaces clearly indicates that the failure in case of W-Ni-Fe-Re was due to transgranular cleavage of tungsten grains, W-W de-cohesion and dimpled failure of the ductile matrix. W-Ni-Fe and W-Ni-Fe-Co alloys also failed by mixed mode failure. However, ductile dimples correspond the failure of the matrix phase was not seen. Thermomechancal processing resulted in significant changes in mechanical properties. Except for the Co-containing alloys all other alloys showed inferior elongation to failure values. W-Ni-Fe-Co alloy showed higher tensile (>1350 MPa) and elongation (12%). A detailed analysis involving microstructure, mechanical properties and failure behavior was undertaken in order to understand the property trends.

Ni-Ti SMA foam using pre-alloyed Ni-Ti powder and ammonium bi-carbonate as space holder

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Attempts have been made to make Ni-Ti SMA alloy foams with open pore characteristics using ammonium bi-carbonate as space holders. The ratio of Ni-Ti pre-alloyed powder and ammonium bi-carbonate in the mixture prior to cold compaction is varied with an aim to get porosity in the range of 60 to 80%. Similarly the size of ammonium bi-carbonate was varied in the range of 50 to 200 µm to get the cell size in the same range. The powder mixture was mixed with PVA binder and cold compacted at pressure between 100 to 200 MPa to make the cold compacted pallets. These pallets are then dried and Debinded to remove moisture and organic binder. Finally the pallets are sintered under vacuum (>10^{-4} mbar). At varying sintering temperature, the sintered pallets were characterized in terms of XRD, plateau stress, densification strain and strain recovery. It is noted that the foams are oxidized (5 to 10%) and the cells are interconnected. The cell walls also contain micro porosity. The plateau stress and energy absorption of these foams follow power law relations but the densification strain follows a linear relationship with relative density. Because of the microporosity in the cell wall and oxidation of the foams, strain recovery is noted to be only 0.8 to 1%. However, the strength and energy absorption of these foams are reasonably higher than that of conventional Ni or Ti foam. The process to be fine tuned to achieve better strain recovery.

Invited talks

Session 2: Nov 12, 2014: [16.00 - 18.00]

Evaluation of injection of agribusiness waste into blast furnace

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The search for alternative fuels for blast furnace has greatly increased in recent years. This is due to the limited resource of fossil fuels, and the increasing demand from the steel industry. The green house gas emissions from the fossil fuels also give rise to a competitive advantage for innovative processes that are less harmful to the environment by giving carbon credit points. This is done to enable the alternative fuels to enter into more competitive new markets, and can even lead to direct profits in accordance with the environmental conservation and earn carbon credits by recycling green house gases like CO_2 and CH_4. The present paper shows the feasibility of using biomass, such as rice husk, sugar cane bagasse, elephant grass, coffee husk and eucalyptus bark as substitute materials for powder coal injection into blast furnace; which leads to the replacement of part or even all of coal injected, which is not renewable and causes great environmental impact during mining andextraction. To simulate the condition of injecting fuel materials in the tuyere and raceway region of blast furnace, physical modeling and experimental setup has been developed and used for this purpose. Further analyses have been conducted for particle size classification, surface area, calorific value, proximate and ultimate analysis. It has been concluded that by using these biomasses it is possible to reduce the specific carbon consumption in the blast furnace burden, which enhances the company's environmental commitment, and moreover, it is possible to utilize waste generated in the agriculture.

Contributory papers

Session 1: Nov 12, 2014: [13.30 - 15.30]

Optical dilatometer: a new way to understand and quantify sintering kinetics of iron ore pellets

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Pelletizing and briquetting are the agglomeration techniquesmajorly practiced across the world nowadays. Fine particles adhered, and strengthened due to incipient fusion (sintering) when exposed to high temperatures. Sintering is an important phenomenon for powder compacts and pellets during their induration process. Sintering is becoming more important for a wide range of materials from the field of ceramics, composites, pharmaceutical, refractory, nuclear, minerals and ores. Sintering mechanism was determined by capturing their shrinkage and estimating their kinetic parameters. A recently innovated Optical Dilatometer captures the shrinkage two dimensionally, instead of traditionally used linear push-rod Dilatometer. Experiments with iron ore pellets (magnetite and hematite) of 10 mm diameter have been performed to compare both the dilatometers. Optical Dilatometer works on the principle of optics capturing shadow images, hence eliminating the physical interference with pellets. Therefore, the kinetic parameters estimated for different materials are more representative of the actual process. This gives a relatively simpler and more precise methodology to estimate the optimum process parameters for various raw materialswhich can be beneficial considering their variabilityand mix.

A study on microstructure of Vizag steel sinter

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The primeiron bearing feed material for Blast Furnace operations is Sinter. There are certain advantages of using sinter as opposed to using other materials which include recycling the fines and other metallurgical waste products which include flue dust, mill scale, lime dust and sludge. Simultaneously, a required amount of available lime in sinter eliminates limestone from the burden; thus calcination, which is highly endothermic reaction, can be avoided to take place inside the furnace. Theoretical calculation shows that approximately 0.26 kg of carbon is saved for every kg of limestone removed. Hence, it is known as the pre cooked food (prepared burden) for the blast furnaces. For the best results in the Blast Furnace, Sinter as a burden material must possess certain properties which are controlled by the composition and distribution of the different mineral phases formed. During the sintering process, the chemical reactions are taking place at high temperature and iron ores and fluxes are combined together to form a sinter cake composed of iron ore, SFCA, dicalcium silicates and glassy phase. Sintering reaction also regulate the volume fraction of each mineral which may affect sinter quality and hence, the performance of the blast furnace. The main objective of this study is to examine the influence of sinter chemistry on the phases present which are visualized through microstructures.

How dilatation helps in designing coal blends in metallurgical coke making

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Metallurgical coke is a vital raw material for steel making industry with blast furnace root of iron making. It constitutes about 40-50% of the cost of hot metal depending upon the raw material resources it owns/procures. Therefore, all efforts are being put by the steel makers to minimize the cost of coke by designing coal blends with maximum proportions of low cost coal sources (LCCS). These designs are either based on past performances and experiences on proportions of hard coking, Semi soft and other coals they use or by laboratory tests followed by pilot oven confirmation tests with considerations on oven health and target coke qualities (CSR, CRI, M40, M10 and AMS). The laboratories generally design the blend maintaining traditional coal characteristics like CSN, Fluidity and lateral expansion (for oven health and ease of operation) whereas the dilatometer test results can have more scope for further better proportioning of coal sources for coke quality maintenance as well as for oven health point of view. The study is aimed at with stamp charging technology of coke making which charges coal blend with higher bulk densities compared to top charge.The Audibert-Arnu Dilatometry test results are expressed in terms of expansion and contraction and on summing them; it is the total dilatation of the coal or coal blend. In the present study, the individual coal dilatation was used for taking decisions of coal blend constituents and their proportions. While higher expansion is detrimental for oven health, the same can be adjusted by incorporating fillers or inert type of coals. As it indicates the extent of coverage of inter particle interactions (solid-liquid phases) they encounter and also extent of duration particle wettability leading to comparable compatibility of the coals of the blend.

Modelling of iron ore sintering process

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Sintering is an agglomeration process which is an integral part of iron making plant. Fine iron ore, limestone and coke are homogeneously mixed with proper amount of water sprinkled to it. This homogeneous mixture is loaded to a moving strand. As material passes through the ignition hood, the top layer of the mixture is ignited which facilitates the sintering under the suction of hot process gases from the bottom. In present study, the sintering process has been modelled considering the heat transfer, gas flow, coke combustion, limestone dissociation, drying of grain, condensation of moisture and melting/solidification phenomena. Optimisation of various parameters used in the sintering process such as coke content, limestone content, moisture content, coke particle size, limestone particle size, grain size, gas velocity, bed porosity and ignition hood temperature has been carried out and presented here with respect to the burn through point.

Improvement of sintering operation by addition of hydrated lime solution in sinter mix

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It is well known that when burnt lime is added to sinter mix, the handling of feed materials increases the size of quasi-particles, improving sinter mix permeability, resulting in an increase in sinter productivity. The effect of burnt lime added to the sintering process, as a binder, to improve the granulation of sinter mix, is dependent on its completion of hydration process. In order to enhance effectiveness of burnt lime, it is sufficiently hydrated ($CaO + H_2O = Ca(OH)_2$) within the granulating time inside the mixer. However in the

conventional burnt lime addition system, where burnt lime of (-) 3 mm size is added in sinter mix in dry powdered form, does not get sufficient time to completes its hydration process, lead to loss of effectiveness of burnt lime in sintering operation. Additionally, burnt lime is also lost in handling them as dry granulated form during addition in sinter mix. These two major factors of the conventional burnt lime addition system led to poor granulation of sinter mix, low permeability of sinter bed, loss of valuable burnt lime etc., which consequently lead to fall in sinter productivity. In order to negate the above drawbacks of the conventional burnt lime addition system, R and D Centre for Iron and Steel, of Steel Authority of India Limited has successfully carried out laboratory experiments at its pot sintering set up in development of an innovative method for addition of burnt lime in sinter mix for improving the effectiveness of burnt lime in sintering operation. In the newly developed technique, burnt lime is premixed in water in a slaker separately to generate 'hydrated lime solution' at an optimum concentration ratio of lime to water. Then the hydrated lime solution is added in sinter mix in a mixer for effective balling and mixing of sinter mix. In this technique, burnt lime has been provided sufficient time for completion of its hydration process. Due to completion of hydration of lime in water, the effectiveness of lime as a bonding agent in sinter mix increases significantly, lead to increase in granulation of sinter mix in a mixer and permeability. This effective hydration process has also helped in decrease of burnt lime consumption in sintering operation. The successful laboratory experiments on hydrated lime solution of optimum concentration ratio of lime to water, addition in sinter mix, indicates improvements in various technological parameters of sintering such as, increase in productivity of sinter by 5 % to 8%, increase in strength of sinter by 2 %, significant reduction in consumption of burnt lime etc.

Introduction of AC drives in skip charging mechanism of blast furnace

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Skip winch charging system is a critical mechanism of Blast furnace. Around 24 blast furnaces of SAIL units below 4000 m^3 are equipped with Skip winches for charging the burden into the furnace.The skips of Blast furnace, travel upward to 85 meter from skip pit to reach to the discharging point. The velocity and rate of acceleration of skips need to be varied at different points as inclination of the skip bridge rail track is not uniform throughout the length. The skip, 98% of travel period is pulled up by motoring action and is decelerated by regenerative action at creeping speed to discharging position.Skip winches of the Blast furnaces are driven by DC motors and DC drives in-spite of many deficiencies. Stringent quality norms, power quality, energy conservation, productivity and non-availability of DC motors are the prime considerations which has led us to develop state of art controllers. Recently, AC drives have assumed paramount importance especially in the Steel industries and are capable to respond efficiently to all time critical applications. Active front end, 4 quadrants, AC drives with Induction motors were introduced in Skip mechanism in Blast furnace no.3 of Durgapur Steel Plant in April'2014. The project was successfully implemented for the first time in any Indian Blast furnace and fourth Blast furnace in the world. Sizing of motors was always critical since angle of Skip Bridge was modified from 52^0 to 54^0, travel distance exceeded by seven meters and filling capacity of skips was enhanced. Motors were selected by evaluating tangential load on the periphery of the rope drum for different burdens. AC drives were selected matching the overload requirement of the motors. This paper is an attempt to compare the performance of AC drives and DC drives.

Short reduction trial in tunnel kiln for pre-reduction of oxidized chrome ore

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Pre-reduction of oxidized chromite ores is novel process for minimizing the energy and coke consumption in Ferrochrome production. In this study, feasibility of carrying out pre-reduction in tunnel kiln was examined through a short reduction trial using coal composite agglomerates prepared from oxidized chromite ore with objective to understand the effect on extent of chromium metallization. Composite agglomerates in the form of spherical pellets, pillow shape briquettes and bricks placed in open as well as closed silicon carbide (SiC) boxes or saggar's were subjected to heating cycle in a tunnel kiln. Agglomerates in open boxes were reduced in presence of as is kiln atmosphere whereas in case of closed saggar's, the materials have experienced reducing atmosphere created due to reduction gases in immediate vicinity of the composite agglomerates. The tunnel kiln used for the short one trolley trial was 115m long having hot zone temperature of 1450-1500 ^{0}C and residence time of 4h in hot reduction zone at given trolley pushing rate. The temperature and reduction time in short trial were on higher side compared to sponge chrome process requirement of 1350-1400 ^{0}C and 0.5h residence time in hot zone. Pre-reduced pellets and briquettes after reduction obtained from the close SiC boxes have shown chromium metallization up to 80%, compared to brick shape samples having 40% Cr metallization. The highly metallized products have also resulted in material sticking inside the saggar's. The composite agglomerates reduced in as is kiln atmosphere have shown chromium metallization in the range of 10-20%. SEM analysis of products revealed three major phases namely partially reduced chromite, metal and slag phase. The short reduction trial indicated the suitability of tunnel kiln with SiC saggar's for further scale up trials of the process.

Prediction of iron ore sintering properties on the basis ofregression analysis and artificial neural network

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Iron ore sintering is a complex process of agglomeration of iron ore fines in presence of flux and solid fuel. Drastic change in the characteristics of the iron ore having low iron and high gangue content forced to understand their impact on sintering process. Recently, the iron content of iron ore varies between 50 to 60 %. Hence the prediction of qualities of the iron ore sintered product is necessary. Statistical tools are useful in determining the effect of individual parameters on sinter properties. The present study aims at predicting mean particle size (MPS), tumbler index (TI) and reduction degradation index (RDI) of Iron ore sinter by developing the Prediction model. Correlation coefficient, Regression Analysis (RA), and Artificial Neural Network (ANN) are used in the present statistical analysis study. RA technique uses only linear relationship where as ANN is a hyper-nonlinearity and ultra large-scale dynamic system, with strong robustness. As factors complicatedly affect each other during the sintering process, this work adopts the ANN analysis to construct the sintering quality prediction model. The prediction accuracy of MPS, TI and RDI using RA prediction model are 38, 72 and 48 % where as the accuracy are 79, 91 and 76 respectively when ANN model was used. It is observed that ANN model is advantageous over RA. The data used for

building this model is large set of data from Sinter Plant and a user-friendly interface has been developed by using C code for system software program.

Effect of bed height on the properties of hybrid pellet sinter produced from Bailadila slimes

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During iron ore mining and processing, around 10 to 12 % slime is being generated, which is creating environment and space constrain at mine site. The slimes are relatively very fine in size and contain high alumina and silica. Studies for beneficiation and subsequent hybrid pellet making have been taken up at National Mineral Development Corporation Ltd. The beneficiated slime is converted into micro pellets using disc pelletizer and used in place of iron ore fines during sintering. Effects of kinetic parameters including micro pellet size, suction pressure, the amount of coke in the sintering charge and limestone particles size on product sinter quality and productivity of sintering process were studied. Detailed studies were undertaken for studying the effect of bed height, keeping other parameter constant, on physical and metallurgical properties of hybrid pellet sinter. The sinter bed height was varied from 300 mm to 450 mm at a suction pressure of 1100 mm WG. The bed height has great effect on the productivity of the sinter and also has direct impact on sinter properties. Overall, the results from the laboratory scale tests suggest that the sinter produced at 400 mm bed height was meeting all the desired metallurgical characteristics of blast furnace feed with a resultant productivity more than 1.31 T/m^2/Hr.

Optimization of slag granulation parameters in iron making for its usage as sand

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With environmental restrictions on indiscriminate dredging of river sand there has been a growing need in civil fraternity for identification of alternatives to fine aggregates. Steel plants generate large amount of Granulated Blast furnace Slag (GBS) and research carried out in developed countries established GBS as a replacement of natural sand for various civil engineering works. A major drawback associated with the use of blast furnace slag in construction is the lesser bulk density (1000 -1100 kg/m^3) in comparison to that of river sand (1300-1600 kg/m^3) which results in lower strength of concrete. Microscopy studies showed that lower density in slags is due to its vesicular structure with pores generated during granulation. It was observed that the structure and bulk density of granulated slag varied significantly from the tap start to tap end. This confirmed the fact that slag density can be altered by varying the granulation parameters. Slag Granulation is primarily affected by water temperature, water pressure and water flow rate. In the present work water temperature, pressure and flow per ton has been varied during granulation and collected samples were analyzed for specific gravity, bulk density and structure through microscopy. It was found that under optimum conditions of granulation parameters, the bulk density of the slag can be increased to > 1400 kg/m3 as more compact structure can be developed in slag granules. Concrete and mortar cubes were made and tested for 3/7/28 day compressive strengths. With the high density slag the

strength of concrete cubes improved to meet the IS specifications. Granulated BF slag has been established as a good alternative to sand in construction.

Session 2: Nov 12, 2014: [16:00 - 18:00]

Mathematical modeling of blast furnace burden distribution

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Quantification of radial distribution of ore and coke is important for proper control of blast furnace operation. It has a close relationship with wind acceptance and gas utilization in a furnace and also influences the fuel rate, productivity and production of the blast furnace. For prediction of the radial distribution of ore and coke, a mathematical model is proposed for blast furnace burden distribution.The gamut of burden distribution model can be subdivided into two main parts: prediction of landing position of the material on stock level and prediction of heap shape. Burden trajectory model, based on single particle kinetics considering the geometry of the distribution system is proposed. The model is validated with the measurements using trajectory probe in several furnaces of Tata Steel.Based on detail experiments carried out at scaled down model and 1:1 scale model of blast furnace, mathematical expression for heap shape is proposed. The proposed correlation is independent of the dimensions of the system. The prediction of burden layers based on trajectory model and heap formation model matches well with the profil-o-meter measurements. The model is in use for control of blast furnace.

Blowing down and salamander casting of blast furnace no.1 for category-1 capital repair at Vizag steel, RINL

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In order to take the blast furnace out of operation for the start of the rebuild, the furnace must be emptied of coke and iron bearing materials. The process of blowing down the furnace involves operating the furnace without charging ferrous burden materials and allowing the coke to burn out up to the tuyere level. Immediately after blowing down the salamander castings are to be opened at hearth level sequentially. At the completion of salamander castings, furnace is to be quenched by discharging calculated quantity of water from nozzles installed at furnace dome & throat level. Blowing down, salamander tapping & quenching the furnace require excellence technological knowledge & rigid procedural approach for successful capital repair of blast furnaces.After operating for nearly 23 years it produced 37.74 million tons of hot metal, the blast furnace No.1 was blown down on 19th October, 2013 for category-1 capital repair. Before blowing down, the furnace went through rigorous preparation and adopted series of operational procedures. The identification of salamander tap holes location was so accurate that virtually no solid salamander was traced inside the furnace after quenching. Therefore activity of risky time consuming salamander blasting was avoided completely. This paper describes all steps and procedures adopted starting from preparation for blowing down till salamander and quenching of blast furnace no.1 of Visakhapatnam Steel Plant, RINL.

Slag regime for high alumina operation in blast furnace

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Blast Furnace feed is mainly sinter, high percentage variation of gangue material in iron bearing materials, is resulted in increase fluxed addition in sinter making. Fluxed Sinter is main feed in BF-3 and BF-4 at JSW steel which increases the high slag rate operation. Alumina (18-22%) decides the Slag rate because alumina decreases the fluidity of Blast Furnace Slag and greatly affects the furnace productivity.In view of the above; one of the options is to produce different Fluxing Sinter to feed the Blast Furnace to handling of high Alumina in Slag by optimum slag regime. To study the effect of different Fluxed Sinter on Slag regime for different basicity of forth phase slag system (CaO-SiO_2-MgO-Al_2O_3) has been studied for different basicties. Viscosity, liquids temperatures and deformation temperature of the four phase synthetic slag has been carried out to find out optimum values to handle high alumina in slag.Experimental results show for synthetic slag (CaO-MgO-SiO_2-Al_2O_3), Composition MgO (8-10%), Al_2O_3 (18-20%) and B_2 (1.00-1.05) are optimum for hot metal quality and lower slag rate. Viscosity of 300-350 centipoises at1500^{o}C and the liquids temperatures of 1400-1430^{0}C with sulfur partition ratio of 20-25 is suitable for the high alumina operations.

Improvement of pellet quality at JSW Steel, Dolvi

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Pellets are an attractive feed for BF because of its spherical shape and controlled size which ensures good permeability inside BF. It is an alternative method of agglomeration of fines which can not be used in sinter plant because of poor effect on sinter bed permeability. Pelelletization is an important process in a steel plant because it allows various waste fines (iron ore, lime, GCP, mill scale) to be re-used which would otherwise needs to be disposed off because fine size makes them unsuitable. Chemistry (basicity) and its physical properties are important parameters which govern pellet's suitability of use in a blast furnace, whereas physical properties such as CCS, AI, and TI determines pellet's tendency for fine generation and its effect on BF permeability. Present study compares pellets based on various features such as color (e.g. red & black). Around 90% of pellets generated are of black-grey color whereas red pellet generation is around 10%. These pellets are compared on the basis of chemistry (basicity, Alumina & Silica content) physical properties (CCS, TI, AI) and microstructure. Linder test was also done to compare reducibility behavior. Microstructure using SEM and XRD was also done for structure property correlation. This work makes an effort to correlate presence of various phases, chemistry, and porosity with physical properties.

Study of mechanical properties and extent of reduction of the agglomerated iron ore nuggets utilizing lean grade coal and coke dust fine with variation of pressure

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The problem of properly utilizing iron ore fines and non-coking coal in alternative iron making processes dates back long ago. Huge amount of dust from iron ore fines is created during, sizing, screening, and transportation in the plant and also during mechanized mining, beneficiation and agglomeration processes. The aim of this work is to achieve a method to utilize the inferior quality of fine raw materials and plant waste. The raw materials are analysed to calculate stoichiometric amount of reductant by proximate analysis, Wavelength Dispersive X-ray Fluorescence (WDXRF) and Thermo-gravimetric (TG-DTA) analysis. Study has been undertaken to assess the properties of Iron ore nuggets which are agglomerates of iron ore fines, boiler grade coal, coke dust fines and fixed binder compacted under four different pressures. Mechanical properties such as density, shatter index, abrasion index and compressive strength are studied for all the nuggets. One of the major advantages of nuggets is that theseare denser and can be produced using low grade raw materials without any restriction on the particle size (unlike the pellets) i.e. we can use very fine particles without Sintering. After that nuggets are charged in the tube furnace for reduction. The nuggets are reduced and the extents of reduction are calculated from the weight loss. The reduced specimens are characterised using X-ray diffractometer (XRD), scanning electron microscope (SEM), energy dispersive X-ray spectrometer (EDX), and chemical analysis method. It is observed that agglomerated iron ore nuggets can be significantly reduced by using the blended mixture of boiler coal and coke dust fines and with increasing pressure they show better mechanical properties and reducibility.

Operations- logistics of blast furnace (BFs) hot metal ladle (both open top and torpedo) movement and operating experience of handling salamander-1and 2 metal from BF#1 in VSP

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Hot metal ladles (HML) and Torpedo ladles (TLC) are being used to transfer BF product “Hot Metal” to steel melting shop (SMS) and pig casting machine (PCM) in VSP. Total no. of Ladles in the fleet is based upon the production of Hot Metal and consumption pattern of SMS. Ladles are moved through the railway tracks and there are many factors which decide the no. of ladle in the overall circulation. Moreover delivering the ladle carrying right quality of metal to the desired SMS and recording all the in process data for future analysis is a challenging job. The objective of the paper is to optimize the track time of HML & TLC as to optimize the total no. of ladles in the fleet. Ladles move from BF No. 1, 2, 3 to SMS 1, 2 and intermediate stations of weighbridge, ladle repair shop (LRS) and torpedo ladle repair shop (TLRS). Different activities of ladle movement circuit requires, receiving of hot metal from BFs into ladles, weighing in weigh bridge, moving to SMS, unloading the hot metal into mixer and then return to BFs for next cycle of tap. In some occasions a few HMLs’ are being sent to PCM and in case of repairs, the ladles

are taken to LRS/TLRS. All these activities are complex and time consuming in nature. Data analysis as made in terms of arrival rate and service rate i.e. waiting and unloading time duration of Ladle in both SMS and PCM, estimation has been made to find the no. of ladles required in circulation. With the help of ladle information management, increases the ladles availability in cleaned condition so as to utilize the maximum capacity of the ladles. It improves no. of ladles delivered per shift, increases transport efficiency resulting in better capacity utilization of railway track and mixer. An attempt has been made to quantify the effect of reducing the track time of the ladles in circulation on ladle life. A generalized conclusion has also been made in this paper based on last one year data of ladle movement in BFs in reducing the track time and enhance the ladle life in RINL-VSP. The paper also reviews operating experience of handling salamander-1 and salamander-2 metal from BF#1(Godavari) and making zero boulder.

Utilisation of titanium bearing magnetite ore blended with hematite ore to produce pig iron using lean grade coal

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The increasing shortage of high grade iron ore (Fe_2O_3) has focussed attention on the use of other sources of iron ore. Titaniferous magnetite ore is a special type of magnetite ore having high titania content (23.23% TiO_2 present in this case). Due to high TiO_2 content and high density, the conventional smelting reduction of TMO is difficult. In the present study, the preliminary characterization of titaniferous magnetite ore (TMO) and hematite ore (HMO) has been investigated by WDXRF, XRD analyses. Again TG/DTA, proximate and ultimate analyses are performed for the characterization of lean grade coal. TMO fines (below 75µm) and HMO fines (below 75µm) have been separately agglomerated with lean grade coal in the form of briquettes using binders like bentonite and molasses. These green briquettes are dried first in oven at 423K for 30 min and then reduced isothermally in tube furnace with varying parameters like temperature (1323K, 1373K and 1423K) and time (30 min and 60 min). After reduction, the reduced briquettes are characterized by XRD analysis. The best reduced TMO and HMO samples are taken and blended in three different weight percentage ratios of 1:4, 1:8 and 1:12 of TMO:HMO. The chemical analysis of three blended samples is carried out and degree of metallisation of Iron is found to contain 83.24%, 92.12% and 93.12%, respectively. These three blended samples are briquetted using binder like bentonite and lime. Thereafter these blended briquettes are separately smelted in raising hearth furnace at 1773K for 30 min. The pig iron formed is characterized using XRD analysis, microscopic analysis and EDX analysis. It can be concluded that more than 90% yield of pig iron can be achieved with the charge mix where blend ratio of TMO:HMO is 1:6.5 and amount of TMO (maximum) is 13%.

Optimisation of sintering operation through permeability control at sinter plant, Bokaro

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Sinter is a feed material to blast furnace in the Steel Industry. In the sinter plant agglomeration of iron ore fines into lumpy porous mass occurs by incipient fusion i.e. sintering. In conventional sinter machine, water is added in the balling drum for getting balling of sinter mix in the balling drum for maintaining permeability in the sinter machine. The optimum addition of moisture in sinter mix is an important factor

in sintering operation. Permeability is the difficulty of gas passing through the solid bed. The fluctuation and change of gas are related to the processes of mass transfer, heat transfer and physicochemical reactions. Therefore, permeability of sintering bed plays a decisive role in smooth operation of sintering process, thus affects yield and quality of sinter. Permeability is commonly referred to as permeability of sinter mix bed. This paper depicts the innovation of a sinter mix permeability control system by controlled granulation and sinter bed voidage for increasing productivity of sinter machine through water flow control in balling drum and a method of its implementation. The invention provides system and method for maximization of bed permeability as a function of green mix moisture wherein the water flow rate is controlled to operate the process maintaining permeability close to the P_{max} value of the parabolic permeability-moisture curve involving continuous on-line feed backward system wherein computer evaluated intended moisture content in the sinter mix is continually fed to continuously activate corresponding changes in flow of feed water into the balling drum implemented through a Level-II PLC based process control system leading to increase in sinter machine speed and thus increasing the productivity of sinter plant.An increase in speed of sinter machine by 16% and in ISO TI of sinter by 3% is observed.

Production of different types of cokes in a coke oven plant, for two blast furnaces of dissimilar capacity

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Coke is one of the most important raw materials fed in Blast Furnace in terms of its effect on Blast Furnace operation and hot metal quality. It is a major input factor in Blast Furnace which accounts for around 60% of hot metal cost. Coke strength after reaction (CSR) and Coke Reducibility Index (CRI) are two important properties of coke that affect the performance of Blast Furnace. This CSR & CRI value drastically affect the Blast Furnace Operation, good CSR value also support to increase PCI Injection rate which reduces the coke rate & hence reduces the operation cost of Blast Furnace. To produce better quality coke we, at JSPL Raigarh, operate with Non Recovery/Heat Recovery coke making technology with 176 ovens 2 blocks of 8 batteries each with 11 ovens in each battery of SSIT China.Since Commissioning of coke oven, single quality of Coke was produced for both the Blast furnaces. The requirement of both the furnaces have been found to be different, so optimized coal blending were used to suit both the Blast furnaces. Targeted value of CSR and CRI was tried near to 64 & 26.But with this value, neither of the Blast furnaces could be satisfied. Frequent problems were observed in MBF due to high CSR. High Coke Consumption in BF - II due to low CSR and so less PCI injection. As JSPL Raigarh is having two Blast Furnaces one of 1681 m^3 and another one is of 650m^3 the requirement of coke strength of both the furnaces is different to met the requirement of both our customers we are producing coke of different CSR & CRI value for both customers in a single day operation by selecting the time duration for different coke blends charging and pushing.

Raw materials security and importance of their properties in iron making

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At present, the production of iron is mainly done in the blast furnace in the form of hot metal (also called pig iron which is 94% pure). The main materials needed for blast furnace are iron ore and coke. But, sometimes to increase the reducing behavior of ore dolomite, manganese ore, quartzite and limestone is added. The main aim of this paper is preparation of burden for the blast furnace which reduces the coal/coke consumption as much as possible without compromising with the production and quality of iron produced. This can be done with the help of increasing the quality of raw materials and addition of waste materials. Since, production of pig iron in blast furnace depends upon many factors such as reducibility, size and size distribution, strength, temperature and range of softening, iron, moisture and gangue contents, swelling and volume change properties etc. By suitable handling of these properties can improve the productivity and overall performances of blast furnace. Also, raw materials accounts 60-70% cost of iron and hence addition of waste plastics along with coal/coke can reduced the cost and consumption of materials without affecting the quality of product formed and at the same time they serves as waste utilisation. Hence, this paper shows a proper handling of factors affecting the production in blast furnace and the waste materials utilization for the reduction of the cost of the product without affecting the quality. More research and study of the same will sure be beneficial for iron making.

Making of sinter cake from mill scale and coal fine

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Mill scale is an industrial byproduct that is available in plenty from hot rolling of steel sheet/ingot. It's a rich source of iron since it is composed of various oxide of iron mainly Fe_3O_4. It is thrown away as waste, which is not desirable for environment. The objective of the research is to find out the effective and economical way for utilization of mill scale. The research works on for making sinter cake from mill scale and coal fine, which are cheaper and easily available. Study was carried out with different amount of coal, under isothermal condition and fixed time of reduction for degree of reduction. Percentages of coals were varied more or less than stoichiometric amount. Reduction was performed isothermally at the 950 °C for 1 hour. Sinter cake was formed after that and tested sinter product for strength and reducibility.

Session 3: Nov 13, 2014: [15:30 - 17:30]

Effect of MgO bearing flux material of different sources in iron ore pellets

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MgO is an important flux material in high alumina hematite ore pellets which can reduce RDI of pellet and improve other properties too. It is available in form of either Magnesio silicate (Pyroxenite, olivine, dunite) or magnesite or dolomite. At present various naturally available minerals containing MgO viz. olivine, Pyroxenite, dunite, dolomite are widely used in different plants. However properties of each of the above fluxes are physically and chemically different and they contain different types and quantities of gangue materials like silica, alumina, and calcium oxide. Though MgO has specific roles, other gangue materials

carried by MgO bearing fluxes may also change the phases or slag bonding in pellet that changes the pellet properties. Thus different MgO bearing fluxes shows different behavior in pellets. It is imperative to study the actual role of MgO and effect of different gangue materials in it. The aim of this study is to examine the actual role of MgO and its optimum requirement as well as the effect of other gangue materials in MgO bearing fluxes. The work was carried out with a high alumina Noamundi hematite ore. As different MgO source, pure MgO, olivine and waste magnesite brick powder have been used separately in pellet making. The properties of green pellets like drop numbers, green compressive strength, dry strength have been measured. The pellets were indurated at varying temperature in a chamber furnace and subsequently characterized by measuring cold crushing strength, apparent porosity, reducibility indices, reduction degradation indices and swelling indices as per standard. The phase identification and morphology has also been studied through XRD analysis and optical microstructure. The study has revealed that olivine fluxed pellet shows highest CCS and pure MgO fluxed pellet shows lowest CCS. In addition to this RDI in pure MgO fluxed pellet is as low as 7.5% for 0.9 % MgO level in pellet; the waste magnesite brick fluxed and olivine fluxed pellets show 28.8 % and 17 % respectively. Reducibility indices for olivine pellet decreases drastically due to the high amount of slag formation by silica in olivine. MgO containing fluxes have high gangue content which is helpful from strength point of view but pure MgO provide good RDI, RI, and swelling properties than other two.

Estimation of state of the Deadman and its relation to the stability of BF operation

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Lower part of blast furnace has been a complex subject of research for long times. The possibilities of direct measurement of internal state of the furnace in this region of liquid generation and flow are practically non-existent. However, there are continuous measurements of the furnace interior at certain accessible locations in the upper part of the furnace. Presently, these measurements are monitored in isolation for determining few control actions. Nonetheless, little effort has been made to correlate these measurements of the upper region with the measured parameters of the lower part of the furnace. Any information which can be extracted by an integrated study of these upper region measurements with the tap variables of the cast-house could be vital in estimating conditions prevailing inside the blast furnace hearth. In this paper, a novel method of blast furnace data analysis is presented to estimate the state of deadman inside the hearth. The data of stock-level (measured continuously through two radars for diametrically opposite radial positions of the top burden profile) is simultaneously examined with the dataset of tap variables measured at the cast-house level. The proposed method of data analysis is shown to provide leading information regarding casting irregularities and furnace instability.

High top pressure operation of mini blast furnaces with enhanced productivity and reduced fuel rate in JSW Steel Limited, Salem Works

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JSW Steel, Salem Works is the only integrated steel plant in Tamilnadu which is also the largest special alloy steel maker in India. This plant has two Mini Blast furnaces of 402 m3 and 550 m3 with CERI, China as technology supplier. Considering the locational constraint of being far away from the raw material supply sources, incurring substantial logistics cost, it becomes imperative to contain the cost of production to deliver the goods in the competitive market. It is indeed a challenge to the entire JSW team to achieve this through process improvements, innovations, standards etc. however miniscule it may be. One of the major process improvements achieved in Blast Furnace II is the High Top pressure Operation, which is a significant achievement in these sized furnaces. High Top pressure Operation has several advantages in Blast Furnace Process starting from higher productivity, lesser fuel consumption, proper gas distribution, cleaner hearth, lesser flue dust losses etc. Modern bigger volume blast furnaces will have in built technology of High top pressure which comes with the technology and design where as these furnaces were not designed with that concept. In mini blast furnace such as Blast Furnace II, for achieving high top pressure operation, detailed study of all process parameters and their various effects on furnace performance, equipment capability, effectiveness and methodologies for strengthening the weak links etc were conducted with meticulous planning. The constraints were addressed through well aligned and systematic technical solutions. In Blast Furnace II, top pressure which was maintained earlier in the range of 0.8 to 0.9 Kg/cm2 was increased up to 1.25 to 1.3 Kg/cm2 which gave exuberant results to achieve and surpass many major milestones. This paper details the necessity for maintaining high top pressure operation, constraints faced in the Blast Furnace, methodologies adapted for overcoming those constraints and finally the results &benefits.

Operating experiences at JSPL, BF-II (India) - Achieving higher performance indicators with inferior raw materials

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Blast Furnace can be considered as the most important reactor in commercial use in modern time, accounting for a majority of world total steel production through this route. Modern steel making demands superior quality and quantity hot metal at lowest cost. This requires an economic modern blast furnace with consistent, reliable and well-balanced operation of the furnace and all its support systems. Efforts have been continuously made for BF process optimization, to improve the energy efficiency, productivity, cost competitiveness and campaign life of a blast furnace. The BF2 at JSPL India, over the years has set-up many milestones, surpassing productivity beyond 3.0 t/m3WV/day with high hot blast temperature >1200 ^{o}C and pulverized coal injection > 200 Kg/tHM, in-spite of not having the best raw materials in industry.This paper deals with the experiences and difficulties faced during the stabilization and operation of that furnace. The various steps taken, process modifications done and parameter tuning like coke quality derivation for different PCI rates, O2 enrichment stabilization & control, PCI stabilization & control, burden distribution & charging control and raw material quality control are described.

Stabilization of sinter plant at IISCO, SAIL, Burnpur

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IISCO steel plant, SAIL, is currently on the path of modernization scheme under its 2.5 MMTPA expansion plant. In order to cater to 80% requirement of Blast furnace burden, a state of art sinter plant complex comprising of 2 X 204 m^2 sinter machines will produce 3.88 MMTPA gross sinter.Sinter plays a vital role when quality and cost of hot metal production are in focus. During startup, we faced some process related problems like high sinter return fines (internal) generation, weak sinter, improper top surface ignition, high waste gas temperature, crack formation on sinter bed, etc. These problems occurred mainly due to inconsistent quality & improper size granulometry of raw material, & improper water addition. We analyzed the cause of problems & took some remedial actions. To minimize sinter return fines (internal) generation, we regulated the oversize in basemix, maintained coke crushing index > 85%, optimum moisture addition and proper sinter bed loading. We installed inclined 40 mm gratings below basemix weigh feeder in its chute to eliminate entry of +10 mm size ore fines and boulders in basemix. For improving balling optimizing water addition we increased the dry mixing zone. For proper sinter bed loading, one V-shaped leveler plate is installed. After implementing these changes, we reduced return fines generation from 35% to 25%. The Tumbler Index value of sinter improved from 72 % to 75 %. The coke crushing index was maintained between 85 to 90%. The waste gas temperature is brought down to 165°C from 175°C. Sinter bed sidewall ignition also improved from 800°C to 900°C. This paper focuses on understanding the factors affecting sintering & quality. It also describes the advanced sintering technology supplied by Outotec at ISP, some practical problems we faced in sintering and their solutions implemented in our plant.

Commissioning and stabilisation of large capacity blast furnace at Vizag steel

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Large capacity blast furnace (BF#3)with a useful volume of 3814 $m^3$of Visakhapatnam Steel Plant was blown-in on 24th April 2012.The furnace is built with state of art technology to produce 2.5 million tons of hot metal per annum. Furnace was blown-in with special burden with an aim to normalise the operations in shortest possible time. This paper deals with aspects of commissioning of large capacity furnaces w.r.t testing, trial runs, burdening, cast house management and logistics. In spite of many challenges, normal operating regime could be attained within 20 days of blowing-in and achieved 50% rated capacity with silicon in hot metal < 1.0%. But owing to limited availability of raw material logistics (delay in commissioning of new sinter machine) and difficulty in off take of hot metal (delay in commissioning of SMS-2), the furnace was run in throttled condition till October'13. Several challenges were experienced while running the furnace in throttled condition. However, after blowing down of blast furnace-1 and subsequent stabilisation of the new sinter machine in November'13, rated capacity of 7150 t/day was achieved almost within one month period by producing 7155t of hot metal on 20th December, 2013. This paper provides an insight into the various challenges faced during commissioning and subsequent stabilization.

Balling drum degree of filling and its' effect on sinter quality

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Balling is a process of layering of fines on bigger particles, and feed is classified into nuclei (>2 mm), intermediate (0.5-2 mm) and fines (<0.5 mm). It depends on feed property (granulometry, morphology etc.) and process operating conditions (water content, drum rpm and angle etc.). Poor crushing index of flux and fuel and higher fraction of ore fines in +8 mm size range resulted large concentration of +3 mm particles (>25%) in feed. During balling process, size dependent distribution of residence time was experienced i.e. bigger particles rolled out faster without sufficient layering mechanism. During subsequent stages disintegration of weak balls caused random distribution of particles across bed. In vicinity of large particles, increased combustion zone thickness and reduced combustion temperature resulted insufficient diffusion bonding and recrystallization/grain growth. On the other hand, localizations with greater fines concentration was characterized with quick reaction and shortened residence time at high temperature. Sinter quality deteriorated, in terms of high return generation (-5 mm at 14.3%) and low Tumbler Index (+ 6.3 mm at 70.10%). Balling drum degree of filling was increased from 5.59% to 11.18%, by resorting to single balling drum operation. As residence time is independent of feed rate, this population enhancement increased layer thickness and ball strength which subsequently resisted disintegration. Near homogeneous distribution of fines in proximity of larger particles could enhance efficiency of heat utilization, resulting enhanced bonding mechanism and strength. Sinter quality improvement i.e. reduction in sinter return (- 5 mm at 9.08%) and increased T.I. (71.37%) could be realized.

Steady state numerical model of COREX melter gassifier

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COREX is one of the alternative iron making processes, which uses non-coking coal and oxygen to reduce iron ores. It consists of two reactors, namely, reduction shaft, and melter gassifier, where former is placed above the later. Unlike other processes, apart from reduction shaft, it consists of one more dome shaped counter-current reactor named melter gassifier, which is divided into four reaction zones. The reuse of top gas as high calorific export gas and absence in requirement of coke makes it more profitable than other processes. It has attracted many researchers toward the challenge in performing the measurement of various parameters inside the furnace. Various mathematical models published in literatures show the variation of some of the operational parameters and their influence on output variables. There is a need of a comprehensive model which can calculate and update the operational parameters, depending on the quality of coal available. This model tries to predict temperature, composition and velocity field of different phases inside the melter gassifier, using published relations among various parameters. Addition of an optimization sub-module to it can help us to predict the variation of these parameters with input quality, and optimal feeding parameters of coaland DRI. The model is validated using industrial data and can be used as a guiding tool for COREX ironmaking process.

Feasibility study for the production of ferro-titanium from titaniferous magnetite ore of East Indian origin through pre reduction and subsequent melting operation

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Thriving demand as well as consumption of steel makes the researchersto think beyond the conventional route. This increases number of attemptto uselean grade iron ore for the production of steel. Titaniferous magnetite ore is an important source of iron, titanium and vanadium. The present work is aimed at studying the feasibility of recovery of valuable metal fraction from Indian titaniferous magnetite ore and subsequent production of ferroalloys. However, utilization of titaniferous magnetite to yield ferroalloys poses a problem as separation by physical beneficiation of ilmenite enriched non-magnetic fraction from the vanadium bearing magnetic fraction is difficult due to the mineralogical and geo-chemical characteristics of the ore. The raw ore is subjected to characterisation after crushing. The crushed ore is subjected to carbothermal reduction with stoichiometric amount of activated charcoal at 1200^0 C for 60 minutes and 120 minutes. The reduced ore is then characterised by XRD, SEM and EDX analyses. The sample with higher reduction is then subjected to magnetic separation. The non magnetic fraction is collected for aluminothermic reduction, where calculated amount of pure aluminium powder is used as a reductant, Barium peroxide powder is used as heat booster and Magnesium ribbon is used as heating agent. Aluminothermic reduction has been carried out with 10 %, 20 %, 30 %, 40 %, 50 % and 60 % excess over stoichiometric requirement of aluminium. The yields of ferro-titanium for all the samples have been thoroughly characterized by XRD, SEM, and EDX analyses. Optimum addition of extra aluminium powder is determined from the graph of percentage of Ferro-Titanium formed with percentage of extra Aluminium added. The optimum amount of percentage excess aluminium is found to be 30%.

Reduction of coke moisture at JSPL to reduce coke rate for blast furnace

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Coal is one of the major and costliest input raw materials in steel industry. Because of its inherent nature and properties it has become indispensible in iron making through Blast Furnace (BF) route. In BF, iron ore is reduced to hot metal with the help of coke which provides heat of reduction and sensible heat. In order to reduce the manufacturing cost of hot metal, it is necessary to reduce mainly raw material cost. The coke comprises of major chunk of the cost of the hot metal in BF. In view of the volume of this material and its cost, BF operator always seeks to operate at minimum optimal coke rate. Moisture content is one of the important parameters among the many variables affecting the coke rate. Increase of moisture in coke increases heat loss due to evaporation and super heating of vapor. The objective of this project is to reduce the moisture in coke from 8 to 3.5% in order to reduce the hot metal cost. Coke moisture and its consistency has significant role in BF. Fluctuations of the moisture level are reflected as fluctuations in the energy supply to BF there by disturbing the process and also wasteful use of this expensive energy source. Therefore, the BF operator always prefers to have low moisture coke. This paper emphasizes the steps that have been taken to reduce the coke moisture in coke oven plant at JSPL, Raigarh unit.

Effect of SiO_2 on the reduction of chromite sinter for ferrochrome production

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Ferro-chrome is presently produced from lump chromite ores in the submerged arc furnace.Majority of the lump ore is very lean (~30% Cr) in nature. Production of a better quality ferro-chrome requires using high-grade chromite ores; but these are fragile and tend to form fines during handling. In order to utilize these fines direct sintering of chromite fines has been carried out to produce fluxed sinter which contains 36% Cr_2O_3, 20% FeO, 8% SiO_2, 9% MgO, 11% Al_2O_3, 6% CaO etc. In the present study the performance of developed chromite sinter to produce ferrochrome in graphite-lined 50 kVA electric arc furnace has been conducted and the effect of SiO_2 on smelting property has been studied.The thermodynamic calculations for the chromite sinter smelting (Cr_2O_3-Fe_2O_3-SiO_2-CaO-MgO-Al_2O_3) were done with the Equilib module of FactSage 6.4 to study the effect of silica in lowering the slag melting temperature. It was found that with increase in SiO_2, the melting point of slag decreases. It has also been found from the calculations that required SiO_2 concentration would be 33% to obtain a slag of melting point around 1660 ^{0}C. Thus calculation suggests external SiO_2 addition of 656 g is necessary for 10 kg sinter. In actual experiment equivalent amount (752 g) of quartzite was added as source of SiO_2.Smelting experiments were conducted in presence of coke as reductant and several parameters viz. tapping temperature, specific power consumption, flux (silica) requirement, metallic recovery has been studied. The slag tapping temperature measured 1789°C (100 °C super heat above the slag melting temperature) and was appeared to be sufficiently fluid, having viscosity 1.1 poise. Thus, quartzite (SiO_2) addition decreases the liquidus temperature of slag, as has been found from the thermodynamic estimation. The obtained Fe-Cr alloy has metallic yield of 81%, chromium recovery of 88% and specific Power consumption of 13 kWh/kg of Fe-Cr alloy. Produced ferrochrome has composition 55%Cr, 36% Fe, 7.8%C, 1% Si and the final slag Cr and Fe was very low (0.76% Cr_2O_3, 0.98% FeO). Thus addition of SiO_2 provides an encouraging result in smelting of developed sinter to produce ferrochrome.

PROCESS METALLURGY FOR FERROUS METALS: SPONGE IRON

Invited talks

Session 1: Nov 14, 2014: [08.30 - 10.30]

Gas based DRI/HBI - an ideal metallic for quality & environment friendly steel making

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For over two decades gas based DRI(Sponge Iron) has emerged as clear winner as a strong substitute for scrap and now it has become necessity to use gas based DRI as feed stock to produce quality steels through secondary (EAF/IF) route . The secondary route steel making process is environment friendly as it uses recycled material for melting and processing into finished steel. Generally speaking, world over secondary steel making is synonymous with EAF technology. India however occupies a unique place in the world scenario, since significant production of steel comes from IFs. There are number of reasons for this, whereas the most relevant one being very low skill of operation, requiring low investments and flexibility to scale up/down production depending on the market forces.

IFs similar to EAF generally require much clean scrap, since their ability to handle large slag volumes is limited. Consequently, refining processes, which entail reactions between slag and metals, are usually not feasible. DRI produced using gas based technology has distinct advantages like High metallisation, Low S&P, high carbon ,very low tramp elements and very low gangue content.

Quality steels require high-tech & sophisticated melting practises, which is possible only in EAF and to some extent in IF's. It is easy to carry out refining operations in EAF to produce cleaner steels. Impurities in steel such as sulphur, phosphorus, tramp elements, nitrogen, hydrogen etc are known to have detrimental effect on the properties of steel. DRI being a very clean metallic has gained tremendous importance and recognition as an important raw material to produce quality steels through secondary route. Continuous depletion in availability of good quality scrap & increase in demand for special quality steels has made secondary route to substitute gas based DRI for Scarp. Usage of DRI in the charge mix decreases the refining requirements resulting in simplification of metallurgical processes. All the recently commissioned steel plants around the globe have selected large sized furnaces with continuous charging of DRI. Secondary steel sector will continue to dominate the production of quality steels in the future .EAF route is environment friendly and accounts for up to 30% of world steel production and is growing rapidly.

Quality control orders – Impact and opportunities

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The article is about the relevance of the Steel and Steel Product (Quality Control) Order issued by Ministry of Steel. The Order was issued in pursuance of decisions taken in the Ministry to ensure availability of quality steel to consumers and empower the consumers to take legal course against supply of substandard steel products. Most of the issues relating to implementation of the Orders have been resolved in

consultation with Bureau of Indian Standards (BIS) and stakeholders. Integrated Steel Plants have already implemented the Order in letter & spirit. Foreign suppliers have also got themselves registered with BIS to enable them to supply quality products as per relevant Indian Standards. Units in secondary sector have reported problems in implementing the Order and issues are under consideration of Ministry of Steel. Non availability of quality sponge iron suitable for production of quality steel particularly, in Induction furnace, is an issue of concern, and is being resolved in association with BIS & other stake holders. None the less, quality is important and quality steel products must be made available for all applications. India is a developing country and going to be a super power in years to come; we must not therefore, compromise in quality of steel used in all critical applications.

Opportunities and Challenges in the Sponge Iron Industry

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The sponge iron manufacturers have entered into a territory of uncharted dangers. The Supreme Court rulings of various contexts and against various appeals have almost closed the access of sponge iron units in the procurement of coal and iron ore. The prices of raw materials vis-à-vis the finished product hardly cover costs of scale up investments and research into new technologies. The rising importance of gas based DRI in the USA and Middle East riding on the recent discoveries of shale gas has undercut the possibilities for India's coal based DRI. Gas based DRI can be transported with greater ease than the coal based DRI and hence the latter remains localised resisting the trends of globalization which means an increased dependence on not only local raw materials but also on local demand for sponge iron. The second kind of danger comes from the processes of integration within the steel industry as those plants with electric furnaces or blast furnaces start to produce sponge iron. The integration of sponge iron into the value chain of steel retards its prospects of retaining its market worth as an independent commodity; future hopes around price subsidizes and there is soon no more interest in the product as a commodity with exchange value although it may continue to have use value. If sponge iron plants set up induction or electric arc furnaces signifying forward integration then there is a clamour among the fellow producers to raise the price of their products in order to earn more surpluses for investments into forward integration. However, if the trend favours backward integration of steel producers into sponge iron production, the situation turns pessimistic for the future of the DRI industry. The sponge iron prices should be such that the producers should earn enough to look towards forward integration into steel rather than steel producers seeking to backwardly link into the DRI industry.

Waste reduction in rotary kiln based dri production, experience in Tata Sponge Iron Ltd.

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Steel making through secondary route using Direct Reduced Iron (DRI) has taken the world industrial scenario towards a new horizon . Particularly in India the production of steel through secondary route adds to about 30% of the total steel production. Because of scarcity of coking coal which is a vital raw material for BF-LD route of steel making and availability of non-coking coal, the growth of sponge iron (DRI)making through Rotary Kiln Technology increased from 1980's onwards and after development of small Furnace like 50 TPD & 100 TPD, the growth reached the peak by mid2000. Non coking coal along

with lumpy iron ore are the vital raw material for DRI production through Rotary kiln process. However Production of DRI through rotary kiln process is highly energy intensive and generation of carbon dioxide is very high. In addition to that as it handles 0–40 mm Iron ore, 0-200 mm non-coking coal and dolomite, the micro-fines generated in various raw materials processing stage is high which pollutes not only air but also surrounding land. Besides that as the solid waste generation is 0.4–05 T/S.I, the handling of waste and storing is a problem for the industries as well as to local vicinity. Further to the pettiness, with deterioting raw material quality day by day, the manufacturers are not only facing low productivity, lower campaign life, high raw material consumption, but also the waste handling, energy consumption and GHG gas emission is increasing. In this paper, an attempt has been made by optimum blending of different raw materials having different properties suitably and by adopting model based close process control to optimize productivity, reduce specific raw material consumption , energy consumption , GHG gas emission and solid waste generation .Further the paper deals with the efforts of TSIL to recycle and reuse the solid waste for making the process a clean technology and ensuring the conservation of natural resources for the future which is essential for the sustenance of rotary kiln based DRI production.

Session 2: Nov 15, 2014: [11.00 - 13.00]

Model-based optimization and decision support system for sponge iron production

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Production of sponge iron or direct reduced iron (DRI) involves reduction of iron ore by coal or by reducing gases such as carbon monoxide and hydrogen in a rotary or a vertical shaft kiln. India is the largest producer of DRI in the world and majority of plants in India utilize non-coking coal for reduction of iron ore in a rotary kiln. The direct reduction process in a rotary kiln involves a number of physico-chemical phenomena such as pre-heating and drying of iron ore and coal particles, devolatilization and gasification of coal, heat transfer between solids, gases and kiln wall by radiation/convection/conduction, step-wise reduction of hematite to magnetite, magnetite to wustite, and wustite to metallic iron by carbon monoxide and hydrogen, flow of solids inside the kiln, and heat transfer across refractory wall by conduction and heat loss from outer shell by convection and radiation. Since the process is complex in nature and involves a number of process variables such as kiln speed, Carbon-to-Iron ratio, air profile, etc., it is difficult to determine optimum settings for key manipulated variables, especially when sponge iron plants have to deal with iron ore and coal from multiple sources. Model-based optimization will be useful in this regard as the plant engineers can utilize software tools for process optimization incorporating various process and equipment constraints in addition to product quality constraints. A representative mathematical model that incorporates the phenomena mentioned above and that takes into account characteristics of iron ore and coal along with air profile and kiln geometry would be a valuable component of a model-based optimization system. Secondly, with the advent of sophisticated process automation and information technology solutions, it is now possible to integrate information from all sources in the plant and undertake global optimization which will help in taking informed decisions. The mathematical model and the optimization system could be integrated with the plant automation systems such as a distributed control system or a manufacturing execution system and with the laboratory information and management systems. The optimization system could be converted into a decision

support system incorporating fundamental knowledge of the process as well empirical knowledge derived through data mining and analytics, and heuristics derived from experienced plant operators. Our work on mathematical modeling of sponge iron production in a rotary kiln, data analytics and thoughts on model-based optimization and decision support systems will be discussed in this presentation.

Contributory papers

Session 2: Nov 15, 2014: [11.00 - 13.00]

Influence of iron ore quality on the yield of coal based DRI plants

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The story of direct reduced iron (DRI) also known as sponge iron industry is very interesting. The three-decade old industry came into full existence very strongly all on a sudden when mini steel plants were looking out raw materials randomly. Production of coal based sponge iron was taken as a viable option. It is a high quality metallic product obtained from iron ore, pellets etc. as a steelmaking feedstock. It offers an alternative steel production route to conventional blast furnace- basic oxygen furnace and scrap-electric arc furnace routes. Here iron ore is reduced in its solid state and then converted to steel in electric arc/induction furnaces. It offers an attractive option for its small scale low capital investment requirements and also its suitability to local raw material situations.Many of the plants are operating with inferior grade raw materials thus producing less than rated capacity. Inferior raw materials speak in terms of performance quality, production and material consumption rates. A 100 tpd plant produces 70-80 tpd and 500 tpd plants produce somewhere around 300-330 tpd. The intermediate capacity plants also produce lower than the designed capacity accordingly. Most of the stakeholders wonder particularly for lower production /yield rates. The yield has been found to be dependent on iron content, loss on ignition, moisture content and laterite present in the iron ore. Present paper underlines the existing truth involved in the lower yield rate as regards iron ore quality.

Reduction kinetics of iron ore coal composite pellets in rotary hearth furnace - effect of pellet shape and bed packing materials

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Most of the iron is presently produced in blast furnace using metallurgical coke. The depletion of coking coal reserves and growing environmental concerns have motivated researchers to search for a coke free and environmentally friendly iron making process. Also large amount of iron ore fines and coal fines are generated during mining and a large fraction of these remains un-utilised. Therefore, several attempts have been made to find alternative routes of iron making where non coking coal and iron ore fines could be used. Based on the above considerations, several alternative iron making routes like Fastmet, ITmk3 etc. have been evolved where iron ore and non-coking coal fines are utilized in the form of composite pellets and reduced in rotary hearth furnace (RHF) to make direct reduced iron (DRI) which can be sent for subsequent melting and slag separation via EAF/EIF route. In RHF, the principal mode of heat transfer to

the pellet bed is by radiation. The major problem of RHF lies in low productivity due to heat transfer resistance through multi-layer bed and consequently limited layers over the hearth. In the present study, an attempt has been made to improve the heat transfer in such system by increasing the specific surface area of the individual pellet, which has been accomplished by changing the shape of the pellet. The effect of different bed packing materials and quality of raw materials on the degree of reduction has also been studied. The reduced pellets have been characterized through measurement of porosity, degree of reduction, degree of metallization, and shrinkage. Although pellets with higher specific surface area yielded higher extent of reduction when treated in the form of single pellet, the pellets of different shape did not show any noticeable change in reduction when those are reduced multilayered bed at RHF. Multilayer pellet bed packed with coal, graphite, and sand shows lesser degree of reduction as compared to the bed without any packing material. It is further observed that for reduction of multilayer bed pellets at RHF, pellets with higher grade coal containing higher fixed carbon and lower volatile shows improved degree of reduction. However, pellets with certain grade coal exhibits higher extent of reduction when the internal coal is reduced even below the stoichiometric requirement.

Developed and undeveloped area of pelletisation technology

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Low grade iron ores are not suitable for direct smelting. These ores require to beneficiate before use in the blast furnace or DRI. Huge amount of fines are generated during mining and beneficiation of low grade ores. Fines cannot be used directly in blast furnace and most direct reduction furnace because they are too fines to use. Recovery of iron bearing minerals from these fines is a vital concern for the resource utilization as well as environmental aspect. There is only one way to use fines through agglomeration root. Hence these fines are pelletized into usable pellets. Pelletisation is a very old technology. Generally pelletisation of iron ore fines is carried out using some binders because binders hold the ore particles together so that agglomerates can obtain. Various types of inorganic and organic binders such as bentonite, lime, $Ca(OH)_2$, slaked lime, dextrose, molasses, sodium polyacrylate (SPA), carboxymethyl-cellulose and corn starch etc. alone or in combination such as CaO+Dextrose, Slaked lime+Dextrose, CaO+Slaked lime+Dextrose, $Ca(OH)_2$+Molasses, Slaked lime+Molasses, Sodium polyacrylate+Slaked lime etc. Pellets made with these binders are indurated for desire strength as per iron making. There is no any proper binder by which pellets are produced with desire strength for iron making without induration. Some chemical binders produce high strength pellets at low temperature but these are very costly. Induration is an uneconomical process and produces large amount of particulates and NO_X, mercury, SO_X, HF and HCL may also be emitted in the atmosphere depending on trace elements present in ore, fuel and binder. In the present review paper an attempt has been made to highlight the area where R&D works are required for beneficial of iron and steel industries as well as environments.

Optimization of iron ore pellet size fraction for enhancing the quality and production of sponge iron

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Pelletization is an agglomeration techniques used to utilize iron ore fines produced during iron ore mining. India is now not only producing iron ore pellets but also exporting now to other countries. Pellets are superior to lumps in the sponge iron making process. Pellet size range of 8 to 16 mm is generally used in sponge iron industry. In the present paper major physical strength parameters for different size fraction of iron ore pellets were examined. Also size variation of raw material feed inside the rotary kiln at different lengths is studied. Magnetic and non-magnetic properties of pellets and its effect on physical strength and internal structure were also analysed. It was observed that tumbler index, RI and CCS were negatively correlated with the pellets size. Also the magnetic content of pellets increased with increase in size.So a suitable size fraction of 8 to 12.5 mm with optimum mechanical properties was suggested for enhancing the Sponge iron production. This suggested size range will also improve the Sponge iron quality. Higher mechanical strength and abrasion resistance of pellets can enhance the production rate of sponge iron by 5-10%, under identical operating parameters. Also, high abrasion resistance will result in low fine generation inside the rotary kiln which will subsequently result in low accretion formation inside the kiln. High mechanical properties will also result in low fine generation during the transportation of iron ore pellets.

Sustainable processes in sponge iron making

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While the blast furnace is expected to remain the world's chief source of iron units for steel making as long as adequate supplies of suitable coking coals remain available, at present sponge iron keeps gaining importance contributing mentionable bulk of the world's total iron making capacity in which India has the top share. Though the sponge iron process does not need highly polluting coke ovens and sinter plants associated with the conventional blast furnace, environment and waste management in sponge iron plants is also of prime importance.Though the major part of DRI is used as a substitute for scrap in electric arc furnace, it has been established that the DRI could also be well used in the Blast furnace itself giving rise to a decrease in coke rate and an increase in productivity. The mechanism by which it is achieved needs the understanding and appreciation of the fundamentals of physico-chemical principles of direct reduction and those in the BF.This paper attempts an analysis of the above guiding points and solutions.

A study on reduction of mill scale by low grade coal

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Mill scale is a hot rolling by-product of steel making, usually of 2mm thickness. It constitutes iron in elemental and combined form as wustite (FeO), hematite (α-Fe2O3) and magnetite (Fe3O4) .Mill scale causes environmental pollution and is thus landfilled. Study was done to recover iron from mill scale through direct reduction with low grade coal (from Nagaur district, Rajasthan) in presence of lime (10% by weight of coal) at 900°C temperature to produce sponge iron. In this paper, size of mill scale was varied, to study its effect on reduction behavior. Detailed characterizations of reduced samples were studied by SEM (Scanning Electron Microscope) and XRD (X-Ray Diffraction).

Life enhancement of injection coal pipe

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The Injection coal pipe is critical equipment in coal based DRI plant. It is used to inject coal from outlet side of Kiln to cater the requirement of coal at middle zone and outlet zone of rotary kilns. The coal which is used as solid fuel and as well as reducing agent in coal based DRI plants is fed from both ends of kiln. The washed coal which is fed from inlet side of Kiln is known as Feed Coal and the coal which is fed from outlet side of Kiln is known as Injection coal which is combination of coal fines and lumps. To inject the coal inside the Kiln, there are 3 equipment's used are known as Roots blower, Rotary feeder and injection pipe. Roots blower gives high volume of air with high pressure, Rotary feeder gives uniform discharge of coal through injection coal pipe. This pipe should in a straight line with center of Kiln to provide uniform coal distribution inside the Kiln up to middle portion of Kiln. Injection coal pipe is a combination of two pipes (MS & SS Pipe) with flange joint. MS pipe is connected to rotary feeder & at the end of MS pipe, SS Pipe connected with a Flange joint. Approximately 35% of SS pipe of its length will be inside the Kiln. These portions of SS pipe have to withstand a temperature of approx. 1050^0C & collide with accretion pieces inside the kiln. Due to high temperature, rapid and continuous collides with accretion pieces, SS pipe got bends at its edge. The desired coal throw profile disturbs due to this bending of SS pipe. After once it was bend, it creates lot of problems regarding disturbance of coal profile, localized overheating and ultimately leads to impact on quality of sponge iron.

Reduction of mill scale and iron ore

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Mill scale is the flaky surface of hot rolledsteel, iron oxides consisting of iron(II) oxide (FeO), iron(III) oxide (Fe2O3), and iron(II,III) oxide (Fe3O4, magnetite). Mill scale is formed on the outer surfaces of plates, sheets or profiles when they are being produced by rolling red hot iron or steel billets in rolling mills. Reduction of mill scale facilitates to obtain sponge iron or iron powder which can be reutilized as metallic load in steel making or can be used in production of iron based powder metallurgy parts. Direct-reduced iron (DRI), also called sponge iron, is produced from direct reduction of iron ore (in the form of lumps, pellets or fines) by a reducing gas produced from natural gas or coal. The reducing gas is a mixture, the majority of which is hydrogen (H2) and carbon monoxide (CO) which act as reducing agents. This process of reducing the iron ore in solid form by reducing gases is called direct reduction. Direct reduction, an alternative route of iron making, has been developed to overcome some of these difficulties of conventional blast furnaces.

IT application on process control in coal based rotary kiln KPI tracker, Tata Sponge Iron Ltd.

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Coal based DRI often face problems of accretion, shorter campaign life, fluctuation in product quality, high coal consumption. The problem is compounded with deteriorating quality of coal and iron ore. The manufacturers, who do not have captive mines, source raw material (coal & ore) from multiple sources with deteriorating and varying quality. The operator is faced with frequent changes in the input characteristics affecting process stability and is required to often manipulate large no. of parameters to maintain a stable process condition, which is a very difficult task. Typically, DRI process involves measuring incoming raw materials (coal, ore & dolo) characteristics for determining their suitability for operation. For day-to-day operational control, hourly samples are taken from circuits for coal and iron ore, and cooler discharge materials. Their characteristics are determined based on laboratory test and intimated to operator/process executives for taking necessary actions for controlling the process. In addition, other parameters such as temperature inside the kiln at different zones, airflow rates, kiln rpm, ore and coal flow rates, finished product output, gas volumes and temperature at different zones, operating conditions of critical equipment and auxiliary systems, etc. are measured. Traditionally, these were recorded in manual log sheets not amenable to quick analysis and decisions for better process control. In later period, SCADA workstations were installed and operating conditions like temperature, pressure, airflow, material flow rates, equipment conditions were captured using PLC systems. To manipulate these conditions, the process operator/executive needed analysis and trends of other interrelated input and output characteristics such as raw material and product characteristics, which was not normally possible with data recorded in manual log sheets. Over 200 variables and 2000 observations are captured per kiln every day. It is obvious that process operators find it difficult to control the process in their day-to-day operations. At best when the process goes out of control, normally the process-in-charge takes decisions based on his experience and often by trial and error method. Hardly, the operator could take decision. With deteriorating raw material quality and multiple sources of varying quality, the process goes out of control most often. The results are shorter campaign life, frequent shutdown, higher raw material consumption and firefighting operation with low operator morale. To improve this situation Tata Sponge adopted Information Technology and used it effectively to manage its DRI operation. It has implemented an in-house designed s/w application named **KPI Tracker**, which captures all data from Quality Management System, E-Log Book, PLC systems, and SAP ERP application into a single repository. It provides information including trends, correlated trends and critical process parameters on-line with respect to its KPI Targets for better process control.The application enables the operator and process executives to control the process by providing on-line information and trends on several process parameters. Some of the important ones are Raw Material Quality, Shell Temperature, C/Fe Ratio, Product Quality (FeM) and break down data of critical equipment's to enhance overall plant productivity.

Invited talks

Session 1: Nov 13, 2014: [09.00 - 11.00]

The role of slags in steel refining and continuous casting

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It is well-known that slags play an important part in refining of steel. In order to optimize the steel-refining processes, it is important not only to understand the properties of slags; but also to apply the knowledge for improving the prosesses. In the case of mould flux slags, viscosity and thermal conductivity are very important for optimum heat and mass transfer. The present lecture illustrates a few case studies with respect to slags for steel refining and continuous casting.

Case Study 1: Oxygen in steel

Ovako steel had experienced problem with their analysis of oxygen in steel. In order to estimate the oxygen content in steel, they had used the "FeO" analysis in slag and calculated the oxygen content in steel by assuming slag/metal equilibrium. The values thus obtained showed a large scatter. Using the THERMOSLAG software, the calculations made via the sulphur route assuming that there was equilibrium between the sulphur in the metal and in the slag. Sulphur analyses could be made with higher accuracy. The oxygen content values thus obtained showed much better consistency and were reliable.

Case Study 2: Slags in dephosphorization

With the sponsorship from TATA Steel, a project was started on the optimization of dephosphorization in steel. Slag path from plant trials were combined with laboratory measurements of phosphorus partition. It was found that the phosphorus partition reaches a plateau at basicities (defined as (CaO/SiO_2ratio) beyond ca 2.5. The optimum value for the FeO content in the slag was ca 20 wt%.

Another off-shoot of foaming studies in slags was the direct addition of $CaCO_3$. Laboratory and plant studies show that $CaCO_3$ addition improves foaming as well as thermal insulation and there is a net saving of energy despite the endothermicity of the decomposition of $CaCO_3$. This step is considered to even enhance dephosphorization and possibly reduce the amount of FeO in the slag minimizing refractory erosion.

Case Study 3: Mould flux slags

An important aspect of mould flux design is to have an understanding of the thermo-chemical and thermo-physical properties of these slags. A database has now been developed which summarizes the properties of mould flux slags which is meant to be of help to the casters. Another important aspect is the melting rate of the flux. X-ray visualization experiments were carried out with Pt markers in order to monitor the melting rate of two different fluxes, one for normal steel and another for stainless steel. The difference in the melting rate was significant and could be quantified.

Session 2: Nov 13, 2014: [13.30 - 15.30]

Critical assessment of morphology & macro-segregation in continuously cast steel slabs

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Over the years, continuous casting (CC) has almost attained the level of maturity and become the most favoured route of production of semi-finished steel products worldwide. However, in spite of several developments, CC products still suffer from various surface as well as subsurface defects viz., cracks, centreline segregation, porosity and pinholes, etc. Most of them have been linked to the heat extraction from liquid steel from the solidifying strand. During solidification of steel and alloys latent heat is released in the mushy zone. Mode of solidification in actual casting is typically dendritic whose morphology changes with progress of solidification. Equiaxed dendrites tend to grow ahead of the columnar solidification front once the liquid steel superheat is almost fully dissipated. The solidifying dendrites tend to reject solute elements in the residual coexisting liquid phase within the interdendritic spaces and in front of them, giving rise to segregation of solutes on both micro as well as macroscopic scales in the solidified casting. Flow and accumulation of highly segregated liquid during the final stage of solidification may cause problem of severe macrosegregation around the cast centre. Such centreline segregation may subsequently undergo undue phase transformations, causing deterioration in mechanical and welding properties of steel besides making the steel susceptible to cracking and severe corrosions. Therefore, reasonably good understanding of liquid steel solidification behavior during casting is prerequisite for the successful production of quality steel. Towards this, judicious application of scientific fundamentals coupled with experimental investigation can help immensely in mitigating some of those problems. Over the years at Tata Steel, several works have been carried out for improving the quality of steel, increasing productivity through smooth casting operations. Some of those investigations include: establishing the characteristics of solidification, determination of severity of segregation in cast billets and slabs, identification of various phases generated in the segregated region besides thermodynamic evaluation of inclusion precipitation during liquid steel solidification. Glimpses of some of those works are presented here.

Contributory papers

Session 1: Nov 13, 2014: [09.00 - 11.00]

Effect of hot metal Si-content on designing the blowing regime in terms of lance height and oxygen flow rate in LD converter steelmaking

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In converter steelmaking, oxygen at supersonic speed is blown on hot metal to remove the impurities like carbon, silicon, manganese, phosphorus. During the blow the lance height determines the slag formation,

decarburization, foaming characteristics, slopping and spitting of metal. These phenomena are only partly understood till date, and to some extent predicted with the help of several on-line measurements and on-line control models. With variations in silicon content of the hot metal, the amount of slag produced changes and the slag volume directly proportion to the silicon content. Variations in volume of slag affect decarburization, slag-foaming characteristic, slopping and spitting; hence it becomes difficult to predict a simple blowing pattern for all kind of input hot metal chemistry. So blowing pattern needs to optimized and different blow patterns with soft jet having less depth of penetration or hard jet with more depth of penetration is required for variable parameters. The depth of penetration varies the metal droplet generation rate hence the refining of the slag. In this investigation wt. % of Si has been categorized as low, medium and high for refining and blowing regime i.e. optimization of blowing lance height as well as oxygen flow rate are established in terms of the foaming index of slag, the metal droplet generation rate, reduction rate of FeO of slag, the values of ratio of depth of penetration to bath height.

Improvement in BOF steel making using sublance and level-II

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Now days in modern steel making, a cost efficient production is of top priority. This can be done by improving production equipment focused on an increase in production capacity, to fulfill an ever increasing market demand for steel. Sublance system along with level-II Static and Dynamic process control Model (SDM) has been a tool, known since 1980's to steel professional for enhancing productivity and quality. Sublance system has been long proven technology for reducing tap to tap time and thus increasing production without any extra capacity addition. Currently more than 175 Sublance systems have been installed worldwide. Advantages of making steel using Sublance and level-II Static and Dynamic process control Model (SDM) was unknown to the Indian steel industry till the commissioning of the first Sublance at SMS-I in February 2013 at JSW Vijayanagar works. One year later, JSW Steel has become the first Indian steel plant to be fully equipped with Sublance system along with level-II Static and Dynamic process control Model (SDM). JSW along with Danieli Corus successfully installed and commissioned 7 sets of Sublance in steel melting shops of JSW Vijayanagar works. With the Sublance system, information on the analysis and temp of the steel bath in the converter can be gathered, without interrupting the blow or tilting the vessel. This is a significant improvement for the steel making practice, because a steel analysis can be obtained, while the steel making process is in progress. In this way, a recalculation for oxygen amount to be blown and coolant to be added can be performed by the level-II Static and Dynamic process control Model (SDM) to reach blow end target without the necessity of a Reblow. At JSW Vijayanagar works with the above mention advantages of Sublance system BOF cycle time, Al Cons, and Reblow is reduced. JSW Vijayanagar is further installing Phosphorus prediction model to have direct tapping/Quick tapping facility. Also in upcoming year the aim will be to have a push button steelmaking in converter to be totally controlled by the Level-2 system.

Modeling of dephosphorization using bloated droplet theory in basic oxygen steelmaking

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A considerable fraction of refining of hot metal in Basic Oxygen Steelmaking is carried out in emulsion layer by interaction between the metal droplets and slag. The metal droplets are ejected into the slag when the oxygen top blowing is started and these droplets undergo refining in emulsion and return back to the bath. During this period, the carbon in metal droplets reacts with available oxygen to form carbon monoxide. Above a certain threshold decarburization rate, the evolved carbon monoxide inside the droplet causes the droplet to swell which increases its surface area. Earlier studies have shown that the swelling affects the residence time and also the rate of certain interfacial reactions. In present work, efforts have been made to study dephosphorization in metal droplet in emulsion by coupling dephosphorization kinetics with the 'bloated droplet theory'. As observed in industrial furnaces, the calculated phosphorous concentration in droplet was lower than that of metal bath and was dependent on residence time, instantaneous diameter of the droplet and slag composition in the converter. The results obtained were in satisfactory agreement with the available data and a step ahead in improving the understanding of dephosphorization in steelmaking.

Improved process technology for production of special steel at SMS-I, RSP, Rourkela

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The first ever installed converters (65t) in India are at Steel Melting Shop-I, Rourkela Steel Plant, Rourkela to produce 0.5 MT of various special steels such as CRNO, Tin Plate (TP), API, SAILMA, SAILCOR etc through BOF-VAD/VOD-LF-CC route. The stringent quality requirements of special steel requires a high degree of precise control of operating parameters as well as immediate response to changes and interferences during production. Operation of small sized converter is always a challenge to meet the first turn down conditions, especially, oxygen potential of slag. This resulted in decrease in converter lining life as well as carry over slag creates difficulty in secondary refining of steel leading to high level of rejection and down gradation. Efforts were made for improvement & optimization of steelmaking practice to enhance steel quality and to reduce the rejections. The BOF flux addition system and its addition pattern have been modified for improved slag formation. Secondary refining practice was redesigned to improve de-oxidation, de-sulphurisation, and steel cleanliness by optimizing the characteristics of ladle top slag. BOF slag FeO was controlled in the range of 20-25% in majority of heats by adoption of accurate and timely flux addition. This led to increase in lining life from 1500 heats to 2016 heats. Suitably designed ladle top slag w.r.t. composition & fluidity enhanced de-sulphurisation level to 58% as compared to 40% in conventional practice. Lower sulphur (<0.015%) helped to improve core loss values for CRNO grades resulting in up gradation to 60% as compared to 52% heats. Concurrent reduction of sulphur & silicon increased the acceptance level of TP grades to 80% as compared to 52% heats. Additional benefit accrued in term of reduction in Al consumption by ~10% for special grades of steel.

Physico-chemical model for deoxidation of liquid steel in ladle refining furnace

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System considered for the investigation is Fe-Mg-Al-Si-O. In this present investigation, physico-chemical model is developed to study the liquid steel deoxidation and to predict the deoxidation products present in the steels. Basic Thermodynamic equations are the governing equations for physico-chemical model. It has two parts Deoxidation Calculator and Inclusion Predictor. Deoxidation Calculator is designed in such a way that the amount of deoxidizers required to add for producing particular grade of steel are predicted and the scientific information like activities of the components present in liquid steel and the slag generated after deoxidation are also obtained. Total nine different deoxidation products from MgO-Al_2O_3-SiO_2 system are considered in this study. Inclusion Predictor gives an idea about the stability of deoxidation products in the liquid steel. Total 5 high temperature lab scale melting experiments excluding the preliminary experiments are planned in the air induction furnace with argon atmosphere based on the model predictions. Micro structural characterization of the steel samples for identifying the type of deoxidation products, deoxidation products distribution are done and the morphology of various deoxidation products are studied under SEM. Powdered slag samples are characterized by XRD technique to identify the complex deoxidation products present in it. Validation of physico-chemical model predictions is done with literature available data, industry data and lab scale experiments. Data collected at RINL and JSW Steel Ltd is used for validation. Deoxidation Calculator predictions are have good agreement with industry data and lab scale experiments shows good agreement with Inclusion Predictor results. Mechanism for the evolution of MgO-Al_2O_3-SiO_2 based complex deoxidation product is proposed in this study based on the results of physico-chemical model, experiments and the characterization studies of deoxidation products. An industry oriented computer program for liquid steel deoxidation is developed and the copyrighted version is named as DeoxyCalc – Version 1.0.

Innovative hearth lining design and repair methodology for twin hearth furnaces at steel melting shop-1, Bhilai Steel Plant

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During the process of steel making, the hearths of the furnaces are subjected to high temperature and high degrees of turbulence and as a consequence, erosion of hearths takes place. However, as a furnace goes on producing more and more number of heats, the degree of erosion suddenly gets severe at times due to an increase in the number of heats tapped at higher tapping temperatures and aggravated further due to inferior quality of refractory mass. Hence the maintenance of the furnaces through techniques of sound repairs is of paramount importance for meeting the targeted steel production. This aspect is then taken care of by the well-defined system of repairs.Twin Hearth Furnaces of SMS-1 at Bhilai Steel Plant, SAIL, were suffering a major setback in terms of crippled furnace availability for production, due to increasing downtime of furnaces, on account of higher frequency as well as longer duration of Hot Repairs. This crucial situation was further aggravated with a couple of Hearth Bottom-Through conditions and other hearths handicapped with severe erosion of Bottom Refractory. This reduction in Furnace availability and utilization rendered curtailed production regimes and tired workforce. The usual solution to the above

problem was going ahead with capital repairs needing more than 25 days which was replaced with faster repair procedures targeted to be completed within 10 days. The modifications, “Selective Dismantling” curtailed dismantling time, followed by addition of a new hearth brick laying design named as the “Bath Tub Design”, were first implemented in hearth 1R. After establishing advantages of the new techniques achieved through pilot study in terms of operational benefits, cost benefits and maintainability of the hearth, these new repair methods were implemented in all the furnaces. Post implementation advantages were studied thereafter the new method of repairs were incorporated as Standard Maintenance Practices of THFs in SMS-1.

Turn around performance by Durgapur steel plant

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Durgapur Steel Plant has been achieving best performances year after year since its inception. In the preceding years, the shop achieved its all time best performance which is quite well above the capacity and in the year 2013-14, the shop has already achieved best ever performance surpassing the previous best performance till now and has crossed 2 million ton mark in the crude steel production for the first time in the history of the plant. It is worthwhile to know that its rated capacity is 1.802 million tons. During the preceding years as has been mentioned there has been significant improvement in major techno-economics of the plant especially specific energy consumption clocking an all time low of 6.38 GCal/tcs which has been the best figure in SAIL so far. This has been attributable to improvement in caster production, coke consumption and TMI apart from other factors like operational efficiency. During the same time, caster output has been also at its all time best and there are remarkable improvements in all the areas. Total caster production has also gone up and slated to cross 1.7 mt this year which is also best ever performance. With this improvement, DSP has scaled new heights and achieved stellar performance in hot metal, crude steel and saleable steel production including techno-economics and has taken a giant leap forward to establish the number one position in SAIL family.

The journey of development of API 5L X-56M grade plates in 22.23mm thickness through TMCP route at JSPL- Raigarh

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Line Pipes are used for mass transportation of gas, oil or water generally in a pipeline or utility distribution system. For efficient transportation, modern pipeline designs use larger diameters and higher operating pressures. This calls for requirement of steels with high tensile properties. To function better in the colder regions, and/or for the transportation of liquid natural gas (LNG) steel should have low ductile-brittle transition temperature. All these prerequisite requirements for the line pipes are specified and documented by American Petroleum Institute (API) standard API 5L. Since its inception in 1948 with only one grade X-42, as of now higher grades are available upto X80 are developed due to ever increasing thrust on cost reduction Jindal Steel and Power Limited (JSPL), Raigarh has been one of the pioneers in rolling API grades. JSPL has successfully developed the API grades up to X70M in lower thickness of plates and coils at Raigarh plant, although in higher thickness JSPL has successfully achieved the desired

properties in plates 15.88 mm of API X56 grade also. However journey of the new product development started with the customer enquiry for API X56 plates in the higher thickness of 22.23 mm. The challenge was to set the optimum chemical composition and rolling parameters considering higher plate thickness and various constraints in the plant design. Trial rolling based selection of optimum chemical composition and thermo-mechanical processing parameters made it possible to achieve the desired results. To meet the high standard quality analysis requirements, the company has all the testing facilities including state of the art HIC & SSC laboratories.

Composition and temperature control augmentation strategy during ladle refining using simplified physics and ThermoCalc

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Ladle or secondary refining is a decisive process in steel making that determines the quality of output steel. Neoteric and ultramodern technologies in steel making process strive for stringent and unerring quality steel with reduced cost of production. Therefore scientific control of process parameters during ladle processing is indispensable. This project deals with prediction of alloying and fluxes additions and requirement of arcing to meet and control the chemistry and temperature at output. Technique used for optimising additions is thermodynamics based mass balance. Temperature based estimation is based on simplified physics and heat balance equations. We have developed chemistry and thermal balance model which has been then used with some knowledge based rules to arrive at control strategy for alloying and fluxes additions and arcing scheme. Mass balance is used to find initial total additives required which is dispensed in 3 steps and finally we used ThermoCalc to find left amount of additives needed to achieve our final composition. We are obtaining amount of various alloy additives and fluxes and arcing power desired for accomplishing the steel's final composition and temperature.

Nitrogen pick-up in steel through EAF Route

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Steel making through electric arc furnace (EAF) is one of the most popular and versatile steel making practices followed all over the world. In recent years, there has been a substantial increase in production of steel making via EAF route. Superior quality with low cost has become a yard stick for the steel manufacturers to meet the customer's demand. The challenges are to produce steel with low residuals and low gaseous elements to serve the critical quality sensitive steel markets like automobile, API line pipe, Boiler and ship building. Absorption of nitrogen during steel making results in interstitial solid solution strengthening and grain refinement due to the formation of nitrides, both the factors increase the hardness of the steel. Presence of high nitrogen content may result in inconsistent mechanical properties

in hot rolled products of steel, embrittlement of the heat affected zone (HAZ) of welded steels, and poor cold formability, reducing the ductility of cold rolled and annealed low carbon aluminum killed steel. The work describes in detail the factors responsible for the nitrogen pick-up in steel produced through EAF operations mainly considering the charge-mix along with tapping variations.

Development of effective slag conditioner for iron and steel making

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Soda Ash, a reagent used basically for desulphurization of hot metal in ladle, but in some of integrated steel plants it is also used as a slag conditioner after external desulphurisation. The main disadvantage of using soda ash is that it generates tremendous amount of fumes which are toxic in nature and not environment friendly. After external desulphurisation, it is essential that slag generated during this operation is removed as much as possible from the ladle. Proper conditioning of slag forms an integral part of total desulphurisation of hot metal. Left over slag after desulphurisation containing high percentage of sulphur can lead to sulphur reversal during further processing of metal. Also slag which is not easily rakeable can lead to considerable iron loss during skimming operation apart from other adverse effect like extra skimming time, associated with high rate of temp drop. The loss of metallic yield forms an important element in the total cost of desulphurization. In this context, a search was conducted to find a suitable solution and JAMIPOL stepped in to offer the same. The company developed a new compound "JFLUD" for slag conditioning which is made of materials in controlled proportion. Trials were conducted in an integrated steel plant using 180T ladle. Trial was started with 20 kg 'JFLUD' compound and gradually brought down to 10kg to find out the optimum quantity at which maximum benefits can be obtained on all fronts. Results obtained were very encouraging and satisfactory. 10kg JFLUD compound was used finally in place of 10 kg soda ash for about 3 ton of slag in 180 ton ladle having 170-175 ton hot metal. During the trial, important process parameters like raking time, iron loss, temperature drop and sulphur reversal were monitored. It was found that JFLUD performed much better as compared to soda ash. Other trials were conducted in different operating condition with slight modification of present JFLUD Compound, which has given excellent result compared to the previous one. 'JFLUD' can now play a very important role in entire desulphurisation process in iron and steel industry.

Development and commercialization of SAILCOR steel for Indian Railways at Rourkela steel plant

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Indian Railways import considerable quantity of weather resistant steel for manufacturing of BOX-N wagon and coaches. In addition to this, application of this type of steel can be used for the manufacture of marine containers, mine cars and other structural fabrication requiring atmospheric corrosion resistance along with decrease in tare weight. SAILCOR is a low alloy structural steel with superior strength and exceptional atmospheric corrosion resistance. This steel is basically a high phosphorus copper bearing steel to which a small quantity chromium an silicon are added to improve the strength along with corrosion resistance Usually Nickel addition equal to the copper content of the steel is also made to counteract the adverse effect of copper on hot ductility. This new class of weathering steel with miner alloying addition of copper,

chromium, nickel was developed and commercialized at SAIL through concerned efforts of Rourkela Steel Plant (RSP) for alleviating the corrosion of railway wagon body in hostile environment for Indian Railways. This corrosion resistance steel (SAILCOR) have been made through BOF-VAR-LHF-CC route with very low Sulphure (0.005-0.02%) along with high Phosphorous (0.08-0.120 %), which is very difficult to cast due to high Phosphorus. This was mainly developed by optimizing parameters through casting at SMS-I and rolling at HSM along with lamellar cooling facility. Due to adequate heating and proper soaking temperature at Reheating furnace, the mill load was maintained well within the permissible limit. It can be seen that through combination of proper chemistry and optimization of thermo mechanical treatment during rolling for grain size refinement, excellent combination of strength, formability, weldability, corrosion resistance as well as Impact properties such as YS, UTS, %El and bend test value could be achieved to meet the requirement of the Indian Railway wagon specification IRS M41 (SAILCOR). Extensive customer interaction and combine with aggressive marketing strategy, resulted in achieving and maintaining desired quality for Indian Railway. The performance and feedback was found to be very good for development as well as commercialization effort have been well appreciated by the customer.

Recovery of additives in induction furnace steelmaking

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Induction furnaces are being used since long for melting stainless steel scraps, the production of mild steel is a recent phenomenon. Several induction furnaces have been installed along with backward and forward integration with varied proportions of input materials depending upon the availability and location of the plant. The old view that induction furnaces do not 'make' but only 'melt' steel has been proved wrong. Although mostly mild and carbon steel is being produced, many units have started diversified product mix. Ladle refining facility and continuous casting machines have been installed to improve melting rate and productivity and make better quality steel. Qualified metallurgists and experienced engineers are employed by the industries. Successful attempts have been made even for dephosphorising the melt. A very careful control is exercised by the smelter as regards chemistry of melt and quality of products. This has raised the issue of additions in the bath and its recovery. The recovery of additives plays an important role in the steelmaking economics for their cost is significantly higher than the cost of steelmaking feedstock. Necessary understanding of the factors determining the recovery of additives significantly saves the cost while producing consistent quality steel products from heat to heat with highly reproducible mechanical properties. Recovery of additives depends on a vast spectrum of parameter such as types of additives, methods of additions and melting techniques. The optimization of additives involves minimizing their specific cost subject to the condition of satisfying the need of customer. Present paper reviews and records some of the practical aspects of the recovery of additives and final steel chemistry at induction furnace steelmaking.

Automation efforts in merchant mill, DSP to reduce electrical & operational delays for improving productivity

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Several electrical & operational delay reduction measures have been investigated in Merchant Mill of Durgapur Steel Plant and following innovative automation schemes have been implemented successfully during last couple of years such as automation of charging & furnace area control drives, application of digital drives for smooth speed control of shear motors, centralised control of cooling bed & bar shear drives, and process monitoring, cobble detection & analysis system. A centralised PLC and remote I/O based monitoring & control system has been introduced for all major equipment, mechanisms, parameters of furnace & charging areas. Earlier, all the drives in these areas were working on relay logic control which was difficult to maintain and trouble shoot in case of breakdown. Improved closed loop speed control of DC motors of shears featuring adjustable field current and armature voltage has been implemented for better speed control, optimized cutting and faster diagnostics The control system reads analogue inputs of speed & shear blade position feedbacks from encoders and several digital inputs including interlock information to generate reference for drives and "ÇUT" command for shear motors. A comprehensive PLC network based automation system has also been executed in phased manner for cooling bed & bar shear drives without taking any additional shutdown especially for it. In this system, any change in control philosophy can be easily achieved through soft logic in place of complex hard wired relay based control circuits used earlier. Modified operator desks at bar shears and Control Pulpit-10 have been connected through cold redundant distributed communication network eliminating multi-core cables. A P-T-Z dome type CCD camera based system has been introduced for on-line monitoring & video recording of the rolling process, specifically at repeater & cooling bed areas. This has enabled early detection of cobble, post operation diagnostics /analysis of cobbles, acceleration of subsequent mill adjustment process. The above innovations have resulted in i) reduction in annual electrical & operational delay of the mill by 50 % and 10% respectively and ii) increase in yield by 0.5%.

Improvement in MgO-C bricks making at SRU Bhilai for production of secondary heats in steel ladle at SMS-II, BSP

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Magnesia carbon (MgO-C) bricks are shaped refractory products made from dead burnt or electro fused magnesia and graphite as carbon source. The bonding system is based on resin (phenol formaldehyde) and various pitches (coal tar and petroleum) including addition of metallics and the products are tempered at 200-300°C. Application under high temperature, the MgO-C bricks form carbon skeleton bonding which are very strong in nature. MgO and carbon do not have any mutual solid solution and hence the superior refractory properties of both the components are retained in the bricks. Moreover, carbon is not wetted by slag and as such good resistance to wear by slag. In SAIL Refractory Unit, (SRU), Bhilai, MgO-C bricks for steel ladles are being made since long years. Over this period various experiments have been carried out for improvement in quality of MgO-C bricks. This includes proper selection and use of natural sintered/dead burnt magnesia with higher B.D (3.40 gms/cc) and crystal size (+100 micron) along with proper CaO/SiO_2 ratio with less SiO_2 to obtain high temperature strength as some of the CaO enters into the solid solution in the MgO phase in order to achieve a refractory dicalcium silicate phase which has high melting point at the periclase crystalline boundaries. Further experiments carried out using 96% flaky graphite and suitable resin having high Fixed carbon(FC) and appropriate non- volatile matter (NVM) and viscosity along with appropriate combination of antioxidant and controlling granulometry yielded reduction in coke porosity(8-10%) and apparent porosity(3-4%) and improvement in cold crushing strength (CCS) (450-600 kg/cm^2) and coked strength (245-350 kg/cm^2). MgO-C brick composition with different carbon content has been formulated to use as zonal lining in slag zone, metal zone and bottom in secondary steel ladle (BF-LF-VAD/RH route) in SMS-II of Bhilai Steel plant. An average lining life of 64.3

heats with maximum life of 78 heats could be achieved during 2013-14 incorporating the said modifications. In this paper a detailed description of work done for improvement in bricks quality vis-à-vis its performance improvement in steel ladle at BSP has been outlined.

Multiphase modelling of bottom stirred ladle for prediction of slag-steel interface, desulphurization and nitrogen pickup behaviour

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Ladle furnace is a key process in which various phenomenon such as de-oxidation, desulphurisation, inclusion removal and homogenization of alloying compositions and temperature takes place. Therefore, processes involved in the ladle play an important role in the quality of steel for the further downstream processes. Prediction of flow behaviour of the phases present in the ladle furnace is required to understand phenomena which take place in the ladle and accordingly control the process parameters. In this study, a mathematical model to analyse transient three-phase flow is developed. Ladle used in this study is argon gas bottom stirred with two off-cantered plugs. Volume of Fluid (VOF) method is used to capture the slag, steel and argon interfaces behaviour. Numerical simulation with different flow rates is performed and results are validated with the literature. Calculation of mixing time is carried out by solving a scalar transport equation in a steady state flow profile to get the time for homogenization of molten steel. Further, slag eye opening, slag steel interfacial area has been calculated for different operating conditions. Desulphurization and nitrogen pickup rate is then predicted using chemical kinetic equations to analyse the behaviour of both the chemical processes happening in the ladle.

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Influence of design and process parameters on re-oxidation of molten steel in tundish using CFD analysis

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The chances of re-oxidation of steel during steelmaking are high because of interaction of atmospheric air, especially in tundish. The re-oxidation leads to steel impurity and uncleanliness and ultimately affects the steel quality and performance. As there is very limited scope of oxygen removal during tundish processing and subsequent operations, the control of re-oxidation in tundish is critical and should be minimized. The phenomena of re-oxidation is directly linked with the disturbance of slag layer where the flow of melt causes turbulence and wave formation and enhances the possibility of oxygen pick up by exposing molten steel to atmosphere. In order to determine the amount of re-oxidation occurring in tundish, a multiphase CFD model involving metal, slag and air is proposed. Free surface model equations are incorporated in the model to study the interactions of molten steel with air and slag. In order to obtain a quantitative measure of re-oxidation, the mixture volumes of slag-molten steel, air-slag and air-molten steel are calculated as a function of various design and process parameters. It is shown that both process and design parameters have significant influence on re-oxidation. Finally, the model is tested with limited data available in literature as well as with plant data.

Optimisation of inclusion floatation in tundish to produce cleaner steel

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Modern casters are expected to produce quality products with least possible defects or inclusions. For this, size and amount of internal inclusions in cast slabs should be strictly controlled. Being lighter than molten steel, inclusions present in steel is removed when it floats to top slag and is captured by it. Argon diffuser is a flow control device used in tundish, which aids in removal of inclusions to top slag. The effect of argon diffusers' size, its location in tundish and argon flow rate through it in tundish of a twin strand slab caster of Bokaro Steel Plant, SAIL has been analysed through mathematical simulation of inclusion floatation, argon bubbling and fluid flow using ANSYS FLUENT® software. The simulation of fluid flow, argon bubbling and inclusion floatation has been performed for four different locations of argon diffuser, five different argon flow rates and ten different inclusion sizes. Fluid flow simulations have indicated that the flow in tundish is inherently transient with flow fluctuating with a time period of 240 seconds. Residence Time Distribution (RTD) analysis suggests that optimum locations for argon diffuser is at locations between 2100 mm and 2400 mm from the center of tundish. Optimum flow rate for argon bubbling in TAD is between 13 to 17 lpm. Analysis of inclusion floatation suggests that for particle sizes of 100 microns and higher, argon bubbling is not required. The inclusions float up easily due to their natural buoyancy. However, for finer particles, lesser than 100 microns, higher argon flow rates helps in improving their floatation. Finer particles have higher efficiency when the argon flow rate is increased. Analysis of total oxygen content in plant trials have shown 10-20 % reduction in total oxygen content in samples taken from slab when the argon diffuser is operated at optimized location and flow rate.

Physical modelling of slag foaming in presence of liquid metal phase

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In steelmaking operations, foamed slags plays an important role in energy efficiency, heat transfer and process kinetics. A cold model simulation of slag foaming phenomenon in steel making operation was conducted to understand the effect of viscosity, density and interfacial energy between steel and slag on foaming. The experimental study was conducted at room temperature using water and oils of different viscosities which represent steel and slag respectively. Experiments on individual bubble motion across water-oil interface showed that the residence time of bubble at the interface increased with increase in interfacial tension. It also showed the foam index was influenced by the carryover of water across the interface by gas bubbles which in turn depend on the velocity of gas, density and viscosity of the mediums. Foaming experiments with oil phase only showed that the foaming height increased with increase in viscosity and decreased with further increase in viscosity by varying the gas velocity. Foam index in the oil

phase experiments decreased with increase in gas velocity. But in water-oil system, the trend was entirely different from only oil phase experiment showing that the foam height and foam index not only depends on viscosity, density difference between the phases but also on the amount liquid transfer that takes place across the interface which affects the residence time of the bubbles.

Effect of particle size and loading on stability of a coalesced particle laden bubble with an un-laden bubble during rise in a liquid column

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Stability of a particle laden bubble during rise plays a major role in separation of inclusions in the flotation zone of a tundish in steel making process. Experiments are performed in a cold model in order to study the effect of particle loading on coalescence and rise of a particle laden bubble. The effect of particle size is also seen for the same process. Hydrophobic Low Density Polyethylene (LDP) particles are chosen for study based on the similarity of three phase contact angle between alumina-argon-steel system and the model LDP-air-water system. Based on this study the optimum particle size range and loading concentration for a given pair of coalescing bubble size is identified.

Recycling of LD Slag

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Recycling of LD slag having 45-50% lime is still a challenging task because of its high phosphorus (P_2O_5) content (>3.0%) which makes it unfit to be used as a flux in iron and steelmaking processes. It is possible to use the slag as building material but due to the presence of free lime and high iron content (>20%), its use is restricted.This paper is based on the development of a process at laboratory scale to remove phosphorus from LD slag to make it recyclable as flux and also to explore the possibility of using the products generated from LD slag for other applications. Amongst the number of reductants available, experiments were carried out only with carbonaceous materials to treat the liquid slag at about 1600^0C in EAF and generated a product which contains less than 0.5% P_2O_5, more than 65% lime and less than 2% iron oxide. It was observed that the treated slag fraction of the solidified mixture progressively crumbles into a fine powder (<100 mesh) which separates from the metal effortlessly and that it is possible to utilise in the iron and steelmaking process as a partial replacement of flux. The generated powder has been already tested as flux in sintermaking and as binder and lime source in manufacturing of briquettes for use in BOF. Further, XRD analysis shows presence of tricalcium silicate as major phase which makes the product a potential agent for cement making. Effort is on to generate the product from LD slag at higher scale to study the feasibilty of its recyclability for various applications.

Study on reducing behavior of mill scale solid waste generated in steel industry and its characterization

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One of the wastes generated in steel plant is the mill scale, formed during the continuous casting and hot rolling process. These are roughly about 2% of the steel produced and are rich in iron content about 72% of Fe. These by products are recycled to produce iron rich charge materials by reducing with different reductants (solid reductants, gas reductants) at various temperatures. The reduction kinetics was determined. The percentage of reduction and Degree of metallization was calculated. The mill scale and final reduced product was characterized by Chemical analysis, X-ray diffraction, and scanning electron microscopy.

Selection of suitable secondary refining unit (SRU) in steelmaking for desired quality of steel with improved performance and productivity

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In modern steelmaking, primary units viz. BOF & EAF have been pushed to very low tap-to-tap time cycle for tonnage production and the role of quality control in terms of cleanliness & chemical composition in closest of tolerances for improving performance, lowering cycle time for improving productivity and meeting stringent specification of critical grades have been left for secondary steelmaking. Secondary steelmaking takes its origin since 1950s but till the 80's it was limited to special alloy grades like stainless steel. Due to tremendous growth in steel consumption and production in last two decades, secondary steelmaking has attained a pivotal role in improving productivity by acting as buffer between caster and primary steelmaking unit. The use of ladle furnace (LF), one of the most common secondary refining units (SRU), has become necessity due to sequence casting requirement of modern continuous casters. In addition to challenge of producing more, today the steelmakers are also poised with the challenge of meeting stringent specification arising from increasingly new product applications, regulations for environment, regulations for public safety both for applications in transport and packaging industry, and improving performance in applications like automotive, shipbuilding, aviation, defense, consumer durables, construction, etc. Tapping the potential of SRU's is the only viable economical solution to meet the above challenges for existing steel plants as well as upcoming steel projects. In this paper, various secondary refining technologies like LF, VD, VOD, VD-OB, AOD, RH, RH-OB, CAS-OB, etc. have been critically analysed for its limitations and advantages against some critical application steel grades like line pipe steel, AHSS family steel grades, silicon steel, advanced stainless steel grades, etc. in order to develop selection criteria for suitable secondary refining units(s) in a steelmaking shop of a steel plant for production of desired quality of steel with improved performance and productivity.

Designing laboratory facilities for quality assurance and quality control in integrated steel plants

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In an integrated steel plant finished products like HR coils, CR coils, structural products, TMT bars, rails, rail-wheels, etc. are produced from starting external input raw materials like iron ore, coal, coke, etc. The finished products have to conform to various national and international standards based on its subsequent applications. Increasingly, the performance indices of the products in every application areas are getting stringent. This poses challenge of quality assurance of the existing products and meeting the emerging demand of new products in the market driven economy. In order to meet the above challenge and keep competitive edge, quality assurance measures are to be enforced at every stage of production which can be properly carried out through a number of well-equipped quality assurance and quality control laboratories with state-of-the-art modern equipments. The laboratories in an integrated steel plant are designed - to collect and prepare samples, analysis and testing of raw materials, analysis of intermediate and testing of finished products, feedback to main production units to enable it to ensure corrective measures in a short time to eliminate process irregularities and rejection of costly intermediate/ finished products, development of new grades, and investigation of customer complaints for addressing it effectively. The new trends in designing laboratory include pneumatic sample transportation, container laboratory, online testing of finished products, automation, etc. An attempt has been made through this paper to briefly illustrate the steps of envisaging quality control facilities and underlying considerations based on requirement of each processing step while designing laboratories in an integrated steel plant.

Nitrogen control in steel making process

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India is the fourth largest producer of steel in the world but in quality of steel it is still lagging behind. If India has to become the best quality steel maker as well, it will have to make modifications in the steel making processes aimed at reducing the impurities which are detrimental to the properties of steel. One such impurity is nitrogen which increases the tensile strength but decreases ductility and adversely affects the formability of ultra low-carbon cold rolled steel. In BOF, a reblow causes around 10 ppm nitrogen pickup. Further pickup is caused during open stream tapping of steel. We found the average Nitrogen at LHF as 30 ppm and the average nitrogen at loading position at caster as 34 ppm .Thus, if we reduce the distance between LHF and caster then nitrogen pickup can be reduced.We also found that damaged gasket, sticky foreign material on the outer surface of the collector nozzle, rough inner surface of shroud, non-vertical shroud, metal level variation in tundish and mould level fluctuation caused a drastic increase in nitrogen pick up during casting.

Effect of calcium carbonate on mill scale reduction

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Mill scale is a black scale of magnetic oxide of iron, formed on iron and steel when heated for rolling, forging, or other processing. It consist iron, both in its elemental and combined form as wustite (FeO), hematite (α-Fe2O3) and magnetite (Fe3O4) and is usually less than 2mm thick. Mill scale is a nuisance when the steel is to be processed. It causes environmental pollution and is thus landfilled. The present study was aimed to recover iron from mill scale through direct reduction with low grade coal (from Nagaur district, Rajasthan) in presence of lime at 900°C temperature to produce sponge iron. In this study, percentage of calcium carbonate (CaCO3) was varied, to study its effect on reduction behavior of mill scale. Detailed characterizations of reduced samples were studied by SEM (Scanning Electron Microscope) and XRD (X-Ray Diffraction).

Improvement in recovery of “B” alloy addition in 'Boron' alloyed DD/ EDD quality steel by Fe-B wire injection at JSW Steel Limited, Dolvi

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DD/EDD Quality Hot Rolled coil steel grade conforming to IS 11513 Grade EDD is used for cold rolling and galvanizing application. As per customer feedback, JSW Dolvi made steel is comparatively harder as compared to our competitors. This is due to much finer grain size in the final HRC which is characteristics of thin slab caster. Due to this high hardness of the material, cold rolling customers were facing problem like high load and low speed, less final % cold reduction, black band formation during cold rolling etc. To overcome this problem, Boron was added to normal DD/EDD grade steel and was found to be beneficial in reducing hardness as well as strain hardening rate (during cold rolling) which in turn improve the cold roll ability. Initially was used as Boron lump for making B bearing steel. However, due to high oxidizing nature of boron, the recovery of Boron from lump was very less (40- 50%) as well as inconsistent. Also due to inconsistency in Boron recovery and formation of BN, surface defects like edge burr/ edge cut was taking place very frequently. Boron in traces found in the next heat of non Boron heat leading to non prime generation. Boron cored wire was tried as we have provision of wire injection system in ladle furnace. The cored wire will be released close to the ladle bottom far away from oxidation sources (slag, air). This leads to a better recovery compared to traditional lumps addition especially for elements which are highly oxidized in nature. Also Lifting % Al was maintained higher side to avoid any BN formation. With this Boron wire addition, the recovery is now higher (75-80%) and more consistent. Also due to consistency in Boron% and higher Aluminum, the surface defects have reduced drastically.

Development of ultra clean steel for automotive applications

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Automotive industry has high demand for steels with improved service life that reduce warranty claims and higher range of mechanical properties that enables component weight reduction and reduced carbon foot print. Service life of dynamic automotive components can be improved by manufacturing ultra-clean steel. Kalyani Steels Limited has developed steel making and hot rolling technology to manufacture Ultraclean steel with Oxygen of ~10 ppm and inclusion SAM D rating ~ 6 for low alloy steels used in power train transmission gear applications. The steels made have been supplied to manufacture critical power train components such as shafts, axle beam, steering knuckles, gears etc. for use in the domestic and export oriented international market. The inclusion size and distribution in some of the steels have been characterized and the rotating bending fatigue tests have been evaluated. This presentation deals with the quality aspects of the steels such as, inclusion in the Ultra clean steel, type and morphology of residual inclusions, microstructure and fatigue performance of some of the steel heats made.

Analysis of sulphide inclusion formation in calcium treated grades

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The customer demand for improved steel quality had led to development and modification in the processing of steel. Calcium treatment was initially envisaged for prevention of the Sub Entry Nozzle (SEN) clogging in aluminum killed steels but now days extensively done for modifying inclusion morphology to minimize the harmful effect on formability and toughness. Calcium addition to the steel transforms alumina inclusions into calcium aluminates and simultaneously reacts with sulphur to form calcium sulphide (CaS) as calcium is powerful deoxidizer and desulphrizer. The presence of sulphide inclusions were studied in this study with emphasis on product quality (inclusion rating<1.5) in the Ca treated aluminum killed steel. The cast heats were categorized into two groups depending on inclusion rating as OK heat (inclusion rating < 1.5) and NOT OK heats (inclusion rating >1.5). The effectiveness of Ca treatment is influenced by number of parameters. The parameters studied in this work are killing time, LHF in sulphur, sulphur level at time of Ca injection to ladle, rate of desulphurization at LHF and Ca/S ratio. The minimum killing time was found to be 12 minutes which varies with the other parameters. The initial sulphur level has great impact on the inclusion rating. The threshold sulphur level causing the difference in the inclusion rating was compared in both the categories (OK and NOT OK heats). The sulphur at the time of calcium injection was found to be higher in NOT OK heats (> 0.009 % S) compared to OK heats (< 0.007 % S). The desulphurization rate was higher in NOT OK heats. The study suggest to limit maximum cap on LHF in S at 0.007 % S, sulphur at Ca injection 0.006 and good Ca/S ratio > 0.6.

Evolution of non-metallic inclusions during ladle treatment

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In the steel production process, secondary steelmaking is a critical step between the primary processes (Basic Oxygen Furnace or Electric Arc Furnace) and casting. To achieve the desired composition of steel as per rated specifications, it is often necessary to facilitate alloying additions and/or remove certain tramp elements. The temperature, internal quality and the inclusion content of the steel also have to be carefully monitored during secondary steelmaking. The slag chemistry plays a vital role in the removal of inclusions from the melt, thereby making it necessary to study the interaction between slag and melt. The formation of inclusions and their types were analyzed using Thermo-Calc software. IRSID database (Slag3) and TCFE7 were used to study the interaction between slag and steel. The evolution of inclusions during ladle refining, holding and teeming was studied in a transient manner to study the inclusion number density, distribution and composition before initiation of solidification. The main focus of this study is to propose a model that can predict the type of inclusions and their distributions during the refining of steel.

Challenges in physical modelling of fluid-flow of tundish

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Physical modelling the fluid-flow of molten steel inside a tundish during the continuous casting process demands solution for a huge range of problems. Since the modelling process considers experimental data superior to mathematical model, it is very difficult to say if the physical model is setup properly. All previously published works cannot be trusted completely since minute details of the experimental setup are often kept confidential owing to the commercial benefits associated with the work. Therefore scientists rely upon their intuitions and experience rather than any tried/tested method. Thus every challenge faced during physical modelling of fluid-flow of tundish needs to be discussed publicly.

Fluid bed technology to the aid of secondary steel producers

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The secondary steel sector in India has always been plagued with the desired raw material input problems. The scrap supply market being volatile, prices unstable and quality unpredictable, taking recourse to DRI/HBI addition as supplements has been the only path to partly overcome the issues. Recent relief through molten metal addition is not for everybody. Thus any development towards bypassing the age-old scrap route on way to obtaining good steel at low cost should be welcome. In that context, the recent efforts on the part of iron carbide pioneers need attention, in view of the injectable nature of this fine material (produced through fluidised bed technology using iron ore fines and natural gas) into EAF that makes it dissolve in molten bath in seconds. The bath turbulence due to tiny carbon monoxide bubbles generated helps drive out dissolved gases and creates foamy slag, so desirable for the EAF operator. Besides, the tramp element problem associated with scrap is absent here and so is the DRI reoxidation issues, thus making it environmentally safe for use and stable for shipping. Above all, the bonus carbon of about 6-7 % with the iron units in carbide, aids admirably in reduction of iron oxides and generating energy. In view of its exciting possibilities, two specific features are being analysed at NIT, one being the fluid bed cold model studies with Indian Hematitic fines that would be projected here. The other is carbon injection modelling parameters, as it hits the molten metal bath. And the third part is developing a collaborative project with industries and Govt. bodies to suitably establish a route for tomorrow's steelmakers, even with fuel sources other than natural gas.

PROCESS METALLURGY OF NON-FERROUS METALS

Invited talks

Session 1: Nov 12, 2014: [16.00 - 18.00]

Energy saving potential in Indian aluminium industry

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The debate about the future of the industry often concentrates on the high energy usage associated with aluminium production. Of course, aluminium is over three times lighter than steel, which means that energy savings can be made over the lifetime of the metal's use if aluminium replaces steel in transport. Also, the overall energy picture looks much healthier if your energy source is hydroelectricity. Existing primary aluminium production processes are energy intensive by nature. The main source of energy consumption during production is the electricity used for the electrolysis process. Also, during the refining of alumina from bauxite ore, a significant amount of energy is required to produce the solution of bauxite in caustic soda, for the calcination process and for the recovery of caustic soda after use. As energy costs are a major part of overall production costs, improved energy efficiency is essential for the aluminium industry, both from an economic and environmental point of view. Improved energy efficiency will also reduce indirect emissions from production of the electricity used in the electrolysis process.

Purification processes ofTe, Cd and Ga to 7N purity and above: An overview

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The purity of Ultra High Pure (UHP) materials needed for producing semiconductor devices depends on the desired performance of the respective devices. Purification processes are also known to be impurities specific, as a variety of impurities can be easily tolerated than the others. The bulk crystal and epitaxial growth processes for cadmium-mercury-telluride (CMT) use greater than 7N (99.99999 at.%) pure elements of Tellurium (Te) and Cadmium (Cd), as the starting materials. One of the major requirements in the research and development of production of Infrared (IR) optoelectronic devices is the preparation of a device grade substrates, which should be of very high purity, especially, the deep level defects acting as recombination centres, should be eliminated to below the acceptable limits. For these reasons, an in-house ultrapure elemental facility or materials purified from a known indigenous source is essential. At C-MET, the clean room facility and the state-of the art process equipment required for tellurium and cadmium purification are designed and fabricated using locally available components, which essentially circumvent the use of imported and costly equipment. The indigenous process technologies developed have yielded in reproducible and repeatable batch scale processing. Output materials quality is assessed by analyzing parts per billion (ppb) level impurities from Li to U using Glow Discharge Mass Spectrometry (GDMS). Thus, purified tellurium and cadmium processed at C-MET, Hyderabad laboratory was used by the

end user at Solid State Physics Laboratory (SSPL), New Delhi, resulted in excellent device properties on par or better than the imported input materials. Present status of Ultra High Purity (UHP) semiconductor materials in general and tellurium and cadmium in particular would be presented. The Processing technology along with results obtained would be presented in detail. The present status of gallium purification in the country will also be covered.

Contributory papers

Session 1: Nov 12, 2014: [16.00 - 18.00]

Preparation of magnesium-aluminum alloy directly from their chloride salts

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Magnesium-aluminum alloys are used in critical applications in automobile, defence, aeronautical and aerospace industries. Conventionally, the alloy is prepared by melting and mixing of pure magnesium and aluminum metals, which has a very high metal loss. The objective of the present investigation is to develop a suitable process for preparation of magnesium-aluminum alloy directly from its chloride salts through molten salt electrolytic co-deposition in a single step. The molten salt electrolysis was carried out in a suitable electrolytic cell which was designed and fabricated locally. The process parameters like bath composition, temperature, current densities and applied potential were standardized and the cell was operated with electrolytes comprising of LiCl (50 %) - NaCl (30%) - $MgCl_2$ (10%) - $AlCl_3$ (10%) {wt.%} at least for 5 hours at 680°C with applied voltage between 3V to 5V. At the end of the experiment, the metallic particles of diameter ranging between 1 mm to 4 mm were collected and stored. The bulk chemical analysis of the particles by Atomic Absorption Spectrometer (AAS) shows that the particles are composed of 29-64 wt% magnesium and 26-63 wt% aluminum. The SEM-EDS result reveals that the particles mostly consist of 55-65 wt% magnesium and 23-42 wt% aluminum. The XRD studies confirm that the alloy is composed of predominantly AlMg, $AlMg_2$, Mg_2Al_3 and $Mg_{17}Al_{12}$ phases. The current efficiency of the process was estimated to be 57%.

Plume characterization using hydrodynamic experiments & CFD study

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The top-blow injection technique of gas-solid mixture through a circular lance is used in the Mitsubishi Continuous Smelting Process. One of the inherent problems associated with this injection is the increased consumption of lances and severe erosion of the hearth refractory below the lances. In order to minimize hearth wear and understand lance consumption phenomena, it is important to know the behaviour of a plume formed as a result of gas-particle mixture jet in the molten bath in smelting furnace. It is not

possible to characterize the plume as the direct measurement can hardly be made in an industrial plant. Therefore to understand and characterize the plume in detail a Computational Fluid Dynamcis (CFD) model and a 1:10 scaled down water model of smelting furnace have been developed.The plume depth has been measured using high speed camera and characterised for various lance heights, air flow rates, particle flow rates and lance diameters. The contribution of air and particle injection in plume formation has been calculated. This information has been used to validate the CFD results. Using CFD model of smelting furnace, an equation which is able to estimate the plume depth in molten bath as a function of lance height, particle flow rate, molten bath viscosity, density etc. has been derived on the basis of the results obtained.

Sublimation reactions in Kroll's magnesio-thermic reduction process for nuclear grade zirconium sponge production process

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In India, Kroll's magnesio- thermic reduction process is employed for the production of zirconium sponge. In this process, sublimed $ZrCl_4$ vapors are allowed to react with molten magnesium to produce zirconium as solid particles. The zirconium solid particles settle down as reduced mass along with $MgCl_2$ and unreacted Mg. The removal of trapped Mg and $MgCl_2$ is carried out in pyro-vacuum distillation operation to get nuclear grade zirconium sponge. ZrO_2 powder is mixed thoroughly with calcined petroleum coke (CPC) as a reducing agent (to lowers the partial pressure of oxygen) in the ration of 5:1 along with starch solution as binder followed converted into the cylindrical briquette. These briquettes are chlorinated to form $ZrCl_4$ at sufficiently high temperature (950-1000^{0}C) in oxide chlorinator reactor. The decrease in the bed temperature (approx. 600-700^0) favors for the formation of lower valence chloride ($ZrCl_2$ – black powder) and accumulation of carbon deposits, due to the formation of CO_2 gas instead of CO gas, which results in high conductivity of the bed. The reaction time on some occasions in kroll's reactor exceed 60 hours due to the presence of lower valent Zirconium halide ($ZrCl_2$) which clogs the baffle plate holes through which $ZrCl_4$ vapors enter the molten Mg kept in the crucible. The generation of $ZrCl_2$ was reduced in the chlorinator by the changing the mixing ratio of ZrO_2 to CPC (10:1) instead of normal ratio of 5:1. This change in ratio helps increasing the resistance of the bed and higher bed temperature at 950-1000^{0}C, which favors formation of ZrCl4. In this way, clogging of baffle plate due to $ZrCl_2$ is avoided. The energy required for the sublimation of 2800 kg $ZrCl_4$ is 12, 41,373 kJ. The optimized average rate of sublimation of $ZrCl_4$ is approx. 214-223 moles per hour.

Mineralogy based process advisor for copper flash smelting furnace

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The flash smelting process, developed by OUTOTEC (Formerly Outokumpu), is being widely used in copper smelters around the world, which accounts for 50% of global copper production. Flash Smelting Furnace

with Outotec Technology has been in operation at Hindalco Industries Ltd, Unit: Birla Copper, Dahej, Gujarat with an installed capacity of 150,000 TPA primary copper. Typical, Flash Smelting Furnace (FSF) consists of the Reaction Shaft (RS), the Settler and the Uptake Shaft (US). Copper concentrate along with silica flux is fed through Central Jet Distributor installed at the top of RS. Flash smelting of the copper concentrate takes place in RS in presence of oxygen enriched air. Provision of fuel fired burners is there to maintain the heat balance of the furnace; generally the burners are not required to be operated during the smelting process as the reactions are autogenous. The phenomena of smelting essentially consist of dispersing and oxidizing the dry concentrate mixture in oxygen enriched air stream. As the gas-solid suspension leaves the central jet distributor and flows downwards through the reaction shaft, the solid particles are heated by convection and radiation heat transfer. When the particles temperature reach the flash point of 400 – 500 °C, the smelting reaction initiates. As a result of smelting reaction, matte (copper sulphide and iron sulphide mixture), fayalite slag (iron oxide and silica mixture) and sulphur dioxide gas is formed. The fine un-reacted particles, called as dust, are carried away by the gaseous stream to waste heat boiler and electrostatic precipitator from where the dust is recovered and recycled in the flash smelting furnace. The copper bearing product matte (58-60% Cu) is further processed to enrich up to 99.5% purity level while the slag is discarded after recovering copper from it. Sulphur dioxide gas is further processed for making sulphuric acid. The plant receives concentrate from different sources and hence the grade and quality vary. As a process requirement, these different concentrates are required to be blended so as to achieve desired feed grade with low impurity level and at the same time ensuring the proper heat balance of the furnace. Thus maintaining thermal balance of the furnace & achieving the desired productivity with varying blends is a major challenge for this process technology. Mineralogy based Process Advisor was developed to predict optimized operating conditions for feed blend changes. This acts as a guidance system, for operators, with an estimate of the RS temperatures, product composition, fuel requirements, etc for the given feed blend. This Mineralogy based Process Advisor comprised of two components (i) Matlab based Mineralogical model: to predict mineralogy from elemental analysis, and (ii) METSIM Process model: to predict FSF operating parameters using the mineralogy. Mineralogical database has been created for more than 20 different mines based on the X-Ray Diffraction analysis of concentrate samples used at the plant. Mineralogical model predicts mineralogy based on elemental analysis of each concentrate in the blend. METSIM platform then imports the blend mineralogy from Mineralogical models output and computes heat & mass balance using sequential modular approach. The model predictions were validated with plant data for several blends. This off-line model helps the operators to predict the performance of the blends and helps to set & tune the operating parameters for smooth & efficient FSF operations.

Lance consumption mechanism in Mitsubishi smelting furnace

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The Mitsubishi technology of continuous copper making works on bath smelting principle. This continuous process has smelting, slag cleaning and converting furnaces connected in series. These three furnaces are connected to each other by covered launders, through which the molten materials (matte, slag & blister) are continuously transferred by gravity. Vertical lances are used in smelting & converting furnaces to feed oxygen-enriched air & charge materials such as copper concentrate, flux in smelting (S) furnace & flux in converting (C) furnace into the molten bath. The high chrome stainless steel lance gets consumed during the course of operation. The lance height is maintained 0.7 m from molten bath by frequently welding new lances from the top of the furnace. The typical lance (4” dia) consumption is 0.4-0.7 m/day in S- Furnace

and 1-1.3 m consumption/ day in C-furnace. The lance consumption is major issue due to changes in concentrate grades and operating requirements. The higher lance consumption changes in lance height from standard set point which impacts process efficiency, dust load and also disturbs the furnace operations. These lances are high chromium steel (26%) pipes as per the design specifications of Mitsubishi Technology. The concentrate particles carried by high velocity air erode the inner wall of the lance whereas the outer wall of the lance is exposed to high temperature gas stream containing SO_2 and gaseous phase of impurities like As, Zn, Pb, etc. The splashing of liquid bath due to interaction concentrate -gas stream from lance with liquid bath, also forms coating of sulphide products on the lance outer surface. The lance experiences corrosive atmosphere & thermal profile during descent inside the furnace which causes sensitization and hot corrosion. Therefore study was carried out to understand the mechanism of lance consumption. For this the samples of the used lance were collected from the plant. The detailed micro-structural study was carried out of samples from different sections of the lines to understand the phase changes in lance materials and corrosion reaction mechanism. The results of study revealed exposure to high temperature 1240 ^{0}C causes sensitization, i.e, segregation/precipitation chromium carbide to grain boundaries. The sensitization reduces the corrosion resistance properties of the lance steel. The Cu and Fe sulfides present on the surface attack the sensitized Cr-lean phases/grain boundaries, thus causing lance to get corroded.The copper-phases present in the coating forms the low melting phases like Fe-Cr-Sulphides & Fe-Cu-oxides. This accelerates the melting of lance tip below furnace operating temperatures, 1240 ^{o}C and thereby increases the lance consumption.

Optimization of leaching parameters for cobalt in Sukinda laterites using Taguchi method

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The sulfide ores have been the major source of nickel to date, which are getting exhausted. Therefore lateritic ores, which constitute about 85% of the known nickel reserves worldwide, are the future source of nickel. India have two primary nickel resources - manganese ocean nodules (1.1% Nickel, 0.1% Cobalt) and the lateritic nickel over burden (0.5 - 1% Nickel, 0.012% Cobalt) of Sukinda chromites in Odisha State. In spite of prolonged efforts over last four decades, no viable technology for recovering nickel from lateritic chromite overburden is available. The authors have attempted to study afresh the dissolution of nickel and cobalt from this ore in detail by using a combination of sulfur dioxide and sulfuric acid. Sulfur dioxide was generated by reacting sodium meta-bisulfite with concentrated sulfuric acid. The gas was injected inside slurry through gas bubbler. In this investigation, the influence of the various parameters affecting the process of leaching cobalt from Sukinda laterites has been analyzed and the process parameters were optimized to maximize leachability. The parameters considered are the acid consumption, SO2 consumption, leaching time and effect of enhancer. The effects of these input parameters on the response (% leaching) have been critically analyzed using Taguchi method. It has been found that the acid consumption is the most important process parameter affecting the percentage leaching of cobalt. Effect of acid consumption was found to be the maximum with 37% share followed by time with 32% share and SO2 consumption with 23% whereas minimum effect was observed for the use of enhancer with 8% share of the total. Maximum percentage of cobalt leaching of about 65% was achieved, when the process parameters - acid consumption, SO2 consumption, leaching time and enhancer were set as 84 kg/ton, 8.8 liter, 2 hrs and naphthenic acid respectively.

Development of Al-Si/SiC-Al_4C_3 master alloy for the grain refinement of Mg-Al alloys

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For hcp materials like magnesium alloys, fine grain size can effectively improve its formability and mechanical properties. Among the various techniques developed for grain refinement of Mg-Al alloys, carbon inoculation method by the formation of Al_4C_3 particles is the only method that works convincingly. However, it has difficulties in carbon inoculants addition and the impurity elements interaction with grain refiners. Development of a generic refiner is still under investigation for Mg-Al alloys. Hence the present work is focused to develop an Al-Si/SiC-Al_4C_3 master alloy for the grain refinement of Mg-Al alloys. Pure Al-SiC and Al-5%Si-SiC composites has been fabricated with 10 wt.% SiC particles (average particle size of 14μm) as reinforcement. To improve the wettability of the SiC particles with Al matrix, considerable amount of Mg has been added (1 and 5wt.%). The stability region of SiC and Al_4C_3 is thermodynamically estimated using Wilson equation with extended Miedema model and the composites were superheated to form Al-Si/SiC-Al_4C_3 master alloy. DTA and XRD analysis has been used to quantify the extent of reaction products in the master alloy. Combining DTA technique and CALPHAD approach the amount of Al_4C_3 formed has been computed based on Si release in the master alloy. Reaction studies indicate that after holding the composites for 3 hours at 900°C, only 50% to 60% of the reaction has occurred in Pure Al and Al-5Si matrix composites. The master alloys are examined for the grain refinement in Mg-3Al alloy. Addition of 1% of Al-5Mg/Al_4C_3 master alloy has significant refinement in the grain size of Mg-3Al alloy. The presence of Al_4C_3 nucleant particles in the master alloy is found to be responsible for grain refinement. The crystallographic features and the size distribution of nucleant particles extracted from the master alloys are examined for the mechanism of grain refinement.

Calorimetric measurement of In-Sn-Bi system

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Measurement of thermodynamic properties as partial and integral mixing enthalpy of liquid In-Sn-Bi alloy have been carried out at 730 K by drop calorimeter along the cross-section X_{Sn}/X_{Bi}=2, X_{Sn}/X_{Bi}=1, X_{Sn}/X_{Bi}=0.5 by varying the composition of indium from 0.1 to 0.9. Thermodynamic properties of boundary binaries of the ternary system In-Sn-Bi have also been measured using the same technique at 730K. Integral mixing enthalpies along different cross sections have been plotted against indium composition and found exothermic in nature in all the cases. Iso-enthalpy curves for integral enthalpy of mixing are also plotted for four mixing enthalpy values of -1200, -800, -400 and -200 Joules/mole. It has been observed that the negative values of integral mixing enthalpy are predominant inside the ternary triangle. It could be inferred that there is not a strong interactions among the indium, tin and bismuth atoms in the solutions because the integral enthalpy values are not so highly negative. The maximum negative value of integral mixing enthalpy has been found to be - 1.2 KJ/mole. It implies that In-Bi binary system has more influence on ternary system as compared to In - Sn and Bi –Sn and Sn has the less influence on In-Bi-Sn ternary system as compared to In and Bi.

Leaching of metal values from copper slag by organic acids

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Solid waste from copper industry causes environmental pollution as well is a major loss of valuable resource of metals that can be recovered. In the present study, the feasibility of base metals recovery was investigated using organic acids on copper slag: converter and granulated, which basically differ in their mode of solidification. Citric acid and oxalic acid were the organic acids used for leaching the slags and a comparative result was obtained. Parameters such as acid concentration, pulp density, leaching temperature and time were optimised to determine best conditions to recover metals from the slag. In case of converter slag, the maximum recovery of 99.04% Cu, 94% Co, 89.16% Ni and 99.2% Fe were obtained by leaching with 2N citric acid at 35°C with 15% pulp density (w/v) in 15 h using finest particles (<45 µm). With granulated slag, the maximum recovery of 4.47% Cu, 88.33% Co, 95% Ni and 93.82% Fe were obtained by leaching with 2N citric acid at 35°C with 10% pulp density (w/v) in 15 h using <45µm particles. The copper recovery in case of granulated slag was too low, owing to which the leach residue was subjected to second stage leaching with sulphuric acid, resulting in 72% Cu in 4h at similar conditions. The mechanism of the copper dissolution was studied by establishing pourbaix diagram for copper-citrate-water system.

PREDICTIVE DATA ANALYTICS FOR MANUFACTURING PROCESS OPTIMIZATION

Invited talks

Session 1: Nov 13, 2014: [09.00 - 11.00]

Process visualization and diagnostics in Iron and Steel Manufacturing

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To demystify complex metallurgical processes of producing coke, sinter and iron- deep-dive analysis is a necessity. Knowledge required for analysis is created by interpreting phenomenon of current practices through regular monitoring data generated from processes. Extraction of knowledge from data is accomplished in various ways. One method is creation of derived parameters by combining few measurements to create higher level of understanding. In another method 'soft-sensors' are developed wherein process knowledge is combined with emerging pattern from data to create new measurements. This enhance in-depth understanding of internal phenomenon within furnaces which otherwise cannot be measured due to inaccessibility or extreme environment of the process. One more type is development of predictive analytics by culminating knowledge through mishmash of various relationships of process parameters attained by churning current data. Also, empirical models based on statistical and artificial intelligence techniques are developed and leverage to predict process phenomenon.

The use of data mining techniques for reducing defective castings in foundries

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Statistical process control techniques have been used in manufacturing for quite a long time for process monitoring and quality control. With improvement in sensing techniques, increased availability of low cost computation power, and development of advanced data analysis algorithms, it is now possible to use advanced data mining techniques to significantly improve both the efficiency and productivity of manufacturing processes. Recent trends in 'big data analytics' is also an indicator of the investment that companies are making for exploiting data to improve overall process performance.Advanced data mining techniques include methods such as clustering and regression which are useful in building process models from data. These techniques are especially useful for complex processes where a first principles approach to modeling is either time consuming or difficult. The models developed from data can be combined with optimization and control techniques for improving process performance. As an example, the problem of reducing the percentage of defective castings from foundries is considered. Defective castings can be attributed to problems with metal composition and/or the properties of sand using in preparing the moulds. Industrial data corresponding to various sand properties and sand related defective castings for different components produced by a foundry has been obtained for a year. The cleaned data was used to

develop regression models for correlating defects to various sand properties at different levels of complexity (overall, component wise and period wise) using a fuzzy classification and regression approach. These models are used to identify and predict best operating conditions for the prepared sand for minimizing defective castings. A commercial cloud based software product called SANDMAN has been developed for a client, which can be used by foundries for data acquisition and storage and for predictive analytics.

Mining time-series data: An Introduction

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Many practical applications collect huge time-series datasets. For example, a data center may record CPU, disk and memory utilization of each sever as a multivariate time-series every day. Then we have a huge database, where each "record" is a time-series. Several operations can be performed on such a database: (1) querying and retrieving a time-series based on whole-series or sub-series match, (2) clustering the time-series records, (3) classification of time-series records (4) summarization of time-series records, (5) identifying "anomalous" time-series records, (6) discovering frequently repeated "motifs" among the time-series, and many others. In this talk, we will review some well-known algorithms for some of these tasks. All these tasks are important for analyzing time-series that occur in the manufacturing domain.

Session 2: Nov 13, 2014: [13.30 - 15.30]

Performance enhancement and defect elimination of products: a few computational intelligence based approaches

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For sustaining the competitive world, it is necessary for the industry to have control over the area where the level of uncertainty is high, but degree of tolerance is low. These are the areas of particular concern for which in depth process knowledge is necessary to achieve defect free products with high performance level. The properties of a product depend on its chemistry and process parameters in a highly complex manner. In most cases no physical model correlating the variables are present, and data driven models are the only solution to control the quality of the product. Several computational intelligence (CI) based techniques, viz. neural networks, fuzzy systems etc can deal such issues and map the non-linear input-output relationship successfully. Sometime enhancement of performance has the objective of improving conflicting attributes, e.g. increasing the quality and decreasing the cost of a product, where multi-objective optimization using evolutionary algorithms are effectively used. In case of product defects, most of the commonly observed defects, having lesser complexity and adequate knowledge of the domain experts, can be avoided through on-line process control. But in some cases, like the surface defects in flat products, they may remain undetected in intermediate processing stages and ultimately show up in the finished product during final inspection. In such cases it becomes difficult to find the actual geneses of the defects. At this point CI based approaches, e.g. rough set, might prove to be an effective way to solve such practical problems, otherwise the product yield could not be improved. Thus the basket of CI tools could

be effectively utilized in different aspects of product manufacturing, i.e. composition-processing-properties optimization and defects elimination.

Data analytics, modeling and cost reduction of industrial heat treating processes

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Modern industrial heat treating operations have sophisticated IT architecture, where significant amount of data in GB/TB per year, characterizing input material (chemistry, prior material quality, charge dimensions, quantity, and configuration), process parameters (furnace temperature, carbon potential and quenching oil temperature variation with time) and product quality (hardness, microstructure, case depth, and distortion) are generated. These manufacturing data are primarily used for occasional process audits and trouble-shooting a specific batch with quality issues. A closed-loop framework has been developed, where the manufacturing data along with domain understanding can be synthesized with physics and data-based modeling approaches to generate insights for process optimization and control. This approach is an expanded and generic view of typical smart manufacturing circle. This framework has been leveraged by synthesizing significant amount of data generated from a modern heat treating operation, along with domain understanding for analytics, modeling and optimization. The major deployable outcomes were new operating regime, new recipes and a recipe selection methodology. The approach also provided several unique process insights. This unique work has resulted in 12.5% productivity increase, along with ease of operation and specific energy reduction. The approach has not completely matured for standard deployments without significant technical expertise.

Model Predictive Control: A touchstone for advanced multivariable control

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Model Predictive Control (MPC) has become a touchstone for advanced control of complex multivariable systems. MPC was originally developed in late 70's in the process industry. With the availability of fast computers and microprocessors, MPC is increasingly finding application in many engineering domains such as robotics, automobiles, mineral processing, nuclear plants, metallurgical and aerospace industries. In a typical MPC formulation, a dynamic model is used for carrying out on-line predictions of the future behavior of the plant under consideration. The model can either be developed starting from the underlying physics of the system under consideration or using input output perturbation data obtained from the system or a combination of both. MPC is formulated as a constrained optimization problem, which is solved on-line, repeatedly, by carrying out model based forecasting over a moving window of time. Major strengths of MPC are abilities to handle multivariable interactions and operating constraints on the manipulated inputs, product quality and those arising from safety consideration in coordinated and systematic manner. More importantly, MPC facilitates optimal control of systems with unequal number of manipulated inputs and measured outputs. This presentation is aimed at proving a brief overview of the

model predictive control technology. The dynamic model used for online predictions is at the heart of any MPC scheme. Thus, an overview of approaches used for the development of the data driven dynamic models is presented first. A typical constrained MPC formulation is explained next. The presentation ends with a brief review of the recent developments in the area of MPC and its various industrial applications.

Role of big data & analytics in Automotive & Energy Sectors

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Big Data frameworks have revolutionized our ability to manage large volumes of data and derive insights. These frameworks are being increasingly leveraged in contexts where the growing needs of Volume, Velocity and Variety of information cannot be met by traditional data processing solutions alone. In my talk, I will cover the current state of being for Big Data - what it is and how it is different from the traditional distributed processing frameworks. The talk will cover discussion on some of the leading Big Data frameworks such as hadoop, columnar databases, schema-less databases and unstructured data processing. There will be a focused discussion on the technical strengths of these frameworks, what makes them scale well and the type of problems they are best suited for. We will talk about how some of the leading companies across the world have adopted Big Data. I will also cover some of the use cases for Big Data across industry sectors with focus on Automotive, Energy and Mining.

Contributory papers

Session 1: Nov 13, 2014: [09.00 - 11.00]

Data science techniques for inverse prediction in materials processing

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A computationally efficient model of processing-property relations is essential for effective exploration of design space for a given series of manufacturing processes. While generally available forward models help answer questions such as “what are the properties of a material when it is subjected to certain processing conditions”, we often need to address the inverse question of “what process parameters should be used to achieve desired properties. Physics based models used generally are not only expensive to simulate, but also, are not very useful for the inverse problem”. This is where data science based models are expected to help. However the quality of data based models critically depends on the quality and quantity of data available. To add to this, the inverse problem is many-to-one in nature, which makes it harder to learn a good model. Often, inverse problems are addressed by posing them as optimization problem around a forward model. In a complex, multi-dimensional problem space such as materials engineering, this is an expensive proposition with no guarantee of finding an optimal solution. In this paper we present an exploratory approach we are working on that uses Bayesian networks to build inverse models. In the network, we model process parameters, properties and intermediate state variables as nodes, and dependencies between them as edges. The network structure is essentially a means to encode prior

domain knowledge, which helps improve the quality of the model and reduce its dependency on data. For inverse inference, we compute the conditional joint distribution of process parameters for given property values. The joint distribution is then post-processed to extract mean vectors of process parameters. This can generate multiple mean vectors all of which are potential solutions. We present the results with an example of carburization, quenching and tempering process as the experimental setting. We compare these results with the results obtained by the optimization approach using a forward model. We also present the results of a hybrid approach that combines the Bayesian inverse inference with the optimization approach.

SUSTAINABILITY: REMANUFACTURING, REFURBISHING & RECYCLING

Invited talks

Session 1: Nov 13, 2014: [15.30 - 17.30]

Sustainability and life cycle assessment of materials – Thermodynamic, kinetic and environmental dimensions

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The term sustainability has become a keyword in the context of resource conservation, social existence, economic prosperity, political policy making, industrial growth and climate change and no subject has been as extensively debated as sustainability in recent times. In this talk, I will address the sustainability of materials especially in the context of Mining, Mineral Processing and Metallurgy. An attempt will be made to analyse the thermodynamic, kinetic and environmental dimensions of sustainability.

One of the comprehensive methods of assessing sustainability of materials is through a life cycle assessment study. The whole life cycle of a material more specifically metal (after the formation of the universe through cosmological processes) would comprise of ore formation through geological processes, mining, mineral beneficiation, metal extraction or production, manufacturing, use and end-of-use which could be recycling or burial back to earth to form the mineral. The life cycle assessment procedure for materials including life cycle inventory analysis and life cycle impact analysis (environmental, human toxicity and raw materials sustainability) will be highlighted. Case studies from literature on the life cycle assessment of some materials will be presented.

A success story of recycled magnetite for coal beneficiation in Tata Steel

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The first coal beneficiation plant in India was set up by Tata Steel in 1951 at West Bokaro and in 1953 in Jharia. Over the years more beneficiation plant capacity has been added in both West Bokaro and Jharia, the coal divisions of Tata Steel. Currently the total capacity available is about 8.5Mtpy. Two processes are used in the coal beneficiation plants of Tata Steel to reduce the ash content of the ROM coal; (a) Dense Media Cyclones [DMC] (b) Froth Flotation. Magnetite is used in the DMC for achieving the desired separation between clean coal and the “discard”. So called discards are further processed in some of our plants to recover a secondary product as power plant feed coal. Finely ground magnetite is kept in perpetual suspension in water and constitutes the Dense Media. Relative densities of separation taking place in DMC, e.g. 1.50 or 1.65 or 1.75, dictate the proportions in which magnetite would be mixed in water.

Magnetite is considered to be the heart of DMC process. Magnetite was a relatively unexplored area at Tata Steel, because of the continued satisfactory experience. In the year 2008 the consumption and quality of magnetite suddenly deteriorated, though the supplier was apparently supplying as per the specifications. Since then considerable in-house work had been carried out to transform the specifications from a general one to plant specific one. Emphasis was also laid on improving the recycling efficiency for magnetite. It was observed that adherence to the specified quality of magnetite improved the recycling efficiency. This led to the reduction in magnetite consumption over these years from 1.2kg per tonne of coal processed to 0.70-0.75kg. The figures are competitive with the world average and in many cases, superior. Further, the reduction contributed to the savings by Tata Steel in thousands of crores of Rupees. Efficiency of magnetite recycling improved the media consistency and that led to significant improvement in separation efficiency, in particular at the coal beneficiation plants at West Bokaro.

Session 2: Nov 14, 2014: [08.30 - 10.30]

Indicators of sustainable metal production using life cycle assessment

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Life cycle assessment (LCA) is now considered as a recognized tool to evaluate various processing routes for metal production. LCA assesses all the components and environmental impacts associated with a product, process or activity during its lifetime. LCA results assist industry/research with decision making. Input data inventory are used to determine greenhouse gas (GHG) footprint of metal production. Other environmental impact indicators (e.g. embodied energy, water and solid waste generation) of innovative metal production processes are compared with conventional processes to assess their suitability. A range of flowsheeting tools, techniques and LCA software, databases and other resources are used to determine the sustainability indicators of metal production processes. Assessing the sensitivity of environmental impacts to key input variables is also part of this process.

The aims of LCA studies are to identify 'hotspots' where opportunities exist for reduction of any environmental impact to improve the sustainability of a product or process. LCA studies of mining, mineral processing and metal production have been carried out at CSIRO Australia. This work has included iron and steel, ferroalloy, copper, nickel, aluminium, gold, lead, zinc and titanium. The toxicity impact from by-products of the mineral industry has also been reported. The published results from some of our case studies will be presented. This research is being conducted under CSIRO's Minerals Resources Flagship and is being used to identify opportunities for reducing impacts in greenhouse gas emissions, water consumption, resource depletion and waste generation, and to improve efficiency of metal production.

Hydrometallurgical treatment of metallic wastes to recover high value products

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Metallic waste generation is an integral part of modern day life. Therefore, it is now becoming much easier to find metallic wastes than any primary metal reserves. Treating these wastes for metal reclamation and value addition not only give the waste a second life but this also helps to conserve valuable natural resources and prevents environmental pollution. Recycling of metallic wastes is inhabitable for sustainability and growth of human civilization. This paper highlights the major outcomes of our continued effort for value addition to several types of metallic wastes at CSIR-National Metallurgical Laboratory (CSIR-NML).

Innovative hydrometallurgical process concepts were adopted to recover different metals not only in pure form but also to produce variety of high value tailor made products. The processes developed for recycling of various metallic wastes includes: spent catalysts, spent rechargeable batteries, e-wastes, alloy scraps, process residues, spent pickle liquor, bleed solution etc. In recent past few of the developed processes has been transferred to industries for its possible commercialization. The brief details of the process will be discussed during presentation.

Contributory papers

Session 1: Nov 13, 2014: [15.30 - 17.30]

Evaluation of electronic waste recovery

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Electronic waste (e-waste) is today the fastest growing solid waste in the world due to its unique characteristics like planned obsolescence, high technology and a fast growing consumer market. This type of waste can contain over 100 highly toxic and potentially hazardous substances to human health and to the environment. However, it can contain precious metals which are economically valuable, such as gold. These factors, amongst others, motivated the governments of different countries to take administrative cum policy action on this issue. In many of these countries, the export of waste is not permitted. In Brazil, from 2014, the 'Política National de Residuos Solidos' (PRNS) or Solid Waste National Policy, takes effect with the creation of legislation no 12.303/10 regulated by decree 7.404/10 in which electronic waste partially fits into Article 13, referring to industrial solid waste where all companies producing electronic items in general will be responsible for their products' life cycle. However, it is still necessary to include a focused regulation about its specification, production, disposal, etc. The recovery of gold from e-waste, also called Reverse Logistics, is an environmental and commercial measure of great importance, which can only bring benefits to all involved. It is known however that the biggest part of the solid waste in Brazil is exported. The aim of this research is to verify the potential economic benefits linked to the recovery of

gold from mother-boards, PCI Express boards and cell phone circuit boards, to which mechanical and hydrometallurgical processes are applied (through extraction by relevant and economically viable solvents for this purpose) encouraging future entrepreneurs to join this excellent idea known as urban mining.

Solid waste management by development of cold bond agglomerates -A zero waste initiative at Essar steel

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The recycling and/or refining of iron-bearing by products from integrated iron and steelmaking production is a vital technological endeavor for today's steel industry. Considerable amounts of fine grained dust, sludge and mill scale are generated from iron and steel production annually. Traditionally, a large part of these byproducts were fed to the sinter plant but strict environmental regulations and increasing economical incentive to recycle by-products on-site have led the industry to consider recycling techniques that are economically viable and environmentally sound. One such technique, Cold Bond Agglomeration is a low energy, environmentally suitable alternative to sintering of these by-products. This paper presents the technology of cold bonded agglomeration either through pelletization or briquetting for effectively recycling various steel plant by-products in the steel melting cycle. Challenge lies in selection of appropriate polymer/cement based binders based on their surface characteristics and to develop products which can be charged in furnaces possessing sufficient cold strength and high temperature stability. Briquettes of coal fines, cold direct reduced iron fines etc. and pellets of fume extraction system dust, sludge have been studied for cold crushing strength. Reactivity index, reaction stability index etc. have also been discussed. The study indicates that these pellets and briquettes are suitable for charging in various Iron and Steel making units.

Evaluation of laser cladding process on AISI H13 hot work tool steel for PDC die repair application

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This study reports a detailed investigation of the microstructure and mechanical properties of laser clads of AISI H13 tool steel on substrate of same steel, in as-clad and postclad tempered (550°C for 2 hours) condition. Laser cladding was carried out using a 6 kW fiber coupled diode laser in continuous wave mode with coaxial powder feeder. Laser processing parameters: power, processing speed and powder feed rate were optimized to achieve defect free clads with good metallurgical bonding. Clad microstructure, phases formed, crystallite size, micro-strain, residual stress and mechanical properties were assessed.A maximum hardness of 650 VHN, which is about 45% higher than the hardness of conventionally hardened and tempered substrate, was observed. Post clad tempering resulted in elimination of grain boundary or inter-dendritic precipitates which were seen in as-clad condition. But appearance of fine carbides in the lath

martensitic structure was observed, perhaps taking the hardness to 600-650 VHN. Residual stress due to laser cladding was found to be compressive, which marginally decreased after post clad tempering. Micro-tensile testing of specimens machined from the clad zone (as clad) transverse to cladding direction showed high yield strength but a brittle failure. On the other hand, specimens longitudinal to direction of cladding showed lower strength but a significant increase in ductility viz., 4.7% elongation was observed. Post-clad tempering resulted in further increase of elongation to 8%. Microstructural variations and mechanical behavior are explained in detail.

Utilization of sludge generated in integrated steel plant by micro pelletisation and sintering

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Sludge is generated at different stages of blast furnace operation, sinter plant and BOF in integrated steel plants from gas cleaning plant. Sludge dumping renders the land unsuitable for any other use; more sludge more land required for dumping. Sludge utilisation therefore lads to reduced land usage, resources conservation through reduced use of virgin ore and improved environmental compliance. Sludge is generated as sticky paste of micro-fines containing high moisture. Thus removal of moisture from sludge and micro pelletisation for further handling and processing is essential before recycling through sintering. Samples of sludge collected from converter, blast furnace and sinter plant of Bhilai Steel Plant were tested in pot sintering facility at RDCIS after pelletisation in Eirich mixer. For micro pellet preparation, all sludge were dried and mixed in a predetermined ratio. Pellets were charged along with sinter mix from 5% to 30%. It has been found that there exists slight improvement in all process parameters with use of sludge up to 20 kg/t of sinter. Above that level, negative effect appears on productivity, yield, return sinter and VSS. Chemical analysis of sinter produced from pot sintering shows that there is slight change in Fe (T) & SiO_2 content in sinter. Thus, sludge generated in major units of steel plants could be recycled through sintering after pelletisation up to 20 kg/t of sinter level.

Prediction of metallic iron in LD slag by determining apparent density

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LD slag is a solid waste generated in integrated steel plant whose utilization is being explored by industry experts. In Tata Steel, LD slag fines are being utilized in sinter pile making whereas lumpy metallic fractions are being utilized as metallic charge/scrap in blast furnace/LD vessel. LD slag not recycled in industrial processes is either sold out or dumped. Any of these applications requires fast and accurate determination of metallic iron in LD slag to have better process control and avoid monetary loss if it is sold out.There are few approaches to test the metallic iron content. The Kentucky transportation issues a method to cover the determination of particles (by mass) containing iron in blast furnace slag. This method applies a magnet to collect the metallic iron particles. Obviously, this method provides an approximate result, metallic particles often being embedded in non-metallic particles in the iron making or steel making slags. Another method uses mercuric chloride to extract metallic iron from slag. However, a protective film is formed on the surface of metallic iron to prevent further dissolution. This method usually gives lower

metallic iron value than the true value. In Tata Steel, metallic iron content in LD slag up to size fractions 80 mm is determined by hammering the sample in three to four steps to separate out metallic part from nonmetallic part. For size fractions greater than 80 mm and upto 300 mm, metallic iron content is determined by water displacement method. Both methods of determining metallic iron content are man intensive and take long analysis time. The objective of this study is to develop a method by which time for determination of metallic iron could be reduced and process would be less man intensive and safer.

Kinetics of nickel extraction from spent catalyst in hydrochloric acid solution

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In this paper, the kinetics of nickel extraction from spent nickel catalyst (NiO/Al_2O_3) by using hydrochloric acid leaching has been studied. During the study, the effect of concentration of hydrochloric acid, temperature, stirring speed, particle size and retention time were investigated. The leached residue at every stage has been subjected to XRD, SEM and chemical analysis. The data obtained had been subjected to kinetic analysis and it has been observed that the reaction is diffusion controlled. The diffusion reaction is well accelerated through the network system of the catalysts with an activation energy of 23.37 ± 0.78 kJ/mol.

An approach to energy minimization through use of steel slag with cement with simultaneous reduction of CO_2 in steel plants

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It is well known that mineral carbonation of EAF steel slags can be used to form thermodynamically stable high strength end product. Such a product can be used as aggregate in cement concrete mixes which is expected to reduce the overall energy consumption in the preparation of such concrete mixes. Energy consumed by the Indian cement industry was 607 Peta Joules (PJ) (10^{15} J) in 2007. This accounted for 9% of the total energy consumed by the industrial sector. By replacing cement, say up to 45 %, we could cut down 273 Peta Joules (PJ) (~45 % of consumed energy) because of the use of carbonated waste product. CO_2 emission of a steel plant is simultaneously reduced. There are two possible approaches to utilize this technique; use carbonated EAF steel slag as aggregate in cement concrete or replace adequate proportion of cement without harming noticeable inherent properties of cement and carbonating the blended masonry slag cement concrete block. In addition to reducing overall CO_2 emission of a steel plant, carbonated slag or carbonated blended concrete block is reported to develop strength of about 10 -40 times greater than their noncarbonated counter parts. Additionally, it is needed to devise a system with optimal process variables that can sequester maximum CO_2 with minimum energy requirement so that above objectives are met. A possible scheme towards that has been discussed in the paper. The study indicates that by admixing cement with steel slag we are increasing the CO_2 sequestration capacity of the steel slag. In addition, carbonated counter parts easily pass the prescribed strength requirement demanded by the standards. Cements containing 15% or 30% slag satisfy the requirements of the strength class 42.5 of EN 197-1. Cements containing 45% slag satisfy the requirements of the strength class 32.5 of EN 197-1.

Metal oxide open foams to produce solar thermal hydrogen via redox cycling

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Fossil fuels are believed to be responsible for the threat of global warming accompanied by a climate change. Development and use of alternative, clean energy sources cannot therefore be overemphasized. When it comes to a realistic clean and efficient energy vector, nothing can possibly beat hydrogen. However, a clean and reliable source of hydrogen is a perennial concern. Fortunately, when a cheap and efficient solar thermal power and heat are factored in, especially for a country like India with an estimated average solar irradiance of about 25 MW/km^2, the advantages of solar-thermal hydrogen production and usage becomes very promising. One of the ways of producing hydrogen is a high temperature Thermochemical Water Splitting Cycle (TWSC) on a partially reduced metal oxide coupled with a separate step of electrochemical regeneration of the spent metal oxide. In this work a set of porous open foams based on oxides of iron and cobalt have been planned to be made via gel casting. The sintered porous foams will be characterized for their porosity, surface area and suitability for a large number of high temperature redox cycling to produce hydrogen.

Separation of metals from waste printed circuit boards by mechanical/physical techniques

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Generation of e-waste is major threat worldwide and in India (0.15 kg/capita e-waste) too.Proportion of PCBs in e-waste is ~3% and its metal fraction is ~30%. Metals present are encapsulated by the plastic and resin which hinder the leaching process. Hence, efficient separation of metals from non-metallic is important. Difference in the properties of metals and nonmetals like- magnetism, electrical-conductivity, relative density, particle geometry etc. are exploited for their separation. Many researchers have practiced corona discharge electrostatic separator in the size range of 5 mm-0.5 mm. Effective separation has also been achieved for higher size fraction. By using Eddy-current separator, Al separation from a coarse mix is reported. Flotation, air-classification, dense media separation, jigging etc. are the other techniques, reported. These processes require crushing/grinding by hammer mill, stamping mill etc. to ensure liberation of metals. Pyrolysis process eliminates need of pre-processing, as the organic materials are decomposed making PCBs fragile and by easy hammering the metal sheets are separated. Production of fuel gases, various chemicals is added advantage, while generation of toxic gases limits its application. Electronic component mounted on the PCBs may be removed by combined effect of rotation and heating at 350°C in diesel. Heating results melting of solder and centrifugal force results separation of solder and electronic components mounted on it. Using ionic liquid 1-ethyl-3-methylimizadolium-tetrafluoroborate instead of diesel, similar results have been reported. It causes swelling of PCBs. Thus delamination and removal of Cu sheet without any further treatment is possible. Swelling of PCBs by N-N-dimethylformamide and dimethyl sulfoxide promoting delamination and easy removal of Cu sheet from resin sandwich has been also reported in literature. However, highly efficient technology ensuring complete separation, cost effective and eco-friendly operation is awaited. That will ensure safer and cleaner environment and cut-down the size of huge stock-piles of e-waste.

Session 2: Nov 14, 2014: [08.30 - 10.30]

Higher waste recycling for fuel reduction in iron ore sintering

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In any integrated steel plant, large quantities of solid wastes are generated during various stages of iron making and steel making processes. Sinter making process gives an avenue to utilize maximum amount of solid wastes which contain useful elements like Fe, C, Si, Mg & Ca etc. There are several solid wastes which contain carbon in considerable proportion like blast furnace flue dust, blast furnace gas cleaning plant sludge etc. Addition of that replaces coke breeze used as a prime fuel for iron ore sintering process. Fluxing elements in steel plant solid waste remain either in the form of free oxides like CaO, MgO or in various complex forms of CaO-SiO_2-FeO-MgO-Al_2O_3 etc. Therefore its utilization does not require excess fuel for removal of combined CO_2 which otherwise require in case of usage of limestone, dolomite etc. That helps in reduction of coke breeze consumption as well as CO_2 emission in iron ore sintering process. Various innovative processes were developed at Tata Steel Jamshedpur to control the issues related to high super fines and chemistry fluctuation in solid wastes as well as control of its undesirable elements like Al_2O_3, P_2O_5 etc. Several new types of wastes were also included in recycling through sinter making like road sweeping dust, muck generated at sinter plant and blast furnaces, etc., which were dumped previously as land filling materials. Achieving Indian bench mark value of solid waste recycling at Sinter Plants to the level of >150 kg per ton of net sinter helped in reduction of coke breeze consumption rate from 80 kg (FY'13) to 73.3 kg (FY'14) per ton of net sinter besides maintaining the sinter quality at desired levels. The present paper analyze the various attributes of solid waste recycling which has helped in reduction of solid fuel rate at Sinter Plants.

Implementation issues of top gas recycle blast furnace in integrated steel plant

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To tackle the issue of CO_2 emission, various breakthrough programmes like ULCOS, COURSE-50, etc. are running in different parts of the world. Under the ULCOS programme, Top Gas Recycle Blast Furnace (TGRBF) has been recognized to have potential for reducing the CO_2 emission significantly by replacing/modifying the existing blast furnace. This furnace is operated under oxygenated blast and the top gas is recycled back through tuyere and shaft, after passing through a CO_2 separation unit such as Vacuum Pressure Swing Adsorption (VPSA), membrane separation etc. It has been observed that under high oxygen enrichment conditions, there is a deficit of downstream fuel availability beyond the iron making system, which is identified as a major issue for implementation of this technology in an integrated steel plant. Based on the model developed for the TGRBF system, an attempt has been made in this paper to identify the optimal conditions for minimizing the net CO_2 emission of TGRBF system along with maximization of downstream energy available (DE), by maintaining feasible internal furnace state

conditions like thermal reserve zone temperature and raceway adiabatic flame temperature. Various input parameters such as O_2 enrichment, hot blast temperature, recycle gas temperature and its volume, are used to generate the function of carbon rate and CO_2 emission, along with constraints of downstream energy available, thermal reserve zone temperature and raceway adiabatic flame temperature. The results reveal that CO_2 emission and DE are in conflict to each other. For any target reduction in emission which presumably leads to a deficit of downstream energy, an additional fuel source needs to be used, but this will again generate additional CO_2 emission according to the fuel used. Therefore, reduction in emission needs to be compromised with the downstream energy requirement of an integrated steel plant complex.

Effective and innovative utilization of solid wastes of EAF at JSPL, Raigarh

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The steel industry in India is growing at phenomenon rate. Crude steel production is expected to go up from the current 81Mt to around 100-110Mt by 2014-15. As per ministry of steel, the target of steel production by 2025 is 300Mt. With this increased steel production, the amount of byproducts is bound to increase. The biggest challenges and threats for the steel manufacturers will be effective utilization of solid wastes to comply with the environmental norms. The recent shift in environmental norms has induced thinking in the direction of economic utilization of solid waste like Electric Arc Furnace (EAF) slag. EAF slag is a strong and durable material which is highly versatile and ideally suited for use as an aggregate in many applications. Studies have concentrated on developing sustainable solutions to efficiently and economically utilize these wastes and to prevent harm to environment.JSPL has adopted the technology from Ecomaister, where Slag Atomizing Plant (SAP) converts waste steel slag into highly competitive product called Precious Slag balls (PS balls) and Ladle Furnace slag balls (LF balls). In addition to that, JSPL also developed many practices for recycling of the solid wastes and using EAF separated slag which is high in iron content, in Blast Furnace and charge mix in sinter plant as fluxing components. The paper focuses on the effective handling of solid wastes in steel melting shop after processing the same to PS balls; usage of EAF separated slag in BF and sinter plant. The paper also highlights the benefits derived by the innovative slag handling practice at JSPL, Raigarh.

Oxidation resistance of magnesia carbon bricks and the recycling viability

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The oxidation resistance of the magnesia carbon bricks is usually an important factor to guarantee a high level performance of the lining, since carbon is easily oxidized at high temperatures. The resistance to decarburization is usually achieved by using metallic powders in the formulation. However, the introduction of these metallic powders, especially Al powder, affects negatively the recycling of such products. Al powder can form Al4C3 during the operation of the furnace and this phase can hydrate in the same way lime does. Because of the possibility of the presence of Al4C3 in the residual brick the recycling operation is more expensive and takes more time. If there is no Al powder in the formulation there is no risk of hydration and the recycling procedure is simpler and cheaper. The use of antioxidants is

recommended if the oxidation is the main wear mechanism of the lining. Even in this case, if the oxidation takes place in the preheating step, the use of a "special coating" can be a smarter option than using antioxidants. If the focus is recycling, the use of metallic powders should not be considered as a first approach to control the oxidation.

Iron oxide waste to clean arsenic-contaminated water

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Serious manifestations of arsenic toxicity in majority of human population consuming contaminated water has led to development of number of remedial methods including adsorption onto iron oxide based natural and synthetic materials. Iron oxide adsorbents generated as waste in industrial processes may be utilised for arsenic remediation. These adsorbents can be considered better in terms of least production cost as well as minimal environmental implications. Here, we studied arsenic removal from contaminated water using iron oxide wastes generated in pickling unit of a steel plant. The iron oxide obtained by steel pickling was subjected to controlled reduction in fluidised bed reactor using gaseous reductant producing magnetic iron oxide. Prior to adsorption studies, physicochemical characterisation of both the iron oxides was undertaken. This was followed by batch equilibrium and kinetics adsorption tests to investigate arsenic (V) removal properties. Several parameters such as time, pH, arsenic concentration, adsorbent dose etc were investigated using synthetic solutions. Arsenic affected ground water samples collected from West Bengal were also tested using both iron oxide adsorbents. Experimental results showed more than 90% arsenic removal within 10 min, not depending on pH of water with appreciable loading (0.12-0.17 mg As/g) on both adsorbents. Presence of anions such as $SO_4^{2-}$and PO_4^{3-} adversely affected arsenic adsorption. While testing real ground water samples, initial arsenic level of 0.010–0.018 mg/L was brought down to 0.002–0.008 mg/L. Our study has established potential use of an industrial waste for the removal of arsenic (V) from water.

Refractory insulation bricks from fly-ash and rice husk

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Worldwide more than 65% of fly ash produced from coal power plants is disposed of as landfills. In India fly ash landfill covers an area of 40,000 acres (160 km^2). More than 300Bt of coal fly ash are produced annually worldwide and in India it is more than 120Mt. Fly ash has become a matter of concern in recent years due to increasing landfill cost and sustainability issues. At present major fly ash utilization is in construction industry for concrete and building bricks making. That constitutes only about 28% of total generation. Another organic waste produced is rice husk. Disposal of that is also a problem. Refractory industry, particularly the insulating refractory brick segment using several thousand tones of traditional fire clay, a alumino - silicate type material, can be a potential opportunity for the utilization of fly ash, a similar alumino silicate material. Insulating refractory bricks used to save energy, reducing heat losses etc, at present are made of plastic fire and saw dust. The saw dust is burned away during the firing of the brick, thereby rendering the brick porous and hence insulating. The term refractory implies the ability to withstand high temperature, e.g. 800°C to 1400°C, or more. The term insulating in the context of the

present work means a thermal conductivity at 100°C of less than 1.0 w/mk. This work evaluates the utilization of dry ash in a composition of refractory insulating body. In place of saw dust, rice husk was used which is eliminated during firing process, generating porosity in the product. The insulating refractory bricks so made with fly ash and rice husk were evaluated in terms of technological characteristics, according to IS 2042. The products were found to be at par with IS 2042 norms, rather better in the same respect.

Pre-treatment of coke oven effluent to control the toxicant load at the inlet of the effluent treatment plant at RINL

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Effluent generated from the coke oven batteries during the carbonization process contains toxic chemicals like phenols, cyanides, ammonical nitrogen, rhodanides and in overall COD content that needs treatment to bring them to statutory norms before its disposal or for any downstream usage. The process of Mechanical, Biological and Chemical (MBC) treatment used to remove them at RINL demands feed consistency at the inlet. The previous pretreatment facility was to remove ammonical nitrogen in three steam stripping columns in a single stage operation that did not deliver satisfactory results. Accordingly several possibilities were explored supported by laboratory studies. Encouraged by the laboratory scale result with a double stage steam stripping and modified soda dozing operation, the plant scale trials were successfully implemented in November- 2013. The modified double stage stripping scheme has resulted in substantial removal of Ammonical nitrogen, Cyanides, Phenols and as a whole the COD content thereby reducing the load at the MBC inlet with desired consistency. The overall effect reflected in bringing down the toxicants level of MBC outlet water close to the pollution norm. The present paper highlights the above effort.

Study about the properties and utilization of red mud

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Red mud is a solid waste residue of the digestion of bauxite ‘ores’ with caustic soda for alumina production. Disposal of large quantities of red mud generated all over the world poses an ever increasing problem of storage, land availability and associated cost and pollution. Because of the complex physico-chemical properties like relatively complex composition, coupled with its fine particle size distribution, high pH, poor settling properties and some toxic rare metal associations make it a very challenging task for the designers to find out economical utilization and or safe disposal of red mud.With increasing production of red mud, the environmental problems caused by it are becoming serious. Thus integrated treatment of red mud appears to be a necessity.This paper provides an overview of the composition and the basic characteristics of red mud. The research progress in comprehensive utilization of red mud in India is also summarized. The red mud can be used as secondary resource to recover valuable metals such as titanium, iron and aluminum and also improves the reinforcement and wear resistant properties of metal matrix composite. The use of red mud as building materials such as bricks, tiles and partial replacement of cement by red mud is the most effective way to reduce the stockpiling of red mud.Red mud used for

environmental remediation materials is a new hotspot and worth promoting for its simple and low cost processing.

Utilization of solid waste generated from steel plants

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Steel industries produce over 3000 varieties of steel and the demand of steel is growing day by day. Generation of solid wastes is also increasing with the increase in production of steel. Waste utilization is prime concern of the industries from environmental point of view. These wastes involve Blast Furnace slag, flue dust, LD slag, LD sludge, mill scale, lime fines. Apart from impurities these waste also contains useful metals which is needed to be recycled. So industries are interested in recycling of such wastes as well as using this waste in different ways. In developed countries solid waste generation is less and waste utilization is more, but in India reverse is the case. This review paper is aimed at a comparative study of generation and utilization of waste in steel industries all over the world. It will throw a light on the future direction of steel industries in India about the generation and utilization of waste.

CONTRIBUTORY PAPERS FOR POSTER

Nov 14, 2014: [10:30 - 11:15]

Change land open die extrusion

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In change land open die extrusion, the land diameter is changed in stepwise manner and large strain is induced in the material. Due to absence of the container, the container-wall frictional force is eliminated and hence there is large reduction of force required for extrusion. In this study we extrude 25 mm aluminum billets of varying (l/D) ratios through the open dies with stepwise decreasing land diameter to obtain the diametric change from 25 mm to 21 mm. The samples were extruded through Universal Testing Machine. Mechanical and microstructural characterization is done on the extruded samples to check its properties.

Creep damage assessment of hydrogen reformer tube

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Service exposed reformer tubes in refineries and petrochemical industries have finite life because of prolonged exposure to high temperature and stress. The tubes experience temperatures beyond 900^0C for a considerable length of time, because of which creep becomes the prime mode of failure. This investigation incorporated measurement of the tensile properties and at 870^oC creep rupture properties. Tensile testing was done to calculate the 0.2% yield stress so that the stress or load required for creep test can be determined, which should be (>UTS/2) for sufficient creep in material. Creep tests were carried out at the temperature of 870^0C and at the initial stress of 50,56,62,68 MPa for both top and bottom part of the tube.Sample for creep test and hot tensile test were cut from the 11 years' service exposed hydrogen reformer tube which was made of HK 40 steel (25 Cr/35Ni/1.5 Si/1 Mn/0.45 C/Ti /Fe). A set of creep curves (4 nos.) were generated for each stress level to examine the scatter characteristic of the material. A Monte-Carlo simulation was carried out to estimate the time to reach a specific damage state. It is of prime importance to mention that the simulation based damage prediction and associated scatter was in close agreement with experimental data. Microstructural analysis of crept sample was carried out to get the relation between void fraction area, creep cavity, location of creep cavity, aspect ratio of voids and average no. of voids with respect to true strain value. Almost all of the voids appeared near grain boundary and the larger of these voids were located at grain edges and corners i.e. grain boundary triple points. Remaining life assessment showed that bottom part had less life left compared to upper part of the tube as bottom part of tube was exposed to severe environment.

Management programme taken on safety for decrease of back pain & sprain

Atindra Nath Misra, AGM, DSO, SMS

During making steel, the dust laden gas is cleaned by water spraying at scrubber. This water is taken at system, where slurry is taken out. Earlier it was manually operated. Two to three persons were required to open the valve. Sprain & back pain were reported frequently. Now only one person can open the valve without any difficulty. Slide gate opening cylinder is hung by chain at turret. At continuous casting plant under steel melting shop, steel ladles are opened & controlled by slide gate cylinder hydraulically. Earlier people had to bend & lift the heavy cylinder. Sprain & back pain were reported. Now people can do it easily at required position.

Synthesis of 1D cuprous oxide nanowires for electrochemically-gated PMOS transistors

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One-dimensional nanomaterials such as nanorods or nanowires (NWs) are very attractive for various Nanoelectronic devices. Cuprous oxide (Cu2O), a well-known p-type semiconductor with a band gap of 2 eV, has cubic crystal structure and is a very good candidate for conversion of solar energy into electrical or chemical energy, optoelectronic or light-emitting devices and as well as for catalysis. We present a low-cost, large scale solution-phase synthesis of single crystalline cuprous oxide NWs that shows very high aspect ratio. Synthesis has been done with hydrothermal treatment of Cu(Ac)2 and pyrrole at 250°C for 5hr. The powder X-ray diffraction (XRD) confirms cubic cuprite structure. Scanning electron microscopy (SEM) images show smooth and straight NWs with minimal surface roughness. The length of most Cu2O NWs exceeds 100 nm, while the diameter varies between 100- 150 nm. Fourier transformed infrared spectroscopy (FTIR) and High resolution transmission electron microscopy (HRTEM) show the presence of organic layer surrounding the NWs which is further removed by DMSO treatment. Subsequently, the cleaned NWs are harvested and used to build low voltage operated, single-nanowire transistors that are gated with composite solid polymer electrolytes and operates as an accumulation mode p-type MOSFET (PMOS); detailed transistor characteristics is also presented.

Successful recycling and conversion of solid wastes generated during manufacture of stainless steel into value added products

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During the production of stainless steel waste products such as EAF dust, AOD dust, grinding dust, hot rolling scale, EAF, AOD and LRF slags and filter cakes from neutralization of pickling spent liquor are generated. For three tons of stainless steel produced nearly one ton of such wastes arise. To ensure a cost effective, environment friendly and sustainable stainless steel production, optimum recycling and reuse of these arising has become essential.This paper describes technologies developed for treating stainless steel

wastes in different counties and covers the technology evolved in our plant based on the composition and characteristics of each type of arising. In this approach EAF dust and filter cake from neutral electrolytic and sulphuric acid pickling lines are briquetted and smelted in submerged arc furnace (SAF). Whereas AOD dust, mill scale and filter cake from mixed acid bath are mixed with carbonaceous reductant, briquetted and fed into EAF. In this manner wastes which are high in zinc and sulphur are treated in such a manner as to obtain desulphurised master alloy containing chromium, nickel, manganese and copper from SAF and concentrated zinc oxide from its bag house. The master alloy is used in EAF for production of stainless steels and zinc oxide rich dust is utilized by buyers to produce micro-nutrients for soil. On the other hand wastes which are lean in sulphur are fed into EAF along with a reductant to recover valuable metallic elements more economically and also provide a part of flux addition to the furnace since AOD dusts are rich in CaO and MgO. Slags from EAF, AOD and LRF have distinctive characteristics. These are suitably processed and sold after recovery of metallic elements for appropriate applications to generate surpluses.

ADDITIONAL INVITED TALKS IN VARIOUS THEME SYMPOSIA

Process Metallurgy for Ferrous Metals: Ironmaking

Session 3: Nov 13, 2014: [15:30-16:00]

Study of raceway effect on gas-fines in decreasing gas velocity in a packed bed

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In this work, the focus is on the numerical simulations of gas and powder flows in a packed bed with a lateral gas-fines injection. As it is known now, BF aerodynamics can be represented more accurately by a packed bed under decreasing gas velocity condition if it is not being considered as a moving bed reactor. Therefore, the decreasing gas velocity condition is employed in this study. As raceway shape and size are most crucial factors in controlling the fluid flow behaviour in the blast furnace, therefore, an attempt has been made to study the effect of raceway shape and size on the distribution of gas and fines flow within the bed. Raceway shape and size in a packed bed has been obtained from the previous investigators study who have used the iso-stress raceway boundary concept to find the raceway shape using a continuum based stress model with lateral gas injection. Turbulent gas and fines flow equations have been developed and solved, for two-dimensional case using finite volume method to describe the flow behavior in a packed bed. The model's predictions have been validated against the published experimental data.

Glass and Advanced Ceramics

Session 1: Nov 13, 2014:[09:30-10:00]

National Fusion Materials Program and the use of advanced ceramics

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Recent advances in the fusion science and technology have dramatically improved the prospects for practical fusion power to be achieved in about 50 years from now. The international effort to build the ITER is seen as the first essential step on the path of demonstrating the scientific and technological feasibility of fusion energy. Further steps of building a Demonstration ('Demo') Reactor and subsequently the Fusion Power Plant, have been planed by world scientific community to achieve the final goal of exploiting fusion energy for the practical use. However, the dream of fusion energy can be realized only if the materials suitable to withstand the harsh environment of high thermo mechanical stresses, high heat loads and severe radiation damage can be fabricated and tested for their feasibility through rigorous R & D.

Different types of materials are required to be developed for the future fusion reactors. These are structural materials, Functional materials, materials for Test Blanket Module(TBM) and other supporting systems. Efforts are being taken to develop various steels such as Reduced Activation F/M steels, ODS steels etc., Composite materials (SiC_f/SiC) and Vanadium alloys as structural materials. Many functional materials are required for Tritium Breeding, shielding, Plasma facing, diagnostics and cooling in the reactor. The advanced ceramic materials like Al_2O_3/ Er_2O_3, $MgAl_2O_4$, Lanthanum Niobatehave been identifiedfor various functional requirements in the reactor. It requires enormous efforts to develop, manufacture and qualify fusion reactor grade materials. Both, Materials Science and Materials technology development programs are required for this purpose.

This presentation will give an overview of fusion reactor materials with main emphasis on structural materials and ceramic materials. Their functional requirements will be discussed along with challenges posed in the reactor environment. Recent advances and directions in high-performance materials R & D will also be discussed.

Materials for Energy Sectors: Thermal, Solar, Hydro and Wind

Session 1: Nov 14, 2014: [08:30-09:00]

High Temperature Boiler Materials forIndian Advanced Ultra Super-Critical (AUSC) Thermal Power Plant Technology

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Coal fired thermal power plants will continue to contribute a significant part of electric power generation in the near future. Significant efforts are being made today towards enhancing the plant efficiency and reducing the carbon-dioxide emissions. To achieve higher efficiency, it is necessary that the power plants be operated at higher steam temperatures. Towards this, a mission programme has been initiated in India towards evolving a design for an 800 MWe capacity Advanced Ultra Supercritical (AUSC) plant with steam parameters of 710^0C/720^0C/320 bar, to achieve high efficiency. To implement this programme, advanced materials that show good performance and life under such conditions are required. In this regard, two high temperature materials, viz., 304HCu austenitic stainless steel (304HCu SS) and modified nickel-base Alloy 617 (Alloy 617M) have been identified for development for boiler tubes.This paper provides the details of efforts made in India towards development of these materials.

For development of indigenous 304HCu SS and Alloy 617M, a stage-wise characterization approach was adopted to ensure establishing optimised process route for obtaining quality tubes. Detailed characterisation of thermo-physical properties, characterisation of microstructure, heat treatment response and evaluation of thermo-mechanical processing map enabled necessary understanding of the behaviour of these materials during mechanical working and heat treatment. Based on these metallurgical inputs, the process route has been optimised for successfully manufacturing 52 mm diameter seamless tubes of 304HCu SS and Alloy 617M, with wall thickness of 9.5 and 11.9 mm, respectively. These 304HCu SS and Alloy 617M tubes met the specifications for all the mechanical properties and grain size requirements, as also non-destructive testing involving ultrasonic examination with defect sensitivity of 3%

of nominal wall thickness, which is more stringent than the 5% notch depth specified in ASME SE-213. Also, 304HCu SS and Alloy 617M welding filler wires, of 2.0 and 0.8 mm diameter, were manufactured.

The welding procedure specifications (WPS) were developed for manual gas tungsten arc (GTA) welding of 304HCu SS tubes using nickel-base ER625 and ER617 as well as matching composition ER304HCu filler wires. ER617 filler wires were used for developing the WPS for manual GTA welding of Alloy617M tubes as also for dissimilar metal tubes welding of 304HCu SS to Alloy 617M.

Detailed evaluation of mechanical properties, viz. tensile, creep, fatigue, impact, quasi-static fracture and fatigue crack growth, of the 304HCu SS and Alloy 617M tube materials and their weld joints have confirmed that these properties are comparable to the internationally reported values as also the codified values in the VdTÜV (German) standard.

THEME SYMPOSIUM ON ADDITIVE MANUFACTURING

Invited talks

Session 1: Nov 12, 2014: [13:30 - 15:30]

Overview

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Hot Iso-static Pressing

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Session 2: Nov 12, 2014: [16:00 - 18:00]

Applications in Strategic Areas

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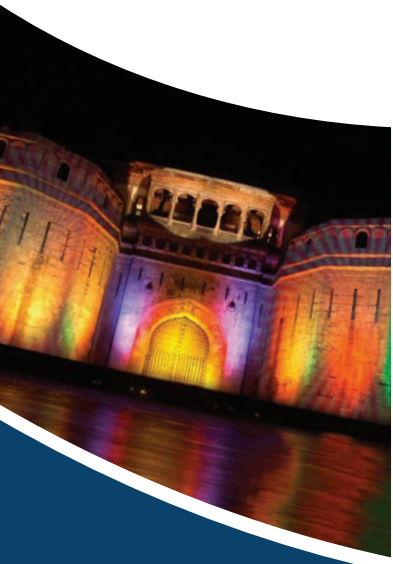

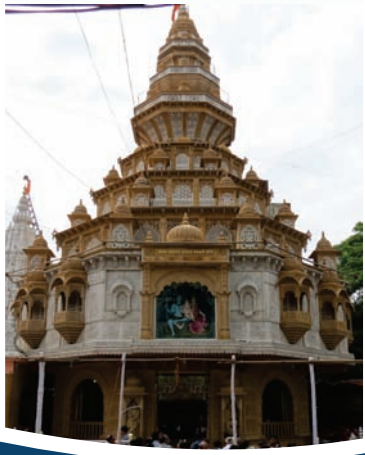

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