3rd Indo-Austrian Symposium on ‘Advances in Materials Engineering’ (AME 2016)

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Jointly organized by:

Indian Institute of Metals (IIM) Mumbai Chapter
Indian Institute of Technology Bombay (IITB)
NonFerrous Technology Development Centre (NFTDC)
3rd Indo-Austrian Symposium on 'Advances in Materials Engineering' (AME 2016)

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Book of Abstracts

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Plenary Lecture:

Materials: An Interface between Society and Science

Indranil Manna
Director, Indian Institute of Technology Kanpur, UP, India
Vice President, Indian National Academy of Engineering
President, Indian Institute of Metals
Coordinator IMPRINT-India

Corresponding author’s email id: imanna@iitk.ac.in; director@iitk.ac.in

Abstract

Man is a social being. The society we live in is an organized collection of individuals with common objectives, belief and culture. Science or scientific interventions has made huge contributions to the society throughout the ages and is principally responsible for all the advancements and differences that we see now compared to the pristine era. In the present day of knowledge based society, science is no longer only a curiosity driven act but is felt as an intrinsic necessity and urge to translate knowledge into societal benefit through engineering invention and technological innovation. If science is driven by the primary desire to learn about nature and origin of certain truth or phenomenon, engineering or technology utilizes that fundamental knowledge to convert it into a viable and useful product or process and offer tangible benefit to the society or humanity at large. In simple words, if science is ‘know-why’ and engineering is ‘know-how’, then technology is ‘know-what (sells)’. In this chain, science-engineering-technology is a perpetual triad or a closed circle with societal aspiration placed as the pivot without a definite start or end point.

In this short discourse, I intend to begin by referring to the early system of pursuing knowledge in India through ‘gurukul’ and defining the present status of our society in the context of population, economic status, opportunities, urges and targets. I shall then cite selected examples of research endeavors concerning engineering materials from our own research group at IIT Kharagpur aimed at fundamental understanding of novel scientific properties, a few engineering projects oriented towards developing innovative products/processes conducted at CSIR-CGCRI, Kolkata. Since the success rate in such endeavor is low it is worth examining what prevents all those efforts proven otherwise successful up to pilot scale, from ultimately turning into commercial product/technology.

I shall complete my commentary with a summary of the recently launched national initiative of Government of India called IMPacting Research INnovation and Technology (IMPRINT) aimed to develop translational research culture in India and accelerate economic growth and societal benefits. IMPRINT is all about pursuing the goal of translational research through collaboration between academia and industry under ten representative technology domains. IMPRINT professes to pursue the motto: million challenges, billion minds.
Plenary Lecture:

Safe Structured Biomimetic Nanotechnology-Based Materials for Technological Applications

Ille C. Gebeshuber
Institute of Applied Physics, Vienna University of Technology
Wiedner Hauptstrasse 8-10/134
1040 Wien, Austria, Europe
Corresponding author’s email id: gebeshuber@iap.tuwien.ac.at

Abstract

Making nanotechnology-based materials, structures and processes safer is a major current undertaking in the European research and development scene as well as in circles concerned with technology assessment, regulation and safety. Various organisms excel at producing structured nanomaterials with inherent functions of potential interest to materials engineers. The three examples from living Nature that are presented in the lecture comprise 1. Non-bleaching structural coloration combined with directed fluid flow and tunable wetting properties; 2. The production of metallic nanoparticles with highly conserved shape and size in plants that accumulate heavy metals in high concentration from polluted soil, wastewater and mine trails; and 3. Functionally structured metals, alloys, ceramics, polymers and composites that are built with the help of proteins at ambient conditions. A further example are species-selective mechanical pesticides based on wax crystals.

As opposed to the huge amount of different materials used in current engineering, plants, animals and microorganisms use a small set of different materials - smartly chemically altered for the related function, and greatly structurally altered - yielding functions of relevance. One of their common properties is safety for the respective organism and its ecosystem.

The talk will reveal some underlying principles of safe natural nanosystems and provide food for thought for the development of a comprehensive nanotechnology for the well-being of all.
A new tungsten free $\gamma$–$\gamma'$ Co based superalloys: A class of alloys that can rival Ni based superalloys

K.Chattopadhyay

Department of Materials Engineering, Indian Institute of Science, Bangalore 560012, India
Corresponding author’s Corresponding author’s email id id: kamanioc@gmail.com;
kamanio@materials.iisc.ernet.in

Abstract

Cobalt based superalloys that are commercially available are primarily solid solution strengthened. The first report of existence of $\gamma'$ ordered structure in Co-Al-W alloys containing $\gamma$ fcc matrix were reported by Charles Samuel Lee in 1973. These were the days of X-Ray diffraction. Exactly the same alloy was revisited by Sato and co-workers in 2006 in a paper published in Science. This led to a resurgence in the search for Co based superalloys with $\gamma$–$\gamma'$ structure. However, addition of fairly large amount of W increased the density of the alloy to an unacceptable level. Following intense effort in the last ten years, the same group came to a conclusion that ‘W’ addition is essential for developing such microstructure and this has set the entire direction for the development of the Co based superalloy with $\gamma$–$\gamma'$ structure. The unacceptable density has cast serious doubt of the future prospect of this class of alloys.

The group at Indian Institute of science have recently shown that the assumption that tungsten is essential for the development of $\gamma$–$\gamma'$ microstructure in Co based alloys is not correct and reported at least two classes of new alloys in Co-Al-Mo system with the small addition of Ta and Nb. These alloys, although at very early stage of development, provide real opportunity for developing high strength high temperature Co based super alloys. These not only overcome the drawbacks of the W containing alloys but also superior in terms of mechanical and other properties. In the present talk, we will summarise the current state of art and present some of the latest results in the development of this alloy.
Unraveling Multi-asperity Contact and Wear in Atomistic Abrasion Simulations
Stefan J. Eder, Ulrike Cihak-Bayr, Davide Bianchi, Andreas Pauschitz
AC2T research GmbH, Wiener Neustadt, Austria
Corresponding author’s Corresponding author’s email id: pauschitz@ac2t.at

Abstract

1 Introduction: Most molecular dynamics (MD) simulations treat twobody wear as a nanometric cutting process and focus on the interaction between a single abrasive particle and a usually flat substrate [1]. However, wear processes may involve circumstances such as changing rough topographies, random abrasive orientation distributions or wear debris build-up between neighbouring abrasives, which cannot be studied using the above approach. The implementation of a statistically based surface roughness in an MD model combined with multiple abrasive particles acting on the surface is therefore useful for more realistic modelling of wear phenomena [2–5]. In this way, the interactions between abrasives/asperities are explicitly included in the model, while the results, if properly evaluated, retain most of the information from the single-asperity level.

In this work, we simulate the abrasion process of an atomically rough ferrite surface with 16 hard abrasive particles implicitly bonded into a matrix using MD (see Fig. 1, left). By dynamically identifying the atoms (that are worn away from the substrate) the wear volume can be quantified and monitored in a time-resolved fashion. Moreover, every nanoscopic wear particle is affiliated with the abrasive particle by which it is produced using an iterative clustering algorithm (see Fig. 1, right). This allows a thorough analysis of the individual contributions of the abrasive particles to the wear volume.

2 Results and Conclusions: The stability of individual wear particles is influenced by:

- The initial position of the abrasive with respect to the original topography as well as the grinding angle. If the first topographic feature an abrasive encounters on its path upon striking the surface is a pronounced asperity, it will quickly produce a relatively large wear particle.

- Existence of deep pits along the trajectory of the wear-particle-producing abrasive. Especially if these pits are encountered soon after the first wear particle has formed, there is a high chance that a large portion of the wear particle will be reincorporated into the substrate there.

- Abrasive shape. Spherical abrasives do not exhibit any large surfaces consisting of crystallographic planes, so wear debris attached to them can be more easily sheared off than from the cubic abrasives, which exhibit bcc (100) planes.

- Abrasive orientation. While almost irrelevant for spherical abrasives, orientation is of high importance for the cubic abrasives as it and the grinding angle determine the rake angle,
depending on which a given abrasive will tend more towards cutting or ploughing. Certain orientations (e.g., crystallographic planes nearly parallel to the surface) may even rule out appreciable wear particle formation except at the highest normal pressures.

- Wear particle detachment. Fortuitous detachment of a wear particle from the surface may prevent subsequent re-incorporation into the substrate and thus leads to a perfectly stable wear particle

3 Acknowledgement: The authors acknowledge the support of the Province of Niederösterreich (Project “SaPPS”, WST3-T-8/028- 2014). This work was partly funded by the “Austrian COMET-Program” in the frame of K2 XTribology (project no. 849109). This work was carried out at the “Excellence Centre of Tribology” (AC2T research GmbH).

References
Applications of Engineered Carbon for Electrochemical Energy Storage

Vijayamohananan K. Pillai
Central Electrochemical Research Institute (CECRI), Karaikudi. India.
Corresponding author’s Corresponding author’s email id: vijay@cecri.res.in

Abstract

Carbon is ubiquitous for many energy related applications, like that of batteries, fuel cells and ultracapacitors. Graphene directly as an additive or indirectly after functionalization or blended with many polymers have been used for a variety of energy generation and storage applications due to their tunable solubility, bio-compatibility, unique optical and electronic properties. For instance, GQDs have been studied theoretically and experimentally for their size-dependent light emission and calculations using nitrogen doping suggest that nitrogen atoms located at their edge and near the edge promote the electrocatalytic activity towards the oxygen reduction reaction, even to replace noble metals like platinum for fuel cell applications. I will describe the usefulness of heteroatom engineered carbon either as graphene nanoribbon or graphene quantum dot many applications after discussing the mechanistic aspects of the transformation of carbon nanotubes to graphene nanoribbons (GNRs) and GQDs using spectro-electrochemical data collected by in situ experiments to unravel some of their unique size dependent features. Multifunctional properties such as electrocatalytic properties, Photoluminescence, Electroluminescence and unique single electron transfer behavior of these individual graphene quantum dots and graphene nanoribbons would be discussed with a view to facilitate their application potential in electrochemical energy storage (1-4).

References:

Keynote Lecture:

**Transmission electron microscopy, imaging and beyond**

Johannes Bernardi, Michael Stöger-Pollach, Stefan Löffler, Peter Schattschneider, Thomas Schachinger

Technische Universität Wien, University Service Centre for Transmission Electron Microscopy (USTEM), Vienna, Austria

Corresponding author’s email id: johannes.bernardi@tuwien.ac.at

**Abstract**

Transmission electron microscopy is nowadays a standard tool for imaging and structural characterization of a variety of different materials. The interaction of beam electrons with the sample allows the chemical characterisation by means of X-ray analysis and electron energy loss spectrometry (EELS). EELS extends the range of physical and structural properties that can be investigated in a TEM with high spatial resolution. At USTEM we apply this technique in order to investigate the magnetic properties of nanostructures by means of Electron Energy Loss Magnetic Chiral Dichroism (EMCD), for the investigation of optical properties with low loss and low voltage EELS but also to study binding and valence states. Examples of these applications will be given. We will further show that the transmission electron microscopes can be used to produce electron voltages, a promising technique for future applications.

Keynote Lecture:

**High Temperature Materials; Design and Assessment for Critical Applications**

G. K. Dey

Materials Group, Bhabha Atomic Research Centre (BARC), Trombay, Mumbai 400085

Corresponding author’s email id: gkdey@barc.gov.in

**Abstract**

Rapid advancements in the area of materials are paramount to scientific and technological excellence. Specially for critical applications like nuclear, space and defense, the systems performance in a reliable manner with minimum chances of failure calls for materials of stringent specifications in terms of purity and microstructure. The raw materials and their processing in to alloys first and subsequently to useful shapes calls for materials and technologies which may not be accessible due to many reasons necessitating indigenous development. The flow sheets for extraction of the base elements from Indian ores for high temperature alloys for applications in nuclear fission and fusion based reactors have been developed in Materials Group, BARC. Techniques for purification of the extracted elements and
creation of alloys by using these elements have been developed. The thermo-mechanical processes involved in giving these alloys useful shapes for different applications have been established. Extensive structure property correlation has been carried out in the case of many of these alloys. Techniques for coating of the alloys to protect these from oxidation at high temperature have been developed. This presentation gives a flavor of some of the aforementioned activities. One of the highlights of the work described here is that the entire work has been done in the country. In addition to the aforementioned, nickel based alloys of both categories, solid solution strengthened and precipitation hardened have been studied in great detail with emphasis on structure property correlation. A number of these alloys are indigenous in origin. This presentation gives a flavour of this activity also.

Keynote Lecture:

Additive Manufacturing of Biomaterials: A New Paradigm

Bikramjit Basu
Materials Research Centre,
Center for Biosystems Science and Engineering,
Center for Excellence on Biomaterials for Orthopedic and Dental Applications,
Indian Institute of Science, Bangalore 560012, India.
Corresponding author’s email id: bikram@mrc.iisc.ernet.in; bikram.iisc@gmail.com

Abstract

Over the last few decades, biomaterials science and biomedical engineering have been perceived as one of the fastest growing areas of research and innovation within the engineering science community, considering the number of scientific discoveries and their societal impact. The upsurge in the clinical demand for reconstructive joint replacements demands new implants with better biocompatibility properties. These attempts were largely directed to re-create functional musculoskeletal systems with considerable potential to treat various types of human diseases, for example, osteoarthritis, osteoporosis, osteomyelitis, so on.

In line with such efforts, this presentation will provide global perspective on the subject with a special emphasis on current efforts to address many of the Indian challenges. In reference to our ongoing multi-institutional research program on biomaterials, I will present some of our recent results to demonstrate the efficacy of the 3D powder printing to fabricate the Ti6Al4V-based metallic as well as Calcium Phosphate (CaP)-based ceramic scaffolds for bone tissue engineering applications. While presenting our research results, a major emphasis will be placed on the binder formulation, post-processing treatment, and micro-computed tomography based analysis of interconnected porous architecture. The challenges in the fabricating patient-specific implants in 3D powder printing route as well as the myriad of opportunities for future research in this emerging field will be highlighted.
This presentation will conclude with the speaker’s thoughts on translational research programs to enable young researchers to think laterally, while blending sufficient knowledge of biological systems with engineering sciences to develop next generation biomedical materials.

**Keynote Lecture:**

**Materials and Components Energy Saving in Transportation Sector**

R Gopalan

Center for Automotive Energy Materials, International Advanced Research Center for Powder Metallurgy and New Materials (ARCI), IITM-Research Park, Chennai-600113, India

Corresponding author’s email id: gopy@arci.res.in

**Abstract**

Electric Vehicles (EVs) / Hybrid Electric Vehicles (HEVS) are emerging as promising technological solution for energy saving in transportation sectors. The National Electric Mobility Mission Plan 2020 is one of the most important and ambitious initiatives undertaken by the Government of India that has the potential to bring about a transformation shift in the automotive industry of the country. The Mission aims to bring faster adoption, domestic technology development (R & D) and manufacturing of full range of cleaner electric vehicle technologies, thereby leading to creation of a strong self-sustaining EV industry in India. In this context, energy saving materials significantly draw the attention for the design of the EV vehicles. Among the energy saving materials, Li-ion battery and magnets (motors) are the critical components of the system to realize EV / HEV technology. Yet another energy delivering material is the thermoelectric material which can be designed to convert waste heat to power in hybrid electric vehicle. The thermoelectric devices placed on the exhaust of the hybrid-electric vehicle, may be utilized to generate electrical power. This electrical power may be stored in an energy storage device to deliver power to alternator or air conditioning system. In line with the Mission of India’s National Mobility Programm, ARCI, Hyderabad / Chennai has dedicated its research effort towards the following: (i) Indigenous development of Li-ion battery materials and cells technology for Electric Vehicle applications (ii) Demonstration of soft and hard magnets technologies at prototype level for motors and alternator applications (iii) Translating high efficiency Thermoelectric Materials Technology towards waste heat recovery applications In this presentation the research activities at ARCI on the above thrust programmes will be discussed.
Keynote Lecture:

Preparation and the Thermal Stability of Nanostructured Materials

Zaoli Zhang, Jinming Guo, Julian Rosalie, Reinhard Pippan
Erich Schmid Institute of Materials Science, Austrian Academy of Science, Leoben, 8700, Austria
Corresponding author’s email id: zzhang@oeaw.ac.at

Abstract

Bulk nanostructured materials show extremely high strength compared to bulk materials, and have received unprecedented attention in material research also due to its potential applications in industry. Severe plastic deformation is an emerging and quite efficient route to generate nanocrystalline materials out of normally immiscible systems.

To form stable newly designed alloys, it would be much desirable to know what the exact amount of maximum solubility via non-equilibrium extreme deformation can be achieved. This provides the essential information for designing new nanocrystalline materials for industry. Here, we use high pressure torsion to severely deform the Cu-Cr bulk material with controllable strains up to an extremely large strain of 16000. Eventually, a maximum dissolution amount of 32 wt.% Cu in Cr matrix and homogenous and stable Cu-Cr bulk nanostructure is achieved, which exhibits excellent mechanical properties.

However, the main issue on nanostructured materials is its thermal stability. In the second part of this presentation, the thermal stability of nanostructured Cr-Cu materials will be explored using modern spherical transmission electron microscopy (TEM) via simultaneous in-situ imaging and spectroscopy analysis. The evolution of the structural and chemical composition in the nanostructured materials with temperature was tracked in real-time. It demonstrates that the nanostructured materials are not only subjected to a structural change but also to an obvious chemical composition fluctuation upon annealing. The destabilization process in the nanostructured materials starts at a quite early stage. Real-time imaging and composition determining reveal the concentration changes with temperature, and allows further analyzing the dynamic behavior in nanocrystalline materials in details.
Keynote Lecture:

**Nano-Tribological and Corrosion Behavior of Ni-TiN and Ni-(Ni\textsubscript{x}Zr\textsubscript{1-x}) Nanocomposite Thin Films**

Rahul Mitra, Mukesh Kumar, and Bibhu Prasad Sahu

Department of Metallurgical and Materials Engineering, Indian Institute of Technology, Kharagpur – 721302, West Bengal

Corresponding author’s email id: rahul@metal.iitkgp.ernet.in

**Abstract**

There is a strong interest in development of nanocomposite thin films for use in protective coatings of load-bearing components subjected to various types of extreme environments. This presentation will focus on the processing-structure-property relations with emphasis on nano-tribological properties and corrosion behavior, with Ni-TiN and Ni-(Ni\textsubscript{x}Zr\textsubscript{1-x}) nanocomposite films as examples. The nano-composite thin films with Ni matrix and in-situ formed TiN dispersoids have been processed by reactive magnetron co-sputtering of Ni and Ti targets using varying negative substrate bias and environments comprising Ar+N\textsubscript{2} mixture. Formation of TiN with stoichiometric composition has been observed for substrate bias of -60 V and Ar:N\textsubscript{2} = 1:2. Further, it has been observed that the volume fraction of TiN increases with increasing negative substrate bias, or with increasing N\textsubscript{2} content of the sputtering gas mixture. Presence of TiN as reinforcement as well as alloying of Ni with Cu enhances the pitting corrosion resistance in 3.5% NaCl solution through formation of passive film of TiO\textsubscript{2}, as confirmed by X-ray photoelectron spectroscopy studies. The nanoindentation hardness and modulus of these composites have been found to similar to or higher than the rule-of-mixture prediction, and the scratch resistance has been found to scale with hardness. Similar behavior has been observed in case of the Ni-(Ni\textsubscript{x}Zr\textsubscript{1-x}) nanocomposite films, which were deposited by co-sputtering of Ni and Zr targets, and exhibit nanocrystalline or mixed amorphous+nanocrystalline structure depending on the substrate bias. The effect of crystallinity of these films on nanoindentation properties and corrosion behavior has been examined.
Keynote Lecture:

Multiscale Modeling of Lowering of Atomic Density in Si-Nanoglass and Li Intercalation in Interconnected Nano-Shells of Amorphous Si

Sudipto Ghosh¹, Arghya Bhowmik², Jaykishan Dora¹, Charchit Saraswat¹, Yelda Natraj³, Shampa Aich¹ and Jeevan Jyoti Chakraborty⁴

¹ Department of Metallurgical and Materials Engineering, IIT Kharagpur, India.
² DTU National Laboratory for Sustainable Energy, Technical University of Denmark, Frederiksborgvej 399, 4000 Roskilde, Denmark
³ Department of Metallurgical and Materials Engineering, NIT Rourkela, India.
⁴ Department of Mechanical Engineering, IIT Kharagpur, India.

Corresponding author’s email id: sudipto@metal.iitkgp.ernet.in, jay769001@gmail.com, charchitsaraswat@iitkgp.ac.in, saich@metal.iitkgp.ernet.in

Abstract

In the present work, using classical molecular dynamics simulations it has been shown that diffusion of free volume from the inter-particle void/pore plays leads to significant lowering of atomic density. The lowering is higher than the diffusion of free volume from glass-glass interface as suggested earlier [1,2]. Earlier [3], based on density functional theory (DFT), it has been shown the lowering of atomic density significantly lowers the intercalation expansion coefficient. In the present study by feeding the expansion coefficient to a continuum deformation model the significant drop in stresses as a result of density lowering has been shown.

References:

1. H. Gleiter, Our thoughts are ours, their ends none of our own: Are there ways to synthesize materials beyond the limitations of today?, Acta Materialia 56 (2008) 5875-5893.
Keynote Lecture:

Lithium ion Batteries and Beyond
S.Gopukumar

Electrochemical Power Systems Division, Central Electrochemical Research Institute (CSIR), Karaikudi – 630006, TamilNadu, India
Corresponding author’s email id: gopukumar@cecri.res.in

Abstract

The lithium ion battery has evolved as the major power source ever since it’s discovery in 1991 by Sony and represents one of the major successes of materials electrochemistry. Lithium ion batteries are becoming more and more popular in view of the multifarious applications arising from their high voltage and high power leading to light weight and smaller size cells/batteries. In view of the growing day to day demand for lithium ion batteries, intensive research is being pursued globally to develop new high performing cost effective electrode and electrolyte materials and importantly without compromising on environmental issues.

In my talk, I shall give you an overview of the recent developments in the area of cathode and anode materials for lithium - ion cells. Details regarding the synthesis and characterization of high voltage (up to 5V) cathode materials based on layered and olivine materials shall be discussed. Normally, graphite powders are employed as anode materials but we demonstrate a new type of lithium ion battery replacing the conventional graphite powder coated on to a copper foil exhibiting high performance even at high rates (2C) when cycled between 2.9 and 4.1V and finds applications in solar lanterns, solar hats etc. These cells could be charged by solar energy. An insight into future sodium-ion cells shall also be presented.

Keynote Lecture:

Improving Toughness of High Speed Steel Laser Hardfacings by Alloying with Niobium
M. Rodríguez Ripoll1, N. Ojala2, C. Katsich1, V. Totolin1, C. Tomastik1, K. Hradil3

1AC2T research GmbH, Wiener Neustadt, Austria
2Tampere University of Technology, Department of Material Science, Tampere Wear Center, Tampere, Finland
3Technische Universität Wien, X-ray centre, Vienna, Austria
Corresponding author’s email id: ripoll@ac2t.at

Abstract

I Introduction

The use of components in highly demanding applications requires the design of new materials that are able to fulfil the increasing demands in terms of high strength, toughness and wear
resistance. Within this context, hardfacing by laser has become one of the most efficient techniques for developing wear resistant coatings thanks to its high deposition rate and efficiency. By using laser hardfacing, metal powder can be welded on the top surface of inexpensive components to enhance its operational performance. In order to increase fracture toughness, doping elements such as niobium were recently successfully added in nickel-based laser hardfacings. However, the role of niobium as alloying element in high speed steels has been seldom researched. Hence, a systematic investigation on the impact of Nb concentration on the mechanical properties of high speed steels is lacking, especially in case of hardfacings. The aim of this work is to shed light on the role of Nb as alloying element on the wear and corrosion properties of laser hardfacings using Nb concentrations up to 3 wt.% [1].

2 Results
The present work shows the impact of niobium content on the microstructure of high speed steel hardfacings and their resulting mechanical and chemical properties. Under the absence of niobium, the hardfacings have a well-known microstructure characterised by the presence of fine carbide networks surrounding the martensitic dendrites. This microstructure is responsible for providing HSS hardfacings with high hardness and excellent wear resistance. However, the toughness of laser hardfacings is sometimes not adequate due to the high cooling rates undergone during the deposition process, which result in a high cracking susceptibility and an excess of brittleness in operation. A first impact of niobium on the microstructure of HSS hardfacings is on dendrite arm spacing refinement. It was observed that alloying with niobium even for concentrations up to 1% is sufficient to reduce dendrite arm spacing up to 36 %, while maintaining hardness of the undoped hardfacing. Niobium-doped hardfacings also have intergranular carbides with a smaller width. When having niobium as doping element, the amount of available carbon for Mo, W, Cr and V carbide formation is reduced, due to the higher affinity of niobium for carbon. A third factor contributing to the improvement of the toughness of the HSS laser hardfacings was found by nanoindentation. The elastic modulus of the dendrites is lower for higher Nb contents, which benefits the elastic strain to failure of the hardfacing. As a consequence of this microstructure, a higher Nb content results in an improved abrasive wear resistance. Up to Nb concentrations of 1 wt.%, abrasion resistance improves by over a factor two, despite all hardfacings having the same hardness (Fig. 1).

3 Conclusions
• Niobium content has an impact on the hardfacings by reducing their dendrite arm spacing, reducing carbide content and increasing elastic strain to failure.
• These microstructural changes result in an improvement of the hardfacing toughness, while maintaining the hardness for Nb contents up to 1 wt.%.
• These enhanced mechanical and chemical properties result in an improvement of low stress abrasive wear and corrosion resistance.
Fig. 1: Hardness and abrasive wear rate of HSS laser hardfacings as a function of the niobium content.

Acknowledgement
This work was funded by the “Austrian COMET-Program” in the frame of K2 XTribology (project no. 849109). The research work was carried out at the “Excellence Centre of Tribology” (AC2T research GmbH). The work at Tampere University of Technology has been done within the FIMECC BSA (Breakthrough Steels and Applications) program.

Reference

Keynote Lecture:

Fretting Mechanisms under Controlled Environment Conditions

V. Chaudhry¹ and Satish V. Kailas²

¹Nuclear Power Corporation of India Limited, Mumbai 400094, India
²Department of Mechanical Engineering, Indian Institute of Science, Bangalore 560012, India

Abstract
Fretting is of a serious concern in many industrial components for the safe and reliable operation. Small amplitude oscillations under constant or variable normal loads induce surface degradation in the form of surface cracks and/or surface wear. Researchers had investigated fretting damage and its characterization under ambient condition, but limited work has been carried out under vacuum and high temperature conditions. Comprehensive experimental studies have been carried
out simulating different fretting regimes with stainless steel spheres on a stainless steel flats, and stainless steel spheres against chromium carbide coatings under controlled environment conditions. Plasma spray and high-velocity oxy-fuel processes have been opted for coating the stainless steel surface. Damage in stainless steel has been observed in the form of material transfer, nucleation/initiation of the cracks at the surface and sub-surface regions, and formation of the metal fragments, whereas, damage in the coatings have been observed in the form of brittle fracture and spalling. Mechanisms associated with the observed damage have been quantified based on different failure modes. Adhesion at the contact interface and instantaneous cohesive strength of the contacting bodies dictates the occurrence of material transfer. Severe plastic deformation, plastic cycling and ratcheting have been observed as the modes for crack nucleation/initiation under cyclic loading condition. Further, propagation of the cracks has been observed under fatigue and their orientation has been observed to be governed by the contact conditions prevailing at the contact interface. Under high temperature, change in the damage mechanism from fatigue to metal flow has been observed. Further, chromium carbide with 25% nickel chrome binder coatings using high-velocity oxy-fuel process on stainless steel show less fretting damage, and can be considered as an effective palliative against fretting damage, even under high vacuum conditions.

*Keywords:* fretting, cracks, wear, coatings, adhesion, damage
Invited Lecture:

**Material Characterization through Ultrasound Resonant Cavity Technique**

Megha Agrawal\(^1\), Ashwin A Seshia\(^2\)

\(^1\) University of Cambridge and Imperial College London  
\(^2\) Department of Engineering, University of Cambridge  
Corresponding author’s email id: agrawal.megha@gmail.com

**Abstract**

The acoustic signature produced by sound waves interacting with a medium can be used to determine the mechanical properties of the medium non-destructively. While high frequency ultrasonic waves have been employed for non-invasive medical imaging, these techniques have received far less attention in other domains. When acoustic wave travels through multiple media, a resonant- cavity interferometry pattern just like standing waves is created. These patterns have been studied in this paper analytically in the acoustic transmission magnitude and phase response. The analytical model was based on transmission coefficient matrix (T-matrix) and modelled on MATLAB. A finite-element model simulation was also performed using COMSOL. The theoretical and simulation response provided an idea of the acoustic pressure distribution and thereby the transmission response received by the receiving transducer at the other end of the sample. Experimental validation was done by immersing 10 MHz ultrasonic transducers in a water bath and studied the samples placed in the path of innsonnication. A good match between the analytical theoretical response, literature values and the experimental results had been observed. Further, characterisation techniques based on acoustic swept frequency broadband spectroscopy were quantified to predict mechanical properties of the samples like density and speed of sound. Such a system can provide numerous advantages as a quantifiable, cheap, quick and non-destructive material characterization as compared to the other destructive and ionizing radiation based methods.
Invited Lecture:

**Green Synthesis and Stability of Pristine Free Standing Metal Nanoparticles by Cryomilling**

Krishanu Biswas, Nirmal Kumar

Department of Material Science and Engineering Indian Institute of Technology Kanpur

Corresponding author’s email id: kbiswas@iitk.ac.in

**Abstract**

Nano materials have received great attention due to their wonder properties at nano regime. Many methods came in lime light in last few decade due to increasing demand of nano materials as well as alert to protect our environment from toxic substances. The widely accepted syntheses for easily preparation of metal nanoparticles are chemical routes in which hazard chemicals being used. Recently, researcher have serious attempts to develop the eco-friendly method to prepare nanoparticles. In the concern of environment, green synthesis of metal nanoparticles are highly desirable. In this paper green synthesis of pristine free standing metal nanoparticles (Ag, AL, Cu, Fe, Zn etc.) has been synthesised by sophisticated method cryomilling. This cryomilling is cost effective rapid and mechanical method to prepare metal nanoparticles in which the powder were milled below temperature -150±10 °C under argon gas environment. The 5-8 nm particles size was achieved after milling 6 hours 30 minutes, which are found to be highly stable at higher temperature. The most of the methods to prepare Ag nanoparticles are wet chemical routes in which, hazards chemicals being used and mixed with surfactant for stabilization of the particles. The presences of surfactant sacrifice the purity and the native property of the particles. In the cryomilling, possible contaminations during synthesis such as oxidation of particles surface, nitridation, and debris from milling tool have been estimated by EPMA and ICP-OES analysis. The Ag metal nanopowder was characterized by X-ray diffraction, scanning electron microscope, transmission electron microscope to confirm composition morphology and size of the particles.

To get smaller particles size along with virgin surface, the Cu, Al, Zn, Fe nanoparticles has been successfully synthesized by cryomilling. The mechanism behind early stage refinement, cold welding, and free standing stability has been extensively studied. The particles size was investigated using TEM (Transmission electron microscopy). Free standing suspension of metal nanoparticles stability has been studies through zeta potential measurement. The results show the formation of nanosized (6-12 nm) free standing nanoparticles during cryomilling. The stability and mechanism of free standing detailed described by experimentally as well theoretically.
Invited Lecture:

Beyond Bragg Reflections - X-ray Diffraction Methods for the Materials’ Investigation of Correlated Disorder Effects

Klaudia Hradil¹, Philipp Hans¹, Reinhard B. Neder²

¹ X-ray center, Vienna University of Technology, Getreidemarkt 9, 1060 Vienna, Austria
² Friedrich-Alexander Universität Erlangen-Nürnberg, Crystallography and Structural Physics, Staudtstr. 3, 91058 Erlangen, Germany

Corresponding author’s email id: klaudia.hradil@tuwien.ac.at

Abstract

The information about the atomic arrangements in crystalline solids and therefore the knowledge of the symmetry is an essential aspect towards understanding the physical and/or chemical properties of the investigated material. In many cases deviations from the ideal atomic arrangement of the near neighbor environment within a few interatomic distances up to the medium range ordering (few nanometers to micrometers) have a significant influence on the chemical and physical properties of materials. Therefore, the precise knowledge of the short range as well as the medium range order is essential to judge the functionality or dedicated properties of materials.

Diffraction techniques together with the various applications are one of the most effective methods to investigate the microstructure of the crystalline parts on different length scales. Quite often mainly the standard methods are applied to characterize the crystallographic properties taking into account the sharp diffraction phenomena and neglecting the disorder scattering within the diffraction diagram. Hence, such investigation provides an averaged structural picture without the local arrangement. Within the contribution we will give an overview on effects of disorder within a material concerning the diffraction diagram and discuss additional methods to analyze the additional diffraction phenomenon.

Invited Lecture:

Frontiers in 3D X-ray Microscopy and Multi-scale Characterization

Hrishikesh Bale

Carl Zeiss X-ray Microscopy, 4385 Hopyard Rd, Pleasanton, CA 94588
Corresponding author’s email id: hrishikesh.bale@zeiss.com

Abstract

Tomographic imaging has opened several new research pathways in Materials Science in recent years. This talk will present an overview of the multi-scale 3D imaging, in particular the
emergence of high-resolution X-ray microscopy (XRM) in the laboratory. Once exclusively in the domain of advanced synchrotron facilities, XRM has rapidly become a common technique in the laboratory setting, combining absorption, phase and diffraction contrast imaging capabilities, spanning both micron- and Nano scale resolutions. The characterization and quantification of microstructure at multiple length scales using a novel correlative imaging approach to study processing or failure mechanisms in an aluminum alloy will be presented, demonstrating how XRM plays a key role alongside other common characterization modalities, such as light, electron and ion microscopy.

CON 1

Effect of 5-7-7-5 Defects on Mechanical Behaviour of Graphene under Tensile and Compressive Loading: A Molecular Dynamics Study
G. Rajasekaran, Avinash Parashar
Mechanical & Industrial Engineering Department, Indian Institute of Technology (IIT) Roorkee, Roorkee-247667, India.
Corresponding author’s email id: rajasekaran.ed@gmail.com

Abstract

One atom-thick sheet of carbon exhibits outstanding mechanical properties in its pristine form but topological defects which are inevitable in graphene due to its production techniques can alter its structural properties. These defects in graphene are introduced either during the production process or intentionally by us to tailor its properties. This research paper discussing about the performance enhancement of graphene by introducing pentagon-heptagon-heptagon-pentagon (5-7-7-5) defects. The influence of geometrical parameters such as nearest neighbour distance and angular orientation between 5-7-7-5 defects on the mechanical behaviour of graphene was investigated in the frame of molecular dynamics. Mechanical properties and failure morphology of graphene under tensile loading was predicted to be the function of geometrical parameters between 5-7-7-5 defects. It has been predicted from the current study that brittle behaviour of graphene can be modified to ductile with well controlled distribution of 5-7-7-5 defects under tensile loading. Also it has been predicted that mechanical properties of graphene under tensile loading can be altered by proper distribution of 5-7-7-5 defects. In this paper, authors were further investigated the influence of 5-7-7-5 defects of graphene under compressive loading. High compressive strength predicted for graphene sheet with 5-7-7-5 defects was attributed to bond realignment and its ability to dissipate energy in the vicinity of defect. The interesting compressive properties observed in graphene also provides a structural basis to enable and advance new branches in future nanoelectronic, nanomechanical applications.
Keywords: graphene, Stone-Thrower-Wales defect, nearest neighbour distance, angular orientation, mechanical properties, Young’s modulus and failure morphology.

CON 2

Visible Light Photoreduction of Cr(VI) over TiO$_2$–Reduced Graphene Oxide Nanocomposites

Priyabrata Mohapatra$^1$, Aasiya Shaikh$^2$, Shailendra Prasad Mishra$^1$, Smrutiranjan Parida$^2$

$^1$ Department of Chemistry, C.V. Raman College of Engineering, Bhubaneswar, India- 752054
$^2$ Metallurgical Engineering and Materials Science, I.I.T. Bombay, Mumbai, India-400076

Corresponding author’s email id: priyabrat.mohapatra@cvrce.edu.in, priyabratm@gmail.com

Abstract

Hexavalent chromium, Cr (VI) is a carcinogenic heavy metal environmental pollutant, where as the trivalent chromium, Cr(III) is a nontoxic oxidation state of the heavy metal, which is less mobile and can be easily separated by precipitation. There are various methods available for the reduction of Cr (VI) to Cr (III). One of the process is the natural process of reduction of Cr(VI) which is extremely slow and requires special environmental conditions, the other method is the chemical reduction of Cr(VI), however chemicals used in the chemical reduction process are harsh and cause secondary pollution. Therefore, there is need of an efficient remediation method for Cr(VI) need to be explored. Photoreduction is one of the remediation methods of the hexavalent chromium Cr(VI). TiO$_2$–xRGO composite catalysts were synthesized using solvothermal method. TiO$_2$ particles in the size range of 4 – 9 nm were formed on the reduced graphene sheets. The composite catalysts have large fluorescence quenching, indicating an efficient separation of photogenerated electrons and holes. The composites displayed excellent photoreduction of Cr(VI) in the visible light, which was found to be a function of the weight percentage of RGO in the composite. The performance degraded with increasing RGO content in the composite, which can be attributed to the higher electron-hole recombination on the RGO surface.
Role of Noise Level, Nonstationarities and Nonlinearities in Reliable Impedance Measurements by Odd Random Phase Multisine Signal Impedance Spectroscopy

Gopal JI, Herman Terryn
Research Group Electrochemical and Surface Engineering, Department of Materials and Chemistry, Vrije University of Brussels, Pleinlaan 2, 1050, Elsene, Brussels, Belgium
Corresponding author’s email id: gopal.ji.ji@gmail.com

Abstract

Single sine electrochemical impedance spectroscopy (EIS) is a very popular tool to study the corrosion behavior of various types of metallic and organic coatings in different electrolytes. In spite of having excellent ability to investigate the nature of corrosion reactions, single sine EIS suffers from longer measurement times and reliability of impedance measurements. The reliability corresponds to the accuracy of estimating the element’s values in an equivalent electrochemical circuit, which is not possible without taking different noise distortions into account. Hence the detection of noise level, nonstationarities and nonlinearities are of great importance; which is extremely difficult for single sine EIS.

In this study, we have investigated the role of noise level, nonlinearities and nonstationarities in reliability of impedance measurements through investigation of corrosion behavior of hot dip galvanized steel in 0.1 M NaCl by odd random phase multisine signal impedance spectroscopy (ORP-EIS). The reliability is also discussed on the basis of residual noise and relative error of the measurements.

Keyword: ORP- EIS, Multisine, Nonlinearity, Nonstationarity, Galvanized steel, Hot Dip.

Excellent Microwave Absorbing and Magnetic Properties exhibited by NiCoZnFerrite Nano Particles

Ravindra N Kambale¹, Lalit C Borde², Vaishali Bambole¹

¹ Dept. of Physics, University of Mumbai, Mumbai, India
² Tata Institute of Fundamental Research (TIFR), Mumbai, India
Corresponding author’s email id: vabphy@gmail.com

Abstract

Microwave absorbing material play a great role in electromagnetic pollution controlling, electromagnetic interference (EMI) shielding etc. Thus the electromagnetic compatibility (EMC)
and electromagnetic interference (EMI) are becoming a serious problem and much attention has been paid towards finding suitable microwave absorber to solve this problem. Extensive studies have been carried out to develop new and high efficient absorbents specially the ferrites like Mn Zn ferrite, Mg-Er ferrite, Ni-Zn ferrite, Mn ferrite etc. We have been successful in synthesizing nanoparticles of spinel ferrites like NiCoZn which is one of the most versatile technological material especially suited to high frequency microwave absorption.

Spinel structure NiCoZn (Ni0.3 Co0.2 Zn0.5 Fe2 O4) have been synthesized by sol- gel auto combustion technique. The structural, Morphological and magnetic properties at room temperature of the product were characterized by X- Ray Diffraction (XRD), Field emission scanning electron microscopy (FESEM), Field emission gun transmission electron microscopy (FEGTEM) and Vibrating Sample Magnetometer (VSM). X- ray patterns confirmed the formation of single phase cubic structure. The crystallite size of synthesized ferrite nanoparticle is within the range of 8 -18 nm. The microwave absorbing properties of ferrite pellet of thickness 1 mm was investigated by using Vector Network Analyzer in the frequency range 20 to 30 GHz. A maximum reflection loss of -17.626 dB was absorbed at 22.42 GHz. The MW absorbing properties increase drastically by forming a nanocomposite with conducting polymer. The results of the nanocomposites of NiCoZn with polyaniline will also be discussed in the paper.

Keywords: Spinel structure, ferrite nanoparticle, conducting polymer magnetic nanocomposites, microwave absorbing property, reflection loss.

CON 5

Effect of Zr Contents and Substrate Bias Voltage on Microstructure and Properties of DC Magnetron Sputtered Ni-Zr Thin Films

Bibhu Prasad Sahu, Rahul Mitra

Department of Metallurgical and Materials Engineering, Indian Institute of Technology Kharagpur, West Bengal, India

Corresponding author’s email id: bibhu.igit@iitkgp.ac.in

Abstract

Studies on Ni-Zr alloy thin films have attracted interest for possibility of developing amorphous or nanocrystalline structures with applications in fuel cells, stressed atomic force microscopy probes, nuclear reactor components as well as for hydrogen storage and separation. This study reports the relationship between the microstructure evolution and properties by varying the Zr contents and negative substrate bias voltage. The formation of the nano-intermetallic phases (Ni5Zr, Ni3Zr) dispersed in Ni matrix by dc magnetron co-sputtering of high purity Ni and Zr targets at ambient temperature have been observed at low Zr contents and unbiased film. However, increasing Zr contents and negative substrate bias voltage, the transition from
nanocrystalline to amorphous structure have been achieved. Contact type surface profilometry and atomic force microscopy tests have been carried out to measure the film thicknesses and surface roughness, respectively, whereas the phases present have been identified using X-ray diffraction, their microstructures have been examined using scanning electron microscopy and transmission electron microscopy on cross-section samples. Furthermore, hardness, crack-resistance and constant strain rate (CSR) measurements have been conducted using the nanoindenter on various as-deposited films and compared with related to Zr contents, negative bias voltage, grain size and processing conditions. The mechanisms governing the relation between properties and microstructures of the Ni-Zr films will be discussed.

Keywords: Thin film, magnetron sputtering, bias voltage, Zr contents.

CON 6

Study of Vacuum on Thermal Insulation of Stainless Steel Vacuum Flask

Shivam Trivedi

Department of Metallurgical and Materials Engineering, Maharaja Sayaji Rao University of Baroda, Vadodara, India

Corresponding author’s email id: shivamtri97@gmail.com

Abstract

This research paper will study the performance of stainless steel vacuum flasks under different vacuum levels for excellent temperature retention. A stainless steel vacuum flask is used domestically to keep fluid hot or cold for extended periods of time and for many purposes in industry. It comprises inner and outer bottles made of stainless steel, the inner and outer bottles being joined together at tip portions of their bottle necks to form a double-walled construction with a space between the two bottles, the space being vacuumized. Heat losses through mode of heat transfer can be minimized by creating and maintaining optimum level of vacuum between the two layers of vacuum flask. The stainless steel vacuum flasks are sealed after attaining the required vacuum. This research study also explores effective vacuum sealing of the vacuum flasks. This includes study of heat transfer rate with respect to attained vacuum level, physical properties of the sealing material such as adsorption, absorption and chemical binding. The study is based on experimentation, which is carried on high vacuum systems. In high vacuum systems, the chamber is vacuumized using a rotary pump in accordance with a holding pump and diffusion pump maintained by a high vacuum valve. The central idea behind this project is to study the current system used in the vacuum flasks and hence propose an improved version of flask which is more effective and sustainable. This research will hence increase the potency of vacuum flask temperature withholding. This research paper works on two proposals: first, optimum vacuum level to be maintained within the two layers of the flask and second, appropriate material to be used for efficient vacuum sealing. Cost efficiency and a high caliber
product for large scale production will be the giving of this research project. These methods will transcend the currently used stainless steel vacuum flasks. This research study is hence a comparative analysis between rate of heat transfer and vacuum level to be maintained. It also deals with the efficiency of sealing the stainless steel vacuum flasks.

Keywords: Vacuum, Rotary Pump, Diffusion Pump

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**CON 7**

**Modelling of Crystalline Behaviors of PP/Coir and PP/EPDM using Artificial Neural Networks**

Shivani Lodha, D. Purnima

Department of Chemical Engineering, BITS-Pilani, Hyderabad Campus, Hyderabad, Telangana, India 500078.

Corresponding author’s email id: dpurnima@hyderabad.bits-pilani.ac.in

**Abstract**

Polypropylene (PP) is a low density, thermoplastic polymer which is widely used in many applications such as textiles, packaging, laboratory equipments, and plastic parts. It has good flexural strength, low coefficient of friction, and high resistance to chemicals. Composites of PP such as PP/coir and PP blend such as PP/EPDM (Ethylene-propylene-diene-copolymer) are used so as to improve the impact strength of the PP, and also to reduce the cost of the product. Processing conditions can affect the mechanical properties of the composites/ blend. If it is not optimized properly, there can be deformations in the composites/blend. The study of crystalline behaviour of the composites/blend is thus very important in optimizing the processing conditions. In this work artificial neural network is used to predict the crystalline behaviours of PP/coir composite and PP/EPDM blend. Artificial neural networks are structured in such a way that they resemble and work in the same way as the human nervous system. They are becoming very popular in the prediction of various properties of polymer composites because of their good prediction quality. The predicted results are compared with the experimental values which are obtained with DSC (Differential Scanning Calorimetry) with 10°C per minute heating rate in nitrogen atmosphere.

Keywords: PP, Coir, EPDM, Composite, Blend, Artificial Neural Network, DSC.
Characterization of PP Based Blends and Composites
Nitisha Mittal, D. Purnima
Department of Chemical Engineering, BITS-Pilani, Hyderabad Campus, Hyderabad, Telangana, India 500078.
Corresponding author’s email id: f2014408@hyderabad.bits-pilani.ac.in

Abstract
Polypropylene is a thermoplastic polymer having low density, partially crystalline and has service temperature of 130°C. It is a versatile material with a wide variety of applications such as packaging, labeling, textiles, stationary and more. Different compositions of polypropylene based blend/composite are taken and its thermal properties have been analyzed by DSC and TGA. Blend/composite of polypropylene like PP/EPDM and PP/COIR is subjected to a temperature range of 35°C to 350°C. With DSC various properties such as melting point, crystallization temperature, rate of nucleation, % crystallinity were obtained. With TGA, temperatures for 25%, 50%, 75% decomposition and % residue were obtained. From the result the comparison on effect of composition of coir or EPDM on crystallinity and degradation temperature of PP were elucidated.

Keywords: DSC, TGA, Polypropylene, Thermal Properties

PI

Study of Tensile and Flexural Strengths of Cocoa and Opuntia Fibre Reinforced Hybrid Composites
V. Navya Geethika, V. Durga Prasada Rao
Department of Mechanical Engineering, SRKR Engineering College, Bhimavaram
e-mail ID: vng.mini@gmail.com

Abstract
In the growing environmental problems, natural fibres seem to be a good alternative since they are abundantly available and there are a number of possibilities to use all the components of fiber-yielding crop; such fiber-yielding plants are Cocoa and Prickly pear (Opuntia). These fibers are characterized by low density, high tenacity and high moisture absorbency in comparison with other fibres. Thus these can be used in manufacturing of hybrid natural composites. In the present work, Cocoa and Opuntia fibre reinforced hybrid composites are produced through hand Lay- up technique. These fibres were treated with 7% NaOH (alkali treatment) and cured for 35 minutes and dried for better fibre matrix adhesion, bending and tensile properties.
The objective of the present work is to investigate the tensile and flexural properties of four varieties of hybrid composites. The composites include Cocoa fibre-Copper foil composite (F1-Cu-F1-Cu-F1), Opuntia Fibre-Copper Foil Composites (F2-Cu-F2-Cu-F2), Cocoa Fibre-Copper foil-Opuntia fibre composite (F1-Cu-F2-Cu-F1), and Opuntia Fibre-Copper foil-Cocoa fibre composites (F2-Cu-F1-Cu-F2). The number of plies of fibre taken in each type of composite are three, and the total number of plies used in each hybrid composite are five. Each of these composites is prepared in 00 orientations (fibres in parallel direction), 450 (fibres at 450 to each other) and 900 orientations (fibres in perpendicular direction).

It is observed from the results that, the tensile strength of F1-Cu-F1-Cu-F1 composite with 00 orientation is high (113.60 MPa) and that of F2-Cu-F2-Cu-F2 composite with 900 orientation is low (30.53 MPa), and it is also observed from the results that, the bending strength of F1-Cu-F1-Cu-F1 composite with 900 orientation is high (31.06 MPa) and that of F2-Cu-F2-Cu-F2 composite with 900 orientation is low (26.66 MPa). Further it is noticed that, except for the F2-Cu-F2-Cu-F2 composite, the tensile strength of all the composites with 0° orientation is high compared to their 45° and 90° orientations. The tensile strength of all the four composites with 90° orientation is found to be low compared to their remaining orientations. It is also noticed that, except for the F2-Cu-F2-Cu-F2 composite, the bending strength of all the composites with 90° orientation is high compared to their 0° and 45° orientations.

Keywords: Fibre, hybrid composites, Cocoa, Opuntia, Tensile, Bending strength

PII

Effects of Swift Heavy Ion irradiation on Electrical Characteristics of Pt/n-GaN Schottky diodes

J.V.V.N.Kesava Rao¹, K.Asokan², R.Padma Suvarna¹

¹ Department of Physics, JNTUA, Ananthapuramu, A.P, India
² Inter University Accelerator Centre, Aruna Asif Ai Marg, P.O Box-10502, New Delhi-110067 , India

Corresponding author’s email id: kesava2007@gmail.com

Abstract

GaN based devices have been investigated for different applications like blue, ultraviolet LED, solar detectors, MESFET, HEMT etc. Metal-Semiconductor (M-S) interfaces form an important research tool for characterization of materials. Studies of high energy ion beam are useful for modification of semiconductor devices in irradiation environments.

Schottky barrier diodes (SBD) of circular area in 2mm diameter were fabricated by depositing 30nm thickness of Pt/n-GaN using electron beam deposition in target laboratory at IUAC, New Delhi. Pt-metal layer semiconductor diode of Pt/n-GaN is subjected to irradiation of Swift Heavy Ion (SHI) beam with incremental varying ion fluence.
Using I-V and C-V electrical characterizations diode parameters like ideality factor, Schottky barrier height (SBH), forward current and reverse leakage current are determined.

Key words: Platinum, GaN, ion beam, Schottky diodes, Electrical Characterization.

PIII

Synthesis, Structural, Electrical & Thermal Properties of Multifunctional Bi(1-x)Nd(x)FeO3 (x= 0.00 & 0.25) Ceramic

Vikash Kumar Jha, M. Roy

Department of Physics, Mohanlal Sukhadia University, Udaipur, India
Corresponding author’s email id: vkJha1988@yahoo.com

Abstract

Multifunctional ceramic samples of Bi(1-x)Nd(x)FeO3 (x= 0.00 & 0.25) have been prepared by standard high temperature solid state reaction method using high purity oxides. The prepared materials have been analyzed by room temperature x-ray diffraction method and structural parameters were refined by the Rietveld analysis. A good agreement between observed and calculated x-ray diffraction pattern was obtained from the Rietveld refinement using non-centrosymmetric space group R3c. The dielectric constant and dielectric loss as a function of frequency and temperature have been measured in the frequency range from 1kHz to 1MHz. The ac conductivity has been calculated by Arrhenius relation \( \sigma = \sigma_0 \exp\left(\frac{-E_a}{Kt}\right) \). The dc conductivity of material has been measured as a function of temperature from RT to 603K and its activation energies are calculated. The specific heat, heat loss, heat flow and other thermal parameters of the compounds have been analyzed by STA-6000 (DTA/TGA). The results are discussed in detail.

Keywords: Multifunctional; X-ray Diffraction; Dielectric; Specific heat
Characterization of Tb-Fe Thin Films having Out-of-Plane Magnetic Anisotropy

Himalay Basumatary\textsuperscript{1,2}, J. Arout Chelvane\textsuperscript{1}, S.V. Kamat\textsuperscript{1} and Rajeev Ranjan\textsuperscript{2}

\textsuperscript{1}Defence Metallurgical Research Laboratory, Hyderabad, India
\textsuperscript{2}Indian Institute of Science, Bangalore, India

Corresponding author’s email id: himab4@gmail.com

Abstract

Tb-Fe films were deposited on Si (001) substrates at room temperature with different thicknesses \textit{viz.}, 50, 100, 200, 300, 400, 600 & 800 nm by electron-beam evaporation technique. Scanning electron microscopy studies indicated presence of uniformly distributed globular islands for all the films whose size increased with increase in film thickness. Structural studies employing X-ray diffraction technique showed that the films were amorphous in nature irrespective of film thickness which were corroborated by cross-section transmission electron microscopy. Interestingly, presence of columnar structure in the form of channels with few nanometers width running from top to bottom of the film perpendicular to the silicon substrate were observed for all the films. Magnetization carried out along the in-plane and out-of-plane direction displayed presence of strong perpendicular magnetic anisotropy for all the films. Coercivity of the films deduced from the out-of-plane magnetization curves increased with increase in film thickness up to 400 nm. However, further increase in film thickness (> 400 nm) led to decrease in coercivity. A large coercivity, as high as 20 kOe was achieved for the 400 nm thick film. The nature of variation of coercivity with film thickness was correlated with the magnetic domain patterns obtained through magnetic force microscopy technique. The magnetization reversal was attributed to domain wall pinning in all the films.
THEME: Materials for Structural Applications

Invited Lecture:

Micromechanisms for Tribology of Advanced Ceramics
B. Venkata Manoj Kumar
Department of Metallurgical and Materials Engineering, IIT Roorkee, India
Corresponding author’s email id: manojfmt@iitr.ac.in

Abstract
Advanced ceramics are being used for several engineering applications where surface contacts are subjected to varieties of tribological conditions. Moreover, the physics of friction and degradation mechanisms are highly dependent on microstructure of the selected ceramic system, which in turn is effected by processing conditions. A thorough understanding on the effect of processing conditions, microstructure and mechanical properties of ceramics is therefore essential for estimating potential of advanced ceramics for use in tribological applications. In the present talk, salient results on tribological studies of microstructurally varied advanced ceramic systems: (i) silicon carbide and (ii) zirconium diboride are provided. For the first study, hot pressed silicon carbide ceramics are subjected to wear in sliding wear conditions. The friction and wear results are explained as function of type and amount of sintering additive, sliding load and counterbody. For the other study, zirconium diboride-silicon carbide ceramic composites are prepared by spark plasma sintering, and eroded by silicon carbide particles at 800°C. The micromechanisms responsible for material removal for the investigated ceramics in wear conditions are particularly elucidated as function of material, testing and environmental parameters.

Invited Lecture:

Microstructure Evolution and Deformation of Severe Plastically Deformed Lamellar Cu-Ag Metallic Composite
Pradipta Ghosh¹, Karoline Kormout¹,², Jozef Keckes¹,², Reinhard Pippan¹
¹Erich Schmid Institute of Material Science, 8700 Leoben, Austria
²Montanuniversität, 8700 Leoben, Austria
Corresponding author’s email id: Pradipta.Ghosh@oeaw.ac.at

Abstract
Nanocrystalline (nc) materials have become popular in recent times due to their enhanced spectrum of properties such as high strength, high radiation damage, strength, etc. Of the various
available techniques for synthesis of nc materials severe plastic deformation (SPD) is most attractive due to its capability for producing bulk and impurity free products. However, studies over past few decades have shown that the grain size in monolithic pure metals usually saturates at 50-200 nm after a certain applied strain, depending on temperature and strain path [1]. In addition, monolithic nc metals exhibit poor ductility and thermal stability. One way out of this constraint is to use immiscible bimetallic systems, where the size of individual phases can be reduced to few tens of nanometers and below [2]. Interestingly, SPD can also cause forced mixing in generally immiscible systems [3,4].

In this study, Cu-Ag nanocomposites are synthesized by High Pressure Torsion (HPT) of elemental powders (Cu,Ag) and cast Cu-Ag eutectic. The microstructure (SEM, TEM) and X-ray diffraction analysis of deformed composites show gradual refinement of Cu and Ag phase in lamellar regions. At larger strains, shear bands appear in thin lamellas and lead to chemical mixing for Cu-Ag powder composites. However, for cast eutectic alloys, no shear bands are observed at similar level of microstructure refinement. The possible reasons for shear band formation and methods for enhancing ductility in these lamellar nanocomposites are further investigated with compression tests.

**Keywords**- Nano composites, High Pressure Torsion, Microstructure, Texture

**Reference**

**Invited Lecture:**

**Advances in PM Processed Composites – Promises and Challenges**

Malobika Karanjai

International Advanced Research Centre for Powder Metallurgy and New Materials, Post Balapur, Hyderabad-500005

Corresponding author’s email id: malobika@arcy.res.in

**Abstract**

Composites and their functionalization are slowly engulfing the conventional component base material systems approach by overcoming the limitations of the range of use and simultaneous
expansion into niche areas of automobiles, aerospace, bio and nuclear, which demand enhanced specific properties for targeted applications. Thus each of these fields demand engineering of tailor made properties and specific processing route involving critical equipment design and selection. Particulate materials (PM) processing for engineering composites is one such arena.

Composites are mostly processed by Particulate Materials (PM) processing technology, which involves raw materials shaping in dies or moulds using room-temperature compaction technique and strength-improvement through sintering or hot consolidation. Advancements in hot-consolidation equipment resulted in elimination of the room-temperature compaction, thereby improving physico-mechanical properties, thereby making composites a very promising and upcoming field for engineering unique properties. In this presentation, some three case studies involving processing and property improvements in Fe-Cu-Sn based clutches and brakes, Fe-based magnetic materials for magnetic materials and Ti-Ca-P bioimplants are being discussed, with special emphasis on bio-implants.

Materials for bioimplants for hips and knee joints have gradually changed from metallic to bioceramic hydroxyapatite coated implants to combination of ceramic and polymer bioimplants. In each case the relative strength of metals, the biocompatibility of ceramics and the flexibility of the biopolymers were exploited to the maximum while mitigating their limitations by engineering the requisite composition of the intended application. Ti-Ca-P biocomposites with controlled porosity were synthesized by PM processing of in-situ biocompatible HA and other Ca-P phases, including bioconductive and bioinductive phases. The problem of stress shielding was avoided and adequate strength comparable to that of natural bone of hip and knee joints obtained. Hydroxyapatite, octacalciumphosphate (OCP) and tri-calcium phosphate (TCP) phases were produced in a matrix of Ti enabling biocompatibility. Service life of implant potentially improved due to better corrosion resistance and reduced wear. The processing and properties are detailed.

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**Invited Lecture:**

**Dynamic Recovery during Ausforming to Produce Nano Bainite**

Prantik Mukhopadhyay

Defense Metallurgical Research Laboratory, Kanchanbagh, Hyderabad - 500058

Corresponding author’s email id: prantikmukherje@dmrl.drdo.in

**Abstract**

Austenite is one of the desirable phases of steel with a good number of qualities for the development of high performance advanced nano bainite, TRIP and TWIP steels. The dislocation structures developed during ausforming to produce nano bainite, processing of TRIP and TWIP steels have been found to be governed by orientation of austenite crystallites. The work
hardening rate defined by dislocation network, dislocation-twin interaction and burger characters of dislocations have been observed to have crucial role in controlling TRIP, TWIP behaviors and for the decomposition of austenite to nano bainite respectively. Generally the austenite is known to have less stacking fault energy. Therefore, the annihilation of dislocations in austenite is formidable by cross slip process. Ausforming has been reported as a prospective process for developing nano bainite. The gliding dislocations on slip plane are found to have crucial effects on the variant selected nano bainite development. The dislocation density, character and distribution have been observed to have noteworthy role for controlling this process. Novel study on ausforming to develop nano bainite has reported that the dislocations should not be annihilated choosing higher processing temperature for producing nano bainite. Therefore, predicting the dislocation density during ausforming is crucial to estimate the associated process parameters. The dislocation recovery during ausforming in steels with formidable cross slip has been postulated by faster advancement of jogs. The jogged dislocations come back from strain hardened state by surplus back stress. That will cause recovery to a greater extent by mutual annihilation with other line defects of opposite signs during gliding. The constitutive equation has been formulated. Taylor factor (T) and stacking fault energy (SFE) have been turned out to be crucial features for controlling dynamic recovery. Gliding plane specific T and SFE qualify this simulator to predict orientation dependent dislocation recovery as well.

CON 9

Microstructure mechanical properties and oxidation behavior of ZrB2+20v/oSiC composite

Jiten Das, J Janardhana Reddy, Sweety Kumary, V V Bhanu Prasad
Defence Metallurgical Research Laboratory, Hyderabad
Corresponding author's email id: das.jiten@gmail.com

Abstract

Oxides, borides, carbides and nitrides of transition metals (Ti, Zr, Hf, Nb, Ta) are considered as high temperature materials owing to their high melting points. Due to poor thermal shock and creep resistance and thermal conductivity of oxides, transition metal carbides and borides are considered as superior than the former. Di-borides of Zr and Hf shows best high temperature oxidation resistance among all these carbides and borides. ZrB2 is chosen in this study because of its lower density and cost than HfB2. To reduce the density of ZrB2 and improve its flexural strength, fracture toughness, thermal conductivity and oxidation resistance SiC is added to it.

In this study, ZrB2+20v/oSiC composite was manufactured by high energy milling of ZrB2 and SiC powder followed by hot pressing of this milled powder at 1900°C at 30 MPa pressure using graphite die. 1 wt.%B4C was also added as a sintering aid. Microstructure and phase identification of this composite was carried out using SEM, EPMA and XRD. Major Phases are
found to be ZrB\textsubscript{2} and SiC with occasional presence of B\textsubscript{4}C phase. Average room temperature Flexural strength (FS) of the composite was found to be 385 MPa. The FS value remained same at even 1450 °C. Rather, FS value was found to improve at 1000 °C due to healing of pores by glassy phase at that temperature.

The ZrB\textsubscript{2}+20v/oSiC composite samples were also exposed to oxy-acetylene flame at ~1600°C temperature for 180 s as well as IR heating at 1500°C for 500 s at a heating rate of 10 °C/s. In both the cases sample did not show any visible crack and weight gain. Further analysis of the exposed sample surface using SEM and EDS showed that, fully protective silicon rich phase formed on the surface of IR exposed sample. Poor formation of such protective film was observed in case of oxy-acetylene flame exposed sample. This may be due limited access of air during the test.

*Keywords:* IR heating, Flexural strength, Zirconium diboride, Silicon Carbide

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**CON 10**

**Development of bulk polycrystalline MgO-based ‘age-hardened/toughened ceramic alloys’**

Luv Gurnani\textsuperscript{1}, Mahesh Kumar Singh\textsuperscript{1}, Ranjan Kathuria\textsuperscript{1}, Parag Bhargava\textsuperscript{2}, Amartya Mukhopadhyay\textsuperscript{1}

\textsuperscript{1}High Temperature and Energy Materials Laboratory, Department of Metallurgical Engineering and Materials Science, IIT Bombay, Powai, Mumbai - 400076, India

\textsuperscript{2}Particulate Materials Laboratory, Department of Metallurgical Engineering and Materials Science, IIT Bombay, Powai, Mumbai - 400076, India

Corresponding author’s email id: amartya_mukhopadhyay@iitb.ac.in

**Abstract**

Overall, the present research is aimed at developing ‘age-hardened ceramic alloys’ in bulk polycrystalline form via facile and cost-effective processing route, as viable alternative for the more ‘conventional’ ceramic nanocomposites (for advanced structural applications), which present significant processing and microstructural challenges. The route is based on the basic physical metallurgical principles of *in-situ* precipitation of second phase particles during aging of the bulk super-saturated ceramic solid solutions. Unlike the route based on sintering of powder mixtures (for the development of ceramic nanocomposites), the present route avoids the usage of starting nanosized powders, is devoid of the challenges associated with grain growth/particle coarsening during densification and does not necessitate the use of pressure/electric field-assisted sintering techniques and inert/reducing atmospheres.

As part of the ongoing research, MgO - 7 wt.% Fe\textsubscript{2}O\textsubscript{3} bulk supersaturated solid solutions were developed directly during pressureless sintering in air at 1650 °C (i.e., sintering-cum-solution treatment) followed by ‘air quenching’ using raising hearth furnace. Subsequent aging treatments of Fe-containing MgO at lower temperatures (1000 °C; based on the published MgO-Fe\textsubscript{2}O\textsubscript{3} phase diagram [1]) and for different durations (up to 20 h) led to the
precipitation of MgFe$_2$O$_4$ spinels as second phase particles, which were uniformly dispersed within the matrix grains, as well as along the grain boundaries and were rendered coherent with the MgO matrix. Such near-ideal microstructure developments (especially the presence of coherent second phase particles) are not possible in the case of ceramic (nano) composites. Investigations of the mechanical properties, using Vickers indentation, have revealed that the bulk MgO – based ceramic alloys containing the precipitated second phase MgFe$_2$O$_4$ nanoparticles possess superior hardness (up to ~ 52%) and indentation toughness (up to ~ 35%), as compared to the phase pure bulk MgO. Furthermore, the as-developed alloys exhibit enhanced wear resistance (with ~30% reduction in wear rate), as compared to the monolithic MgO [3]. Such significant improvements in wear resistance have been attributed to the suppression (by ~60% w.r.t. pure MgO) of brittle fracture induced pull-outs in the MgO - MgFe$_2$O$_4$ alloys developed upon aging at 1000 °C. The flexural strength and fracture toughness of the as-developed ceramic alloys is currently under investigation using a 4-point bend test set-up.

**Keywords**: Ceramic alloys; Sintering-cum-solution treatment; Aging; Wear resistance

Acknowledgment: The authors duly acknowledge the financial support provided by the Department of Science and Technology (DST; vide 12DST041), New Delhi, India and Centre for Excellence in Steel Technology (COEST), IIT Bombay. The authors would also like to express sincere thanks to SAIF, IIT Bombay for allowing the usage of electron microscopes.

**References:**

pressure and 4°C temperature simultaneously. The objective of this present paper is of two fold. Firstly the investigation of metal at high pressure through stress analysis is carried out followed by metallographic studies. The stress analysis results along with micrographs suggest that metals like steel which has BCC structure shows the remarkable decrease in ductility over the range of temperature. It can be said that aluminum having FCC crystal structure are stiff and ductile even at low subzero temperature. The theoretical calculations are done and they are in agreement with the experimental results. It is anticipated that influence of high pressure and low temperature can be criticized by two factors in terms of flow strength and fracture strength of metal. Ductile to Brittle Transition Temperature (DBTT) plays a vital role and can be considered as a measure of behavior of metal under high pressure and low temperature. Further analysis suggests that aluminum has greater deformation than steel. Stresses generated in steel material under similar condition are 2-3 times lesser than that of aluminum. So, it reveals that crystal structures of metals is the major factor which affects the behavior of metal which eventually affect the ductility and toughness of metal when subjected to high pressure and low temperature operations.

Key words: High pressure, Low temperature, Metal behavior, Toughness

CON 12

**Remarkable tensile properties of a thermo-mechanically processed AlCoCrFeNi_{2.1} Eutectic High Entropy Alloy**

Irfan Samad Wani\(^1\), Tilak Bhattacharjee\(^2\), Saad Sheikh\(^3\), Yiping Lu\(^4\), Subhradeep Chatterjee\(^1\), Pinaki Prasad Bhattacharjee\(^1\), Sheng Guo\(^3\), Nobuhiro Tsuji\(^5\)

\(^1\) Department of Materials Science and Metallurgical Engineering, IIT Hyderabad, India)  
\(^2\) Department of Materials Science and Engineering, Kyoto University, Japan)  
\(^3\) Materials and Manufacturing Technology, Chalmers University of Technology, SE-41296, Gothenburg, Sweden  
\(^4\) Key Laboratory of Solidification Control and Digital Preparation Technology (Liaoning Province), School of Materials Science and Engineering, Dalian University of Technology, Dalian 116024, PR China  
\(^5\) Elements Strategy Initiative for Structural Materials (ESISM), Kyoto University, Japan

Corresponding author’s email id: ms14resch11002@iith.ac.in

**Abstract**

The effect of thermomechanical processing on the development of microstructure and tensile properties of a AlCoCrFeNi\(_{2.1}\) lamellar eutectic high entropy alloy (EHEA) was investigated. For this purpose, the alloy was subjected to intense cold-rolling up to 90% thickness reduction followed by annealing at 800°C. The as cast alloy revealed eutectic mixture of L12 (ordered FCC) and B2 (ordered BCC) phases. Evolution of a nanocrystalline structure accompanied by progressive disordering of the L12 phase was observed. In contrast, the B2 phase maintained the
ordered structure during deformation. Annealing of the 90% cold-rolled material at 800°C showed formation of a duplex ultrafine microstructure. The annealed material showed very high tensile strength (>1000 MPa) coupled with appreciable ductility (>10% elongation to failure). The present results showed that the properties of the EHEA could be successfully engineered using thermo-mechanical processing.

*Keywords:* high entropy alloy, ordered phases, cold rolling, annealing, tensile properties.

**CON 13**

**Effects of Hydrogen on Thermal Creep Behaviour of Zircaloy**

Siddharth Suman¹, Mohd. Kaleem Khan¹, Manabendra Pathak¹, R.N.Singh²

¹ Department of Mechanical Engineering, Indian Institute of Technology Patna
² Mechanical Metallurgy Division, Bhabha Atomic Research Centre

Corresponding author’s email id: s.suman@iitp.ac.in

**Abstract**

Zircalloys are extensively used as nuclear fuel cladding. Creep is one of the most likely degradation mechanisms for fuel cladding during reactor operating and repository conditions. Fuel cladding tubes undergo waterside corrosion during service and hydrogen produced as a result of the corrosion diffuses into it. Hydrogen remains in solid solution up to terminal solid solubility and it precipitates as brittle hydride phase in the zircaloy metal matrix beyond this limiting concentration. Hydrogen either in solid solution or as precipitated hydride alters the creep behaviour of zircaloy fuel claddings. The present article critically reviews the influence of hydrogen on thermal creep behaviour of zircaloy fuel claddings, develops the systematic understanding of this multifaceted phenomenon, and delineates the thrust areas which require further investigations.

*Keywords:* creep; hydride; hydrogen; embrittlement; zircaloy

**CON 14**

**Synthesis and Study of Mechanical Properties of Short Bamboo Fiber Reinforced Epoxy Composites**

Anu Gupta

Department of Chemical Engg, JMIT, Radaur, Yamunanagar, Haryana, India

Corresponding author’s email id: anu339@gmail.com

**Abstract**

Natural fiber reinforced composites have generated a lot of interest in the research community as they offer an attractive solution to the ever depleting petroleum resource. Polymers are replacing
the conventional metallic materials in plenty of applications. Bamboo, a natural biomaterial has emerged as a potential material for composites, owing to its unique biological structure and mechanical performance among the natural fibres. Bamboo offers significant applications due to its high strength, easy availability at low cost. In the present paper, The short bamboo fiber reinforced epoxy composites are made by conventional manual stir casting technique followed by light compression moulding technique. A wooden mould having dimensions of 200 × 200 × 20 mm3 is used. The composites of four different compositions (10wt%, 20wt%, 30wt% and 40wt% fiber loading) treated and untreated with NaOH are made. The mechanical properties of the alkali treated bamboo composites (tensile strength, elastic modulus, flexural strength, flexural modulus, toughness, and impact strength) are determined. Higher percentages of NaOH concentration result in poor mechanical properties of composites. The morphology analysis reveals that the bamboo treated with 5% NaOH exhibited better compatibility with the epoxy and resins than the untreated bamboo.

CON 15

**Effect of Temperature on Mechanical Properties of (Nylon 6) PA6 / Talc Composite**

Namrata Verma, S. Aparna, D. Purnima

Department of Chemical Engineering, BITS-Pilani, Hyderabad Campus, Hyderabad, Telangana, India 500078.

Corresponding author’s email: dpurnima@hyderabad.bits-pilani.ac.in

**Abstract**

PA6 is a well-known engineering plastic having high crack initiation energy, moderate tensile properties and good chemical resistance. Talc is used as filler in industry to make products such as laboratory table tops, electrical switchboards and is widely used due to its low cost and abundant availability. Talc has low shear strength, resistance to heat, electricity and acids. For composites, changes in the temperature will lead to the change in mechanical properties due to high anisotropy of the system. In order to observe this phenomena, mechanical properties of the composites is performed at different temperatures. The composite of PA6 and talc of four different compositions are made and mechanical properties were analyzed at different temperatures. Various properties such as tensile strength, modulus of elasticity, tensile strain and toughness are measured for a temperature range of 50°C to 110°C. The obtained experimental results are compared with the theoretical tensile models which would aid in understanding the interactions between PA6 and Talc. Thermal properties are checked using DSC and TGA, wherein the effect of Talc on crystallization, melting temperature and degradation temperature on PA6/ Talc composites are studied.

**Keywords:** PA6, Talc, Tensile properties, DSC, TGA, Effect of temperature.
Deformation Behaviour of Low Carbon High Mn TWIP Steel

N. K. Tewary, S. K. Ghosh, S. Chatterjee

Department of Metallurgy & Materials Engineering, Indian Institute of Engineering Science and Technology, Shibpur

Corresponding author’s email id: tewarynisith@yahoo.com

Abstract

Twinning induced plasticity (TWIP) steel is a typical representative of the 2nd generation advanced high strength steel which exhibits a good combination of high strength and excellent ductility. The present study deals with the designing of low carbon and high manganese TWIP steel (Fe–21Mn–3Si–3Al–0.06C, wt%). Low carbon with high manganese along with addition of aluminum in TWIP steel possesses lowering in weight with higher strain hardening due to formation of twins during deformation, while the twins and austenite structure are the main contributors for ductility. The cold rolling has been introduced to obtain high strength without much loss in ductility. The fully austenite phase is obtained after solution annealing and deformation twins are evident and austenite grains become flattened during application of 10% to 50% cold deformation. With the increasing amount of cold deformation, high dislocation density and more deformation twins are evident along with some amount of noticeable micro shear bands. Deformation twinning can be considered to be the dominant deformation mechanism during the course of plastic deformation applied in the present study. X-Ray profiles confirm the presence of austenite phase before and after cold deformation and do not reveal any deformation-induced phase transformation of austenite to martensite ($\epsilon/\alpha'$) after cold deformation. The cold deformation results in the evolution of sub-structure, comprising dislocations, twins as well as interaction of dislocations and twins which are expected after cold deformation in austenitic steels having lower stacking fault energy in the range of 15-40 mJ/m2. The annealing twins are relatively coarser compared to the newly formed deformation twins in the nature of ripple. It is noticed that the hardness, yield strength and tensile strength of the specimens increase whereas percentage elongation decreases as the amount of cold rolling reduction increases.

Key words: TWIP steel, cold deformation, microstructure, mechanical properties.
The Effect of Heat Treatment T4/ T6 on the Microstructure of Zircon Reinforced Composites

Sandeep Sharma¹, Suresh Kumar², Ranvir Singh Panwar³, O. P. Pandey⁴, Tarun Nanda¹
¹ Mechanical Engineering Department, Thapar University, Patiala-147004, India
² Metallurgical Research Lab, School of Physics and Materials Science, Thapar University, Patiala-147004, India
³ Department of Materials and Metallurgical Engineering, PEC University of Technology, Chandigarh-160012, India
⁴ Metallurgical Research Lab, School of Physics and Materials Science, Thapar University, Patiala-147004, India
Corresponding author’s email id: bvt2275@gmail.com

Abstract
Aluminium matrix composites have spread a wide spread importance in the areas of aerospace, transportation, and defence as they provide a wide range of mechanical properties. In the present comparison study of wear behaviour of Al-Si (LM13) alloy based composite with different reinforced zircon sand particle size (0–20 µm) and coarse (50–75 µm). In this study, the sample prepared at T4 and T6 heat treatment conditions for wear behaviour of composite materials. The specimens were heated at 525 °C and held there for 1 hour followed by quenching in water T4 treatment. After T4 treatment the samples were naturally aged for 500 hours at room temperature. While, a few specimens were artificially aged at a temperature of 204 °C for 1 hour followed by air cooling T6 treatment. Microstructural and wear analysis were carried out on the specimens. Microstructural studies revealed that with heat treatment the eutectic silicon morphology changed from dendritic to globular shape. Further for all the heat treated samples T6 treated samples revealed a better wear resistance in comparison to that of T4 heat treatment conditions.

On The Heat Capacities of Aeronautical Grade Bearing Steels

M Sai Madhav¹, Ranjan Kumar Singh², Manish Roy³
¹ Structure & Failure Analysis Group, DMRL, Hyderabad, India
² Advanced Magnetics Group, DMRL, Hyderabad, India
³ Tribology Group, DMRL, Hyderabad, India
Corresponding author’s email id: ms.madhav@gmail.com

Abstract
Bearing steels for aero applications must meet a variety of requirements principally associated with mechanical properties and environment. There are 73 types of bearings of size from 5mm to 120mm that need to operate at speeds varying from 5000RPM to 60,000 RPM being used in
aircrafts. Ball Bearings or Roller Bearings are fabricated from a wide variety of steels. In a broad sense, bearing steels can be divided into classes intended for normal service, high-temperature service, or service under corrosive conditions.

Bearings for normal service conditions, a category that includes over 95% of all rolling-element bearings, also experience the effects of vibration, shock, misalignment, debris, and handling. Therefore, the steels should possess high toughness, a high degree of temper resistance, and microstructural stability under temperature extremes. The material must also exhibit the obvious requirement of surface hardness for wear and fatigue resistance. However, the deformation behavior of said steels is governed by the thermo physical properties in addition to the mechanical properties.

In view of the above, the present study is undertaken to understand the influence of heat treatment on the heat capacities of these steels. It is also intended to evaluate the effect of temperature on the heat capacities of the steels. In the present study AISI M50, AISI M50NiL, AISI 440C grade steels are selected.

Bearings made from AISI M50 steels are being used in various applications for many years. Jet engine speeds are being continuously increased in order to achieve improved performance and efficiency. The AISI M50 NiL steel which is having low carbon is used where the required toughness of these steels is more than twice that of through hardening steels. AISI 440C steel belongs to a class of corrosion resistant bearing steel of through hardening type. These steels offer good high temperature hardness as well as highest corrosion resistance. These steels are also used as cage material.

The results show that heat capacities increase with increase of temperature for all the steels. AISI M50NiL steel has the highest heat capacity, while AISI 440C has the lowest heat capacity. It is also to be mentioned that the heat capacity is independent of the dimension of the raw material stock from which the final product is manufactured.

Effect of Carbon Source on the Synthesis of Carbon Coated Nbc Nano Composite

Aayush Gupta, O.P. Pandey

School of Physics and Materials Science, Thapar University, Patiala, India
Corresponding author’s email id: present.ayush@gmail.com

Abstract

In this study, synthesis of nano niobium carbide (NbC) at low temperature has been done by using different form of carbon sources. Niobium penta-oxide (Nb2O5), metallic Mg powder and acetone/activated charcoal were used as reactants through carbo-thermal route. The XRD pattern
shows the confirmation of the single phase NbC. The Williamson-Hall analysis is also used to calculate the induced strain in the crystallite. The thermal analysis has been done to analyze the variation in thermal stability of the synthesized product. TEM micrographs show different morphology having agglomerated nanoparticles. Lattice fringing measurements have shown that the obtained product is single phase NbC.

Abstract

In the recent past, investigations are reported on Geopolymer concrete. Geopolymer is known as inorganic polymer. Such materials are useful materials which are capable of replacing constructional materials such as conventional cement. In present investigations, Geopolymer is made from chromium bearing slag obtained from a ferrochrome industry (BRG steel). The mechanism involved is that the silicon and aluminium present in the high carbon ferrochrome (HCFC) slag reacts with alkali liquid, forming Geopolymer which binds other non-reactive materials in the slag. The highest strength level achieved for as-prepared Geopolymers are 11 and 15 MPa by curing 7 days and 28 days, respectively. X-ray diffraction patterns reveal that there is a gradual transformation from crystalline phase to non-crystalline phase which is due to polymeric transformation with increase in curing time. SEM micrographs confirm formation of glassy phases at a higher curing time which also corroborate with mechanical properties such as compressive strength. Distinct changes are observed in FTIR spectra i.e, increase in peak height as well as appearance of many other peaks if compared with FTIR spectrum of virgin material. This strength is due to polymeric reactions and formation of chains with the monomeric structure. Striking features of the TGA pattern is observed for the two materials i.e., uncured and cured samples. DSC isotherms show oozing out of inbuilt water which has accumulated during condensation polymerization reaction. The strength level achieved for optimum combination of variable is found to be comparable to that of standard motar of grade (M15) as is used for constructional purpose.

Key words: Geopolymer, Slag, Cement and Compressive strength
Abstract

In the present study, the effect of different oxygen functional groups on diffusion behavior of water molecules in-between graphene oxide (GO) layers is studied using reactive force field (ReaxFF) based molecular dynamics (MD) simulations. For hydroxyl functionalized GO, more number of hydrogen bonds (H-bonds) were formed between GO layers (Fig. 1(a)), leading to diffusivity values of hydroxyl functionalized GO (~3.89*10^{-9} m^2/sec) to be almost half of that of epoxy functionalized GO (~7.81*10^{-9} m^2/sec) (Fig. 1(b)). The chemical nature of individual oxygen functional groups present on GO surface was found responsible for affecting the extent of intra- and inter-sheet H-bonds, which in-turn decided the interlocking of water molecules in between GO layers and formed the basis for the evolution of water clusters in-between covalently modified GO surfaces. The slope of the mean square displacement (MSD) and interlayer spacing between GO layers indicated a clear dependence on the type of attached oxygen functional group on GO surface. Present research suggests that the diffusion behavior of water molecules in-between GO layers can be tuned by tailoring of oxygen functional groups attached to individual GO layers through hydrogen bonding and thus opens up the possibility to obtain controllable motion of water molecules through selective surface modification of GO.

Figure 1 (a) Total numbers of H-bonds in the system, H-bonds within water molecules and water mediated H-bonds for GO with different oxygen functionalization and (b) Diffusivity of water molecules between GO layers with different chemical functionalization at 300K.
Keywords: Hydrogen bonds (H-bonds), diffusivity, chemical functionalization, mean square displacement, interlayer spacing, and molecular dynamics.

Microstructural Evolution and Mechanical Behavior of Electrodeposited Nanocrystalline Ni and Ni-Al Nanocomposites

Sumit Chhangani, M.J.N.V. Prasad

1 Department of Metallurgical Engineering and Materials Science, IIT Bombay, India
Corresponding author’s email ids: sumitchhangani@gmail.com, mjnvprasad@iitb.ac.in

Abstract

Mechanical behavior of nanocrystalline metals have attracted significant interest in materials science community since last two decades which is usually attributed to high grain boundary fraction of these materials. But the high fraction of grain boundaries inevitably imparts structural instability in these materials due to large driving force available. Various strategies have been suggested to improve the microstructural stability viz. by thermodynamic approach and/or by kinetic approach.

In this study, pulsed electrodeposited nanocrystalline Ni foils with varying saccharin content (1 g/L - 20 g/L) and varying Al nanoparticle content (1 g/L - 10 g/L) were used to study their room temperature mechanical behavior and microstructural evolution at high temperature. As-deposited nano-Ni shows higher yield stress with higher saccharin content. Similarly, as deposited Ni-Al composites with lower Al particle content exhibited significant improvement in yield stress and nano-hardness. Upon heating the nano-Ni above 773 K, faceted abnormal grains with different size and fractions along with significant textural changes were obtained with different sulfur and Al content. Grain growth study over a range of annealing temperature (573-923 K) and time (0.5-18 h) revealed that secondary atoms control grain growth kinetics of nano-Ni.
THEME: Degradation and Protection of Materials

Invited Lecture:

Friction and Wear Behavior of Phosphate Conversion Coatings

Andreas Nevosad¹, Heike Zacharias², Saranya Azharudeen¹, Nicole Dörr¹, Jürgen Klarner², Ewald Badisch¹

¹ AC2T research GmbH, Wiener Neustadt, Austria
² voestalpine Tubulars GmbH & Co KG Austria
Corresponding author’s email id: andreas.nevosad@ac2t.at

Abstract

Introduction
Phosphate conversion coatings are applied as corrosion protection and bonding agent for paints on steel, non-ferrous metals and metallic coatings since the early 20th century [1, 2]. Due to their non-metallic nature, good adhesion to the substrate and surface structure, which can enhance the effect of lubricants, phosphate conversion coatings are promising solutions for various tribological applications.

Experimental Methods
The investigated phosphate conversion coatings were prepared on steel substrates by a wet-chemical process. The tribological behavior was evaluated with respect to initial damage mechanisms and high stress low cycle stability. For the evaluation scratch testing and reciprocal sliding experiments in lubricated and dry condition were employed.

Results
The coatings which we investigated in this study consisted of a dense layer of crystal-like substructures with a size of several µm. It is shown that these coatings increase lifetime and reduce friction in dry and lubricated high stress friction conditions. In lubricated conditions, the asperities of the coating act as carrier for solid and liquid lubricants. In contrast, in dry conditions the coating reduces friction and prevents galling due to the plasticity and non-metallic nature of the coating. Since the performance of the coating depends on the surface structure, the influence of the surface treatment prior to deposition, like machining, cleaning or activating, on the surface morphology was also investigated. With the deeper understanding of the coatings’ tribological performance in terms of controlled friction and wear resistance new applications can be developed and the lifetime of components further increased.

References
Acknowledgements
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Invited Lecture:

Various Phosphate Treatments on Molybdenum Disulphide based Lubricant

Indumathi Vijey, K. Gayatri, B.V. Subbarao, C.V.S. Murthy and D.R. Yadav

Defence Research and Development Laboratory, India
Corresponding author’s email id: indu.vijey@gmail.com

Abstract

The corrosion resistance and lubricity characteristics of zinc and manganese phosphate coatings followed by molybdenum di sulphide based solid lubricant coatings on AISI 4130 steel are investigated by means of electrochemical measurements, salt spray test chamber and pin-on-disc. These results indicate that the corrosion resistance and lubricity of Manganese phosphate conversion coating followed by molybdenum di sulphide coating is better than zinc phosphate coated AISI 4130 steel substrate followed by molybdenum based lubricant coating.

Keywords: phosphate coatings, solid lubricant coatings, corrosion resistance

Invited Lecture:

Corrosion Resistant Cr-Coating on Mild Steel by Powder Roll Bonding

S. Khara, S. Choudhary, S. Sangal, K. Mondal

Department of Materials Science and Engineering, Indian Institute of Technology Kanpur, Uttar Pradesh, India, 208016
Corresponding author’s email id – kallol@iitk.ac.in

Abstract

Chromium powder coating on mild steel was developed by roll-bonding followed by annealing at 1200°C for different duration in hydrogen atmosphere to promote diffusion bonding and finally furnace cooling. All the coated samples were characterised using SEM, EPMA, bend test and potentiodynamic polarisation test in 0.2N H₂SO₄ solution. Diffusion layer thickness was in the range of 170-250 μm depending on the annealing time, and the Cr content reached up to 13 wt% in the diffusion layer. Diffusion layer exhibited high ductility and adhesion, since no crack was observed even after 180° bending. The chromium coated mild steels showed comparable
stable passivity and corrosion rates in 0.2N H₂SO₄ solution as observed in a 304 stainless steel. The corrosion rate decreased from 5.51 mm/y for the uncoated mild steel to 0.2-1.0 mm/y for the coated mild steel sample.

Keywords: Chromising; Roll-bonding; Diffusion coating; Passivation.

Invited Lecture:

Corrosion Analysis of Hardfacing Alloys through the Use of Pourbaix Diagrams

Varmaa Marimuthu, Krishnan Kannoorpatti
School of Engineering and IT, Charles Darwin University, Darwin, NT 0909 Australia
Corresponding author’s email id: Krishnan.Kannoorpatti@cdu.edu.au

Abstract

Hardfacing alloys based on metal carbides are popular in the industry to provide wear resistance. However, the alloys are also exposed to corrosive electrolytes. There are limited studies on the corrosion behaviour of the hardfacing alloys. The alloys are exposed to high pH solutions in the alumina industry, low pH is the sugar industry and in drilling operations, and neutral pH solutions in dredging. Further, the alloys are exposed to chloride environments when used as bio-implants and drilling and dredging implements. Hardfacing alloys based on chromium carbides were exposed to high-, low- and neutral-pH solutions. Potentiodynamic measurements were conducted to evaluate the materials under anodic polarisation. It was found that carbides corroded when exposed to high-pH solutions; carbides appeared to be intact when exposed to low- and neutral-pH solutions. Presence of chlorides hindered protection by inhibiting formation of passive films. The corrosion mechanism for the High Chromium White Irons (HCWI) in high-, low- and neutral-pH solutions was found to be galvanic. From these experimental results, the corrosion trend of HCWI in different environments was studied using the superimposed Pourbaix diagram of metal carbides. It was explained that the formation of passive films reduced the wear resistance of hardfacing alloys as carbides had to dissociate to form the passive films. The results of this work would provide significant contribution in understanding the corrosion behaviour of HCWI hardfacing alloys and possibly for design of crushers and drill, and other wear-resistant surfaces involving both corrosion and wear conditions.
Effect of Heat Treatment and Intercrystalline Corrosion Resistance on Low Nickel Cold Rolled Austenitic Stainless Steel

Sourabh Shukla

Department of metallurgical and materials engineering, VNIT, Nagpur, India
Corresponding author’s email id: sourabhshukla9876@gmail.com

Abstract

Due to the less availability and high price of nickel, Cr-Mn ASS or low nickel austenitic stainless steels (ASS) are more preferable for many industrial uses. In the present investigation, microstructural, mechanical properties and electrochemical behavior of Cr-Mn austenitic stainless steel with 15% cold reduction, followed by thermal ageing (800, 900, 950 and 1000 °C) were investigated using scanning electron microscopy, microhardness, tensile test and double loop electrochemical potentiostatic reactivation (DLEPR) test. The result shows that with cold rolling, thermal aged for 1, 2 and 3 hours, there is high sensitization in the temperature range of 850 °C and 900 °C but at the temperature of 950 °C and 1000 °C there is re-homogenization of samples. With cold rolling, there is a reduction in grain size as compared to base metal but as thermal aging temperature increases, grain size increases. During cold reduction, hardness also increases due to the formation of strain induced martensite. XRD analysis was carried out on Cr-Mn stainless steel for the 15% cold rolled which indicate the formation of strain induced martensite in which (110), (200), & (211) shows α'-martensite and (101), (102) peaks shows hexagonal ε-martensite. With the increase in thermal duration (1, 2, 3 hours) there is an increase in martensite formation at temperature of 850 °C and 900 °C and at the temperature of 950 °C and 1000 °C the work hardening diminishes. It is also observed that the increase in martensite in the sample increases the % degree of sensitization (DOS).

Keywords: cold worked, strain induced martensite, grain size

Microstructure and Corrosion Behavior of Shielded Metal Arc Welded Duplex and Austenitic Stainless Steel Dissimilar Joint

Jagesvar Verma, Ravindra Vasantrao Taiwade

Department of Metallurgical and Materials Engineering, VNIT, Nagpur, India
Corresponding author’s email id: jageswarverma28@gmail.com

Abstract

Duplex stainless steel is a classical futuristic material and a better candidate against the austenitic grade in many corrosive environments due to its versatile properties in terms of strength and
corrosion resistance which also shows the inevitability of dissimilar joint for cost flexibility. In the present investigation, an attempt has been made to the joint dissimilar metal of duplex and austenitic stainless steel, employing shielded metal arc welding process with E2209 duplex electrode and studied the microstructural and corrosion resistance of the weldment. The results of the analysis showed the balanced austenite/ferrite phase with the complex microstructure of grain boundary austenite, widmanstatten austenite and intragranular austenite developed from ferrite matrix. Weld metal showed higher pitting corrosion resistance compared to austenitic base metal. The Modified strauss test revealed the less sustainability of austenitic base metal.

**Keywords:** duplex stainless steel, austenitic stainless steel, dissimilar weld joint, widmanstatten austenite, strauss test.

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**CON 19**

**Comparative Evaluation of Sliding Wear Response of Various Bearing Steel**

R. Toppo, Manish Roy  
Defence Metallurgical Research Laboratory, PO: Kanchanbagh, Hyderabad: 500058

**Abstract**

Bearing steels are special variety of steels which are used in tempered martensite heat treated condition. These steels during operation undergoes degradation due to sliding wear, abrasive wear etc. Over the years significant work has been done on the sliding wear behaviour of various bearing steels. However, literature on comparative evaluation of their performances is limited. In view of this, present investigation has been undertaken to compare the sliding wear response of four types of bearing steels namely, 440C, M-50 Nil, 52100 and 4340 variety of steel which are used in the bearing assembly. Towards that purpose all these steels were subjected to sliding wear at ambient condition against 52100 steel balls as counter body. The microstructural features and mechanical properties of these steels are evaluated using optical, scanning electron (SEM) and transmission electron microscope and tensile testing machine. The worn surfaces are examined with SEM. The observation indicates that the friction coefficient decreases with increase in applied load. For a given load, in general, 52100 steel exhibits highest friction coefficient. It is observed that the wear depth is nearly independent of the applied load and test materials except for M 50 Nil. The height of the pile up materials around the worn grooves increases with applied load.
Mechanical and Tribological Properties of 4D C/C Composite

Sourav Sarkar¹, Manish Roy²

¹ Advanced System Laboratory, PO: Kanchanbagh, Hyderabad, 500 058, India
² Defence Metallurgical Research Laboratory, PO: Kanchanbagh, Hyderabad: 500 058, India

Corresponding author’s email id: souravsarkar_04@yahoo.com

Abstract

The Re-entry structure of a long-range missile will experience severe aero dynamic load during flight. The nose tip of the missile shall be capable of withstanding these severe aerodynamic stresses arising out of different loads in all direction. The present investigation has been undertaken to explore the possibility of using multi-directional reinforced carbon-carbon (C/C) composite such as 4D C/C composite for this application. C/C composites were fabricated using pitch as matrix material. Mechanical and tribological properties of this material have been evaluated. Tensile strength and flexural strength are 90.7±7.8MPa and 104±9.4MPa respectively. Solid particle erosion resistant and CIAT (Continuous impact abrasion test) erosion resistant are 400(Kg/Kg) and 20000(Kg/Kg) respectively. Result reveals this composite is very much comparable for this type of aerospace application.

Keywords: Carbon, fretting, CIAT

Short and Long Duration Hot Corrosion Behaviour of Ni-base Superalloy CM-247LC

Mahesh K. Kumawat, N. Hazari, Dipak K. Das

DMRL, Hyderabad, Telangana, India

Corresponding author’s email id: mahesh.kgp.iit@gmail.com

Abstract

Hot corrosion is a material degradation phenomenon observed for aeroengine components, especially those operating near sea shore. This degradation takes place in the temperature range of 650°C – 1000°C in presence of molten salt such as Na2SO4 and V2O5. The present study tracks hot corrosion degradation of CM-247LC Ni-base superalloy for various durations of exposure ranging from 5 min. to 1000 h. Hot corrosion testing of the above alloy was conducted by depositing a layer of Na2SO4 salt and exposing the salt coated samples to 950°C in air. The initial weight of deposited salt was kept constant at 3-4 mg.cm-2 for all the samples. Change in the weight for the samples was measured after each duration of exposure. Microstructural characterization of the corroded samples was carried out by using SEM, XRD and EPMA techniques. The results showed that the above superalloy degrades rapidly in the presence of
molten Na2SO4 salt as compared to the case when salt is absent, i.e. under pure oxidation in air. The initiation and propagation stages of hot corrosion degradation could be identified by analysing specimens exposed for short durations. The transition from a slow rate (initiation stage) to an accelerated rate (propagation stage) of degradation was found to occur after about 30 min. of exposure. The degradation was found to increase with the duration of exposure in the propagation stage. Long durations of exposure up to 1000h indicated that the degradation slowed down after about 500h as no further significant weight gain was recorded. However, the microstructure of the corroded specimens revealed that the alloy was almost consumed by the degradation after 1000h exposure. The findings of this study can have major implications of the use of this alloy in marine environment.

*Keywords:* Hot corrosion, Ni-base superalloy, gas turbine

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**CON 22**

**3D Finite-Element Analysis of Single Asperity Abrasion: Effect of Indenter Geometry**

Kannaki Pondicherry, Tongyan Yue, Stijn Hertelé, Jacob Sukumaran, Dieter Fauconnier, Patrick De Baets

Labo Soete, Department of Electrical Energy, Systems and Automation, Ghent University, Ghent, Belgium

Corresponding author’s email id: KannakiShanmughan.Podicherry@UGent.be

**Abstract**

Abrasive wear of materials occurs due to particles sliding along a surface (two-body abrasion) or in between two surfaces (three-body abrasion). This wear process may lead to early failure of many machine components exposed to e.g. slurry, sand, wear debris etc. Despite the tremendous efforts which have been invested to rank materials according to their wear resistance using different wear tests, there is still insufficient insight into the fundamental interaction process between the asperities and the material. This motivates the need to investigate and understand the fundamental interaction by means of single asperity abrasion tests or simulations. In this work, a 3D finite-element model has been developed to simulate single asperity abrasion, allowing more fundamental insight into the abrasion process. The 3D finite-element model simulates the ploughing or scratching of a rigid conical indenter into an elasto-plastic material, in this case steel S355. Hence, the model is representative for conical indenters with a hardness that is at least an order of magnitude higher than the hardness of the deformable material. The effect of nose tip radius and the cone angle of the rigid indenter on the material deformation and the wear mechanism is investigated in detail. Future work involves coupling this macro-abrasion model with microstructure based material model to assess the influence of microstructural characteristics.

*Keywords:* 3D Finite-Element Analysis, Single asperity, Abrasion, Wear
**Effect of Abrasive Hardness on Slurry Abrasion Response of Heat Treated En24 Steel**

Avishkar B Rathod, S. G. Sapate, R. K. Khatirkar, R. V. Taiwade  
Department of Metallurgical and Materials Engineering, VNIT Nagpur, India  
Corresponding author’s email id: rathodavishkar@gmail.com

**Abstract**

Wear by slurry abrasion is one of the potential problems in engineering components subjected to particulate flow such as slurry pumps, pipes carrying minerals and ore slurries in mineral processing and mining industries. In the present investigation, slurry abrasion behavior of En24 steel (Cr-Ni-Mo steel) under slurry abrasion conditions using different abrasives (silica sand, iron ore and limestone) was investigated. En24 steel specimens were hardened and tempered at different temperatures to obtain different microstructures. The heat treated En24 steel specimens were characterized using Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD) analysis. The slurry abrasion wear tests were carried out by using slurry abrasion tester with limestone, iron ore and silica sand as abrasive media with varying hardness. The wornout specimens were observed under scanning electron microscope to study the mechanism of material removal. The slurry abrasion wear loss exhibited an increasing trend with increase in the abrasive hardness, although the increase was not proportionate. The beneficial effect of increase in hardness of En24 steel on slurry abrasion resistance was significant with softer abrasive particles. The important material removal mechanisms were observed to be ploughing and microcutting, the relative contribution of each being influenced by the relative hardness of abrasive particles. The inadequacy of wear coefficient in fully describing a wear system has been presented.

**Influence of Processing Condition on Erosive Wear of Hardfaced Stellite Layer**

Venkatesh B. 1, Bharti Malvi2, Manish Roy3  
1 Vardhaman College of Engineering, Shamshabad, Hyderabad: 501218, India  
2 National Institute of Technology, Jamshedpur, India  
3 Defence Metallurgical Research Laboratory, PO: Kanchanbagh, Hyderabad: 500 058, India  
Corresponding author’s email id: bvt2275@gmail.com

**Abstract**

Solid particle erosion is an important material degradation process. One way of improving the erosion resistance of a material is to suitably modify the surface. Hardfacing is a well known surface modification processes in present days. Operational simplicity, low capital cost and low
operational cost of the hardfacing have made it attractive for engineering industries. Stellite is considered to be potential hard material, for erosion resistant application. This material can be deposited by hardfacing. In view of the above, the present investigation has been undertaken to evaluate the influence of processing condition on room temperature erosion response of stellite layer deposited by hardfacing. Towards that purpose, stellite layers were deposited on mild steel (MS) by hardfacing. The microstructural features and mechanical properties of these coatings were characterized with the help of optical microscopy, scanning electron microscopy (SEM), X-ray diffraction technique and micro hardness tester. Solid particle erosion rate was determined employing an erosion test rig. The morphology of the eroded surfaces and the areas beneath the eroded surfaces were examined by means of a SEM. The results showed that the stellite coating by hardfacing improves erosion resistance. Most coatings exhibit ductile erosion response, Material loss from hardfaced coating is by formation of lips and their subsequent fracture.

PIV

Slurry Abrasion Behaviour of Ni-Cr-Si-W-B Thermal Spray Coating

Mithun M, A.B Rathod, Nitesh Vashishta, R K Khatirkar, S.G Sapate
Department of Metallurgical and Materials Engineering, VNIT Nagpur, India
Corresponding author’s email id: mithunm.tkm@gmail.com

Abstract

The surface coatings deposited by thermal spray technique are widely used for industrial applications involving wear, corrosion and high temperature. Some of the commonly used coatings are WC-Co, CrC2 – NiCr , NiCrSiWB and oxide ceramics. The selection of a particular coating material is decided by the prevalent service conditions. Austenitic stainless steels possess good high temperature properties, but lack wear resistance, e.g. in coal water slurry nozzles where both high temperature properties and slurry abrasion resistance are required. In the present work, slurry abrasion response of NiCrWSiB coating deposited by thermal spray and fuse technique was studied. The structural characterisation of the coating was carried out using Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS) and XRD. The slurry abrasion experiments were carried out using silica sand slurry with two different particle sizes; 300-425 µm and 150-212 µm. The effect of sliding distance, load and slurry concentration on slurry abrasion mass loss was studied. The slurry abrasion mass loss was influenced relatively more strongly as compared to load and sliding distance. SEM studies were carried out to study material removal mechanisms. The effect of test parameters on abrasive particle fragmentation during slurry abrasion was evaluated. The results of the present work were rationalised with respect to microstructure and material removal mechanism under different test conditions.
Effect of Calcium Addition in Mg-Li alloy and Study Their Microstructure, Corrosion behaviour and Mechanical properties

P.Bhagat Singh, Deepak Kumar Sahu, S.Kumaran
Dept. of MME, NIT Trichy
Corresponding author’s email id: bhagatsinghmsm@gmail.com

Abstract
Magnesium alloy is gradually becoming important since it has low density, high specific strength, high dimensional stability and is recyclable. Among the various magnesium alloys, Mg-Li alloys are getting more attention among various magnesium based alloys due their light weight and better ductility. However, the poor corrosion resistance of Mg–Li alloy is restricting its applications in various engineering sectors. To improve the corrosion resistance of Mg-Li alloy, calcium is considered as a ternary alloying element. Calcium can act as a grain refiner and also improves the creep resistance. It can provide strength through forming a second phase. In this study, the changes in microstructure and mechanical properties of α-phase of Mg–Li alloy with calcium have been studied. Initially, Mg-Li-Ca alloy was developed using stir casting process. Cast billets were hot extruded at 300 C with 4:1 ratio. The mechanical testing hardness and tensile tests were performed. The corrosion tests had been performed on all the samples. Calcium addition in Mg-Li alloy enhances the strength and corrosion resistance.

Keywords: Mg-Li-Ca alloy, Extrusion, Corrosion, Hardness and Tensile testing.

Corrosion Behavior of Friction Stir Processed SS316L Marine Steel

Karthikeyan Selvam, H. S. Grewal, H. S. Arora#
Department of Mechanical Engineering, Shiv Nadar University, Gautam Budh Nagar, India
#Corresponding author Corresponding author’s email id: harpreeet.arora@snu.edu.in

Abstract
Corrosion is an age old problem affecting longevity and maintenance of metals. Marine components and off shore structures, in particular, are constantly exposed and operates under extreme aqueous corrosive environment. Development of superior corrosion resistant alloys such as stainless steels show passivation under corrosive environment. However, further improvement in corrosion behavior of marine grade steel can result in significant cost savings. Tailoring the surface properties of materials through high strain deformation is an economical way for addressing degradation by corrosion. Friction stir processing is one of the emerging surface modification technique which is widely used for tailoring surface properties. In the current study, corrosion behavior of friction stir processed marine grade steel, SS316L, was investigated. FSP
was done under controlled cooling conditions over a wide strain rates. Microstructural characterization was done using electron back-scatter diffraction (EBSD) which revealed ultra-fine grain structure for samples processed under sub-merged condition. Corrosion behavior of all samples was evaluated using potentiodynamic, electrochemical impedance spectroscopic (EIS) and pitting corrosion tests in 3.5% NaCl solution. The as-received SS316L sample was also tested under similar conditions. The results indicate higher corrosion resistance of the processed sample compared to the as-received alloy which is mainly attributed to the higher stability of the passive oxide layer. This study gives a fundamental insight on designing materials for advanced marine applications.

PVII

Comparative Evaluation of Gas Tungsten and Shielded Metal Arc Welding of Chrome Manganese Austenitic Stainless Steel

Ankur Bansod, Awanikumar P. Patil
Metallurgical and Materials Engineering Department, VNIT Nagpur
Corresponding author’s email id: ankur.1754@gmail.com

Abstract
Austenitic welds (Cr-Mn ASS) prepared by two different welding processes is studied in this paper. The two welding processes considered are shielded metal arc welding (SMAW) and gas tungsten arc welding (GTAW). GTAW exhibited equiaxed grains as compared to SMAW welding process. The welding specimen of GTAW had superior tensile and hardness. The tensile ductility of GTAW as also on an average marginal greater than SMAW welding. Both the the process shows low ductility as compared to base metal. The corrosion resistance of GTAW was higher than SMAW welding process. This is due to more δ ferrite in the weld matrix. However in 0.5 M H2SO4 there is higher dissolution of δ ferrite in GTAW as compared to SMAW.

PVIII

Superior Corrosion Resistant High Entropy Alloy: Potential Candidate for Marine Applications

B S Rakesh¹, H S Arora¹, Sundeep Mukherjee², H Singh³, H S Grewal¹*
¹ Department of Mechanical Engineering, Shiv Nadar University, Gautam Budh Nagar, India
² Department of Materials Science and Engineering, University of North Texas, Denton, USA
³ Department of Mechanical Engineering, Indian Institute of Technology Ropar, Rupnagar, India
Corresponding author’s email id: harpreet.arora@snu.edu.in

Abstract
Corrosion is a serious issue in many industrial applications such as marine, agriculture and mineral handling. The surface degradation due to corrosion lowers the durability of the
components, hampering their service life. The degradation due to corrosion, however, inevitable, can be controlled through judicious material selection. High entropy alloy (HEA) represents a new approach for the design of multi component alloy formed by equi-molar /nearly equi-molar fraction. HEAs have shown attractive properties for use as structural materials. The objective of present work is to investigate the corrosion behavior of a single phase solid solution Al0.1CrCoFeNi HEA. For comparison, conventionally used structural materials, stainless steel SS316L and cast iron were also evaluated. Detailed microstructural and mechanical characterizations were conducted using scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS), x-ray diffraction (XRD) and micro-and nano-hardness testings. The electrochemical corrosion behavior was evaluated using potentiodynamic and electrochemical impedance spectroscopy (EIS) in 3.5 wt% of NaCl solution. HEA showed passivation behavior with nobler corrosion potential and low current density indicating its superior corrosion resistance compared to stainless steel and cast iron. The EIS studies complement the potentiodynamic results. The high corrosion resistance of HEA is attributed to its stabilized atomic structure as a result of high configurational entropy which helps to form stable and homogenous passive layer. The current study suggests high entropy alloys as potential candidate materials for marine applications.

PIX

High Temperature Erosion of Hardfaced Stellite Layer

Venkatesh B, Manish Roy

1 Vardhaman College of Engineering, Shamshabad, Hyderabad: 501218, India
2 Defence Metallurgical Research Laboratory, PO: Kanchanbagh, Hyderabad: 500058, India
Corresponding author’s email id: bvt2275@gmail.com

Abstract

Erosive wear or solid particle erosion is defined as material degradation due to impact of particles travelling with some significant velocity. At elevated temperatures number of industrial components such as compressor blades of gas turbine, burner nozzles, super heater in combustion systems, turbines, lock hopper valves in coal gasification systems etc. are subjected to erosive wear leading to materials degradation at elevated temperature. The material removal during elevated temperature erosion is governed by the synergistic effect of erosion and oxidation. The synergy between erosion and oxidation, in turn, is determined by the thickness, pliability, morphology, adhesion characteristics and toughness of the oxide scale that forms at elevated temperature.

Present investigation has been undertaken to examine the elevated temperature erosion behaviour of stellite layers on mild steel obtained by TIG welding. Towards that purpose, stellite layer was deposited on mild steel using TIG welding process. Hard faced satellite layer on mild steel was subjected to solid particle erosion at elevated temperature up to 1073 K. The mechanical
properties of these alloys are determined at different temperature. The dependence of erosion rate on impact angle and impact velocity has been evaluated for five different test temperatures. The morphology of the eroded surface is examined with scanning electron microscopy (SEM). The area beneath the eroded area is characterised using SEM and electron probe micro analyser (EPMA). The results show that erosion rate is not related with the mechanical properties of the alloys. In the experimental regime, metal erosion dominates. However, erosion at higher temperature is characterised by formation of a mixed layer containing the metal and the erodent.

Understand the Wear and Friction Behavior of Zicaloy-4 in Pilgering Process

Dewika Mishra, Farjana J.Sonia, M.K. Jangid, Ranjan Kathuria, Amartya Mukhopadhyay

High Temperature and Energy materials Laboratory, Department of Metallurgical Engineering and Materials Science
Indian Institute of Technology Bombay, Powai, Mumbai 400076, India
Corresponding author’s email id: amartya_mukhopadhyay@iitb.ac.in

Abstract

In Pilgering Process, maintaining good lubrication regime in the inner surface of the tube is a major problem and causes increase in surface roughness. In the present study, an investigation of inner surface topography of partly pilgered zircaloy tubes has been carried out in terms of wear mechanisms. SEM analysis of worn out surfaces indicate adhesion, delamination and abrasion as major wear mechanisms. With degradation of the lubricant, wear mechanism changed from adhesive wear to abrasive wear and delamination. Area fraction of wear in zircaloy samples also pointed towards improvement in surface finish as pilgering progressed. Additionally some tribological test of zircaloy-4 was examined at room temperature and different loads in linear reciprocating tribometer. Wear test were carried out at under dry and lubricated condition by using different lubricants to study the effects of process parameters on the wear mechanism. Coefficient of friction and wear rate were measured under both conditions with and without lubrication. It was found to coefficient of friction slightly decrease with increase in load and comparative study of lubricant behavior noted that volumetric wear rate is less by graphene used as a lubricant.

Keywords: Pilgering, Zircaloy, Tribology, Wear, Lubricant

Acknowledgement

The authors would like to thank BRNS, India for provided financial support for this project and NFC, Hyderabad India for providing the sample used in this study.
Carboxyl Terminated Polyethylene Glycol Succinate (CTPS) Modified Epoxy Anticorrosive Coating System for Marine Application

Rashmi David¹, V S Raja¹ and S K Singh²

¹Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Mumbai-400076, India
²Naval Materials Research Laboratory, Shil-Badlapur Road, P.O.Anandnagar, Ambernath-421506, India
Corresponding author’s email id: rashmidavid@gmail.com

Abstract

Epoxy resins based anticorrosive coatings are known to have excellent adhesion on various substrates, good chemical and corrosion resistance. However, these resins are brittle in nature making them unsuitable for applications where high toughness and abrasion resistance are required. A suitable modifier is needed to overcome this inherent drawback of these resins. Carboxyl terminated polyethylene glycol succinate (CTPS) is one such modifier that can offer excellent flexibility owing to the presence of carbonyl linkage as well as long aliphatic chain in the backbone. In this study, CTPS was synthesized and the same was used to modify bisphenol-A epoxy resin. The unmodified epoxy and CTPS modified epoxy resins were cured using polyamide hardener. Mechanical properties of the free films were studied. So produced restores were coated on mild steel specimens and evaluated for their adhesion strength and resistance to corrosion. The corrosion resistance of the coatings were evaluated through salt fog, sea water immersion tests and electrochemical impedance spectroscopy (EIS). This study revealed that 5 wt. % CTPS modified epoxy resin (CPSE5) has superior mechanical properties, adhesion strength and corrosion resistance as compared to the other compositions and unmodified epoxy resin.

Keywords: Epoxy, Carboxyl terminated polyethylene glycol succinate (CTPS) and Electrochemical impedance spectroscopy (EIS).
Invited Lecture:

Influence of defect chemistry on the performance of Li\textsuperscript{+} or O\textsuperscript{2-} ionic conducting materials for electrochemical energy storage.

Markus Kubicek,\textsuperscript{1} Andreas Welzl,\textsuperscript{1} Tobias Huber,\textsuperscript{1} Alexander Schmid,\textsuperscript{1} Daniel Rettenwander,\textsuperscript{2,3} Reinhard Wagner,\textsuperscript{2} Georg Amthauer,\textsuperscript{2} Herbert Hutter,\textsuperscript{1} Jürgen Fleig\textsuperscript{1}

\textsuperscript{1} Institute of Chemical Technologies and Analytics, Vienna University of Technology, Getreidemarkt 9/164EC, 1060 Vienna, Austria
\textsuperscript{2} Department of Chemistry and Physics of Materials, University of Salzburg, Hellbrunnerstraße 34, 5020 Salzburg, Austria
\textsuperscript{3} Center for Materials Science and Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, Massachusetts 02139-4307, United States

Corresponding author’s email id: markus.kubicek@tuwien.ac.at

Abstract

Finding new and improving existing renewable energy solutions is a global challenge of the 21\textsuperscript{st} century. Solid ionic conductors play an important role for several new energy technologies such as Li batteries or solid oxide fuel cells. Their performance as electrolyte and their reactivity as electrolyte is granted by ionic defects. Understanding defect equilibria in ion conducting materials is therefore key to improve existing materials and developing more efficient materials and devices. In this contribution two examples how to measure and manipulate defect chemistry are presented: Fast Li ion conducting garnets as Li battery electrolyte and mixed oxygen ionic and electronic conducting electrodes for solid oxide fuel cells.

The garnet Li\textsubscript{7}La\textsubscript{3}Zr\textsubscript{2}O\textsubscript{12} (LLZO) has received a lot of attention towards replacing today’s liquid electrolyte Li-batteries by all solid state, which would be desirable to avoid safety and durability issues such as dendrite driven short circuit or thermal runaway. Essential for high room temperature Li conduction is the stabilization of cubic phase(s) and avoiding the tetragonal phase. The effects of donor doping and sintering parameters on the defect chemistry of LLZO and associated with that the crystallographic phase and the electric performance is discussed. Importantly, not only cation defects are present in LLZO, but also oxygen vacancies have been found to have an influence. Experiments proving their existence in LLZO single crystals and polycrystals are presented and their impact on the cation defect concentration is discussed.

Mixed oxygen ionic and electronic conducting perovskite are state of the art cathode materials for solid oxide fuel cells. Their defect chemistry determines not only their partial conductivities, but also surface catalytic properties. By deliberate shifting and manipulation of their defect chemical parameters it is possible to determine, change and optimize materials properties. Three
examples of different acceptor-doped perovskite oxides (La$_{1-x}$Sr$_x$MO$_{3.5}$, M=Mn, Fe, Co) are presented. Different drivers for directly affecting defect chemistry such as electric potentials, oxygen partial pressure or mechanical strain are probed in different experiments and discussed towards their implication on materials properties.

Invited Lecture:

**Unraveling the Critical Role of Materials Design on Electrochemical Performance**

Aninda J. Bhattacharyya

Solid State and Structural Chemistry Unit Indian Institute of Science, Bengaluru

Corresponding author’s email id: aninda_jb@ssc.iisc.ernet.in

**Abstract**

Ion and electron transport at small length scales (as well as at reduced dimensions) play a critical role in determining the effective macroscopic parameters of electrochemical devices. The materials composition and structure has a major influence and intrinsically determines the electron and ion transport in the bulk and at various interfaces. Composite and hybrid (inorganic-organic) assemblies, where synergistic and confinement effects play a critical role, offers multiple opportunities for the design of advanced functional materials for efficient energy generation and storage. The talk will highlight and discuss through a few case studies the importance of chemical design of materials on the effective electrochemical phenomena in the context of rechargeable batteries.

Invited Lecture:

**Electrochemical Behavior of LiFePO$_4$ Cathode: Function of Nonstoichiometry, Doping and Impurity**

B. P. Mandal

Chemistry Division, Bhabha Atomic Research Centre, Mumbai – 400085, India

Corresponding author’s email id: bpmandal@barc.gov.in

**Abstract**

The olivine LiFePO$_4$ has been attracting lot of attention as promising cathode material for Li ion batteries. However, its poor electronic conductivity is a major challenge for its industrial applications. Different approaches have been taken to address this problem. Here, we report a method of improving its electrical conductivity by varying the annealing condition and doping with indium ions at the Fe site of LiFePO$_4$. Another set of samples were also prepared in
presence of fatty acid like lauric acid as carbon source. X-Ray Diffraction and Raman spectroscopy confirm that the olivine structure remains unchanged upon doping with indium. The electronic conductivity increased from $10^{-9}$ to $10^{-4}$ Ohm$^{-1}$ cm$^{-1}$ upon doping and coating with carbon. SEM studies show a bigger grain size of ~300-500 nm in doped LiFePO$_4$ which decreased to ~50 nm when the materials were synthesized in presence of lauric acid. In another attempt, the content of carbon also varied so that optimum coating thickness could be achieved. In next step Li concentration in LiFePO$_4$ has been varied to investigate the correct Li and Fe ratio for better electrochemical performance. Half cells were fabricated as against Li metal electrode and the galvanostatic charge-discharge performance of the cell has been investigated at different current rate. It has been observed that capacity of the cell reaches as high as 142 mAh/g at C/2 rate. The cell performance remains nearly undisturbed after 500 cycle.

**Invited Lecture:**

**Systems & Solutions for Researchers in Energy Studies from Metrohm India Ltd**

Santosh Kulkarni

Metrohm India Ltd, Chennai

Corresponding author’s email id: santosh@metrohm.in

**Abstract**

Research in the area of Energy Conversion & Storage Devices is of primary importance to the scientific communities’ world over and more so in India. The researcher in this field study material properties of different constituents as well as complete cell assemblies and stacks. For some of the researchers aging of materials and complete cell assemblies are subject of interest.

Metrohm India Ltd, being the leading supplier of total solutions to their customers, are active in this field. To cater to the growing and diversified needs of our customers we have range of products in our product basket.

In this talk we give an overview of the solutions offered by MIL to the researchers in the field of Batteries, Flow Batteries, Super Capacitors, Solar Cells, Solar Hydrogen Generation & Fuel Cells. We briefly discuss different topics viz: electrochemical measurements on energy conversion and storage devices, different hyphenated measurements on energy conversion and storage devices & also the aging related tests on batteries and super capacitors.
Invited Lecture:

Metal Carbides: Futuristic Materials for energy devices

O.P. Pandey

School of Physics and Materials Science, Thapar University, Patiala 147004
Corresponding author’s email id: oppandey@thapar.edu

Abstract

The current requirement of energy saving materials in different forms has put the scientists to work and search new materials. In this regard, transition metal carbides (TMCs) are different class of materials and have been offering better properties. These materials are very good catalyst to replace Pt, Pd, Rh, Au metals and hence helping in cost reduction. Moreover, very high melting point with very good thermal stability is an additional advantage to use them even at high temperature. Despite of all these properties, TMCs have not become so popular as expected. The reason for it is the associated carbon atoms inside the lattice and also the carbon coating on outer side of the particles. In this work, all these aspects have been analyzed and presented. The work highlights the fundamental aspect to use them as catalyst and to further calibrate their applications for long duration testing. The work done on different metal carbides is basic in nature and will be described in different ways to search a suitable application aspect in energy associated devices.

CON 25

Evaluation of Anodically Deposited CeO2-x Coatings on SS316L in Proton Exchange Membrane Fuel Cell condition

Nitish Kumar¹, Raghuram Chetty², Lakshman Neelakantan¹

¹ Department of Metallurgical and Materials Engineering, Indian Institute of Technology Madras, Chennai, India-600 036
² Department of Chemical Engineering, Indian Institute of Technology Madras, Chennai, India-600 036

Corresponding author’s email id: nitishkashu92@gmail.com

Abstract

Proton exchange membrane fuel cell (PEMFC) is considered as an alternative and efficient energy conversion device for transport application. A major component of the PEMFC is bipolar plate (BP) which encompasses around 25% of the cost and 80% of weight in a fuel cell stack. A commonly used BP material is graphite, however, metallic BPs have gained much attention in recent time because of their high corrosion resistance, high electrical conductivity, high gas impermeability and low-cost machinability. Even though, metallic BP provides certain advantages over graphite BP the acidic condition in PEMFC results in corrosion of metallic BP.
This further degrades the polymer membrane by the leach out metal ions leading to reduction in the fuel cell performance.

In this work, stainless steel (SS) 316L was coated with ceria and its performance in simulated fuel cell conditions was studied. Non-stoichiometric CeO2-x film is known to exhibit good electrical conductivity and Ce ions are well known as effective reversible scavengers of •OH, an intermediate formed at the fuel cell cathode. CeO2-x deposition on SS 316L was performed by anodic electrodeposition and compared with bare SS 316L surface. DC and AC methods like potentiodynamic, potentiostatic, electrochemical impedance spectroscopy and Mott-Schottky analysis were employed to understand the electrochemical behaviour. It was found that the CeO2-x coating exhibited better corrosion resistance than the bare SS surface.

CON 26

Study of Transition Metal Olivines as Intercalation Host for Rechargeable Mg-ion System

Katja Fröhlich, Hristina Vasilchina, Simeon Stankov, Atanaska Trifonova

AIT Austrian Institute of Technology GmbH, Mobility Department, Electric Drive Technologies; Giefinggasse 2, 1210 Vienna, Austria
Corresponding author’s email id: katja.froehlich@ait.ac.at

Abstract

Magnesium-ion batteries have great potential for the replacement of conventional lithium-ion systems in future electrical energy storage applications. Magnesium is environmentally benign, shows higher charge per volume due to its bivalency, and has no dendrite formation. Unfortunately, magnesium-ion secondary battery systems still have challenges to overcome, for example slow ion diffusion in the intercalation host, low cell voltage, and a small electrochemical window of the electrolyte.

Development and design of battery materials requires deep understanding of the relation between their chemistry, structure, and properties. Ab initio calculations provide information from which the materials’ usability in electrochemical cells can be predicted and further details about their structure and stability are obtained. In order to investigate new magnesium-ion battery materials, density functional theory was used as simulation tool on the atomic scale to find the most promising cathode structure.

In this work, first-principles calculations of different reasonable intercalation host matrices as cathode materials for Magnesium-ion cells were carried out using Wien2k software invented by the Computational Quantum Chemistry Group at the Technical University of Vienna [1]. The most promising cathode materials, MgMn2(PO4)2 and MgCoSiO4, are presented. In addition, the results relevant to battery applications will be discussed and compared to experimental data.
Financial support was provided by the Austrian research funding association (FFG) under the scope of the Mobilität der Zukunft program within the research project N° 840457


**Keywords:** Mg-ion batteries, transition metal olivines, ab initio calculation

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**Preparation and Electrochemical Evaluation of Cr-doped Lithium Vanadium(III) Phosphate**

Arlavinda Rezqita, Simeon Stankov, Atanaska Trifonova

AIT Austrian Institute of Technology GmbH, Mobility Department, Electric Drive Technologies; Giefinggasse 2, 1210 Vienna, Austria

Corresponding author’s email id: arlavinda.rezqita@ait.ac.at

**Abstract**

The development of new cathode materials with high energy density is a focus nowadays for further evolution of the Li-ion technology. In general, cathode materials should meet the requirements such as high specific capacity, excellent cycle life and safety. Lithium vanadium phosphate (LVP) based cathode is promising due to high theoretical capacity (197 mAh g⁻¹) and operational potentials. However, this compound suffers poor electronic conductivity and short cycle life. An efficient electron wiring such as carbon layer on the surface of the material’s particles and partial substitution of vanadium with metals represent a possible route to overcome the aforementioned problems.

This study focus on partial substitution of V³⁺ by Cr³⁺ in LVP resulting compounds with general formula: Li₃V₂-xCrₓ(PO₄)₃-C. A two-step sol-gel combustion method was used to synthesize the series of samples with different amount of doping agent. X-ray powder diffraction (XRD) and Scanning Electron Microscopy (SEM) were conducted to analyze the powders. The materials were also tested in galvanostatic rate capability mode and cyclic voltammetry.

**Keywords:** Lithium vanadium phosphate, Li-ion batteries, Cr-doped LVP
Insight into the Li-storage of Well-Ordered Few Layers Graphene: a Combined Experimental, Analytical and Computational Study

Farjana J. Sonia\textsuperscript{a}, Manoj K. Jangid\textsuperscript{a}, Balakrishna Ananthoju\textsuperscript{b,c}, Ravi Kali\textsuperscript{a}, M. Aslam\textsuperscript{c}, Priya Johari\textsuperscript{d}, Amartya Mukhopadhyay\textsuperscript{a}

\textsuperscript{a} HTEM Laboratory, Department of Metallurgical Engineering and Materials Science;
\textsuperscript{b} IITB-Monash Research Academy;
\textsuperscript{c} Nanomaterials Laboratory, Department of Physics;
Indian Institute of Technology Bombay (IITB), Mumbai, India
\textsuperscript{d} Department of Physics, School of Natural Sciences, Shiv Nadar University, Uttar Pradesh, India

Corresponding author’s email id: amartya_mukhopadhyay@iitb.ac.in

Abstract

Though graphene has recently been observed to possess enhanced Li-capacity, in comparison to graphite, the most commonly used anode material of Li-ion battery, the lithiation/delithiation mechanism and mechanical integrity of graphene upon repeated Li-intake/uptake is still under debate. Hence using CVD grown fairly well-ordered few layers graphene (FLG; \textasciitilde 7 layers), as model material, and performing extensive structural/electrochemical investigations as well as innovative \textit{in-situ} studies, we have been able to develop better understanding of the inter-related phenomena concerning lithiation/delithiation mechanisms leading to higher capacity, stress developments at different state of charges and mechanical integrity. The overall capacity, obtained with the FLG was \textasciitilde an order of magnitude higher than that of similarly grown thicker bulk graphite (TBG). Moreover the presence of distinct features of formation of Li-intercalated graphite compounds in cyclic voltammogram and choropotentiogram of FLG below 0.25 V (against Li/Li\textsuperscript{+}) indicates that Li-intercalation/de-intercalation in the inter-layer spaces take place even in those reduced dimensions of graphite. Furthermore, the capacity obtained for FLG within just the same lower potential window and even after removing maximum possible contribution from Cu substrate is still \textasciitilde 3 – 4 times greater than the overall Li-capacity obtained for TBG and had contributions from both the diffusion as well as surface controlled processes of Li-storage. Modelling Li-storage in graphene via density functional theory indicated that the excess Li-capacity (beyond formation of LiC\textsubscript{6}) is associated with additional stable Li-storage on the surface in form of multiple Li layers (but different from Li-plating) and segregation close to the stepped edges. The \textit{in-situ} monitoring of stress development in the FLG during the full range of lithiation/delithiation has indicated that the mechanisms of excess Li-storage (i.e., other than Li-intercalation in interlayer spaces of FLG) leads to comparatively much less dimensional changes. Moreover via \textit{in-situ} stress monitoring and geometrical modelling, it has also been observed that the major structural degradation in graphenic structure takes place in the initial stages of lithiation and later stages of delithiation, i.e. at the potentials higher than 0.25 V against Li/Li\textsuperscript{+}, giving rise to capacity fading of FLG, primarily in the that higher potential window.
Experimental and Theoretical Study of Doping in Li$_2$FeSiO$_4$

Shivani Singh$^a$, Anish Raj K$^a$, Priya Johari$^{a,b}$ and Sagar Mitra$^{a,*}$

$^a$Electrochemical Energy Laboratory, Department of Energy Science and Engineering, Indian Institute of Technology Bombay, Powai, Mumbai, 400076, India
$^b$Department of Physics, School of Natural Sciences, Shiv Nadar University, Gautam Budh Nagar, Greater Noida, Uttar Pradesh-201314, India.

Corresponding author’s email id: sagar.mitra@iitb.ac.in, priya.johari@snu.edu.in

Abstract

Multi-electron charge transfer leads to higher theoretical capacity in polyanionic based silicates (Li$_2$FeSiO$_4$), resulting in almost double discharge capacity i.e. 333 mAh g$^{-1}$ compared to commercially available alternatives. These materials are greener, safer and lower in cost since Fe and Si are available in abundance in earth’s crust. Li$_2$FeSiO$_4$ can be synthesized experimentally in three different space groups {($Pmn_2_1$, $Pmn_\beta$ (orthorhombic) and $P_2_1/n$ (monoclinic)} by varying underlying experimental conditions such as calcination time and temperature [1-6]. However, low electrical conductivity and inability to extract two lithium ions completely from the crystal structure are the main obstacles for its commercialization. Electrical conductivity can be triggered by utilizing conductive carbon additives [7] along with nanosizing the active materials [8]. Efforts have been undertaken in the present work to improve the electrochemical activity and power performance by nanosizing the particles (Li$_2$FeSiO$_4$) and making a composite with multi-walled carbon nanotubes (MWCNTs) as conductive additives. As synthesized Li$_2$FeSiO$_4$ spherical particles (70-120 nm) are composed of primary nanocrystals (5-10 nm). The bulk and surface properties of Li$_2$FeSiO$_4$ have been characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), Fourier transformed infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS) and Raman spectroscopy.

The first discharge capacity at the current rate of 16.5 mA g$^{-1}$ is 223 mAh g$^{-1}$, indicating the reversibility of 1.35 Li$^+$ ion. The capacity decreases to 126 mAh g$^{-1}$ after fifty cycles. Whereas, the first discharge capacity at the current rate of 33.2 mA g$^{-1}$ is 169 mAh g$^{-1}$ which decreases to 122 mAh g$^{-1}$ after fifty cycles. The battery loses ~ 41% of its initial discharge capacity after 50 cycles at the rate of 16.5 mA g$^{-1}$ in potential window of 1.5-4.8V at 20 °C, whereas this loss is around 27% when cell is run at the higher current rate of 33.2 mA g$^{-1}$. Electrochemically driven phase transformation along with structural instability associated with the Fe$^{4+}$ and formation of O-2p ligand hole influences electrode performance in lithium iron silicate cathode material, which results in to the capacity fading in subsequent cycles. The capacity fading is also associated with the electronic properties of the material.

So, in order to understand the impact of electronic properties on electrochemical performance we have undertaken anionic doping in Li$_2$FeSiO$_4$ that could enhance the electronic conductivity of the electrode and concurrently enhanced the electrochemical performance. The doping has been confirmed by XPS analysis in doped sample. Moreover, the distribution of doping has been
confirmed by elemental mapping which confirms uniform distribution of dopant. Finally, the electrochemical characterization has been performed for doped sample which results in to improved performance in case of doped sample compared to pristine material.

References


Real-Time Stress Measurement during Electrochemical Cycling of Sn and Sn-Cu Composite Thin Film Electrodes for Li-Ion Batteries

Ravi Kali, Yaadhav Krishnan, Amartya Mukhopadhyay

High Temperature and Energy Materials Laboratory, Department of Metallurgical Engineering and Materials Science, IIT Bombay, Powai, Mumbai 400076, India
Corresponding author’s email id: amartya_mukhopadhyay@iitb.ac.in

Abstract

Sn is a promising candidate to replace graphitic carbon as anode material for Li-ion batteries due to its lower cost, higher Li-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. In order to improve the cyclic performance of Sn electrode, several methods have been reported, one possible method is that Sn-Cu intermetallic, where Cu acts as buffer matrix which forms a stable structure and suppresses the volume expansion during the reaction. But the specific capacity of such intermetallics (with ~200-250 mAh/g usually achieved) is lesser even compared to commercial graphite electrode. Against this backdrop, we report here the development of Sn/Sn-Cu based
‘composite’ thin film electrodes, having desired microstructure-type, via simple annealing treatment of as-deposited Sn on Cu. Furthermore, the simple thin film electrode architecture also allowed monitoring of the in-plane stress developments in-situ during electrochemical lithiation/delithiation, by monitoring the substrate curvature using multi-beam optical sensor (MOSS), during electrochemical cycling against Li metal in custom-made electrochemical cell (for the first time with Sn/intermetallic electrodes); throwing some valuable insights into the suppressed mechanical degradation and significantly improved cyclic stability for such electrodes (developed via simple heat treatment), as compared to the pure (as-deposited) Sn electrodes. The experiments have been performed with Sn thin film electrodes (~ 200 nm thick), deposited via e-beam deposition, on Cu foil (~30 µm thick), as well as on thicker (~ 0.5 mm) and stiff quartz substrate coated with Cu (~ 100 nm), acting as current collector. In order to form the Cu-Sn intermetallic phase, the as-deposited Sn films were heat treated at 250°C for 2 h in Ar atmosphere. Such heat treatment resulted in the formation of Sn-Cu intermetallic phases (due to reaction with the Cu foil), which co-existed with the ‘unreacted’ Sn. Important insights into the effects of the presence of the Sn-Cu intermetallic phases on such improved mechanical integrity have been obtained upon monitoring the stress developments in-situ during galvanostatic cycling. Such real-time monitoring indicated that the overall stress magnitudes recorded upon full lithiation of the Sn/intermetallic based electrodes were lower than the corresponding values for as-deposited Sn by factor of ~3. Furthermore, no signature for mechanical degradation could be observed in the stress-time profiles recorded with the annealed Sn/intermetallic electrodes, unlike those for the as-deposited Sn electrodes.

Keywords: Intermetallic, annealing, in-situ stress measurement, electrochemical behavior

PXV

Synthesis Optimization of LiNi1/3Co1/3Mn1/3O2 Cathode Material for Li-Ion Batteries

Evgeny Legotin, Atanaska TRIFONOVA

AIT Austrian Institute of Technology GmbH, Mobility Department, Electric Drive Technologies, Austria

Corresponding author’s email id: evgeny.legotin.fl@ait.ac.at

Abstract

NMC333 (LiNi1/3Co1/3Mn1/3O2) is considered as one of the most perspective cathode materials for high-power lithium-ion batteries. Commercial NMC cathode materials are nowadays manufactured in time-consuming and expensive multi-step ways. The aim of this work is to optimize the conditions of NMC synthesis to adjust it for single-step large-scale production. In this study, NMC was synthesized by two different wet-chemical techniques, viz. solvent evaporation method and spray pyrolysis. Either respective metal chlorides or nitrates were used as precursors.
In the spray pyrolysis method, the characteristics of the desired material can be controlled via tuning the process parameters such as temperature or spraying rate [1,2]. Process parameters were optimized to produce a commercially attractive material. The stoichiometry of the materials was confirmed via X-ray fluorescence spectrometry (XRF) and Inductively coupled plasma atomic emission spectrometry (ICP-AES). Morphology, particle size distribution, surface area, porosity and crystallographic characteristics of the obtained powders were studied.

It seems that the production of the lithiated layered material from chloride precursors can’t succeed as a “one pot” synthesis. However, the above material can be produced in a two-step process, including spray pyrolysis production of the mixed Ni-Co-Mn oxide precursor and its subsequent lithiation. In contrast, using nitrate precursors was shown to enable production of NMC in only one step, which is feasible for industrial scale manufacture.

Keywords: Li-ion battery, NMC, spray pyrolysis, production scale-up

Acknowledgements:
We are grateful to the Austrian Research Promotion Agency (FFG) for financial promotion of this study.

References:

PXVI

Electrocatalytic Properties of Carbon Coated Molybdenum Carbide (Mo2C) Nano Powders Synthesized via Solvothermal Route

Rameez Ahmed Mir, O.P. Pandey

School of Physics and Materials Science, Thapar University, Patiala-147004
Corresponding author’s email id: mirrameezee@gmail.com

Abstract
Carbon coated Mo2C nano powders were synthesized through simple reaction of molybdenum tri-oxide (MoO3), magnesium (Mg) as reducing agent and high density polyethylene (HDPE) as carbon source in an autoclave. Rietveld refinement of XRD data confirmed the formation of stable hexagonal Mo2C (P63/mmc) phase. Reaction temperature as well as reaction time facilitated the reduction and carburization of MoO3. The synthesized samples were characterized by TGA to estimate the thermal stability in air atmosphere. The morphological features of synthesized samples has been analyzed with the help of FESEM and TEM. Electrocatalytic tests
were performed in acidic medium and results have been analyzed through cyclic voltammetry (CV) plots and results predict that synthesized powders can be used as better catalyst and electrodes for various electro oxidation reaction. These results also confirmed that this simple method is as effective way to synthesize active carbide catalyst.

**Abstract**

Over the past decades Lithium – Air batteries attracted much attention as a promising successor of current lithium-ion technology, due to its high theoretical energy capacity similar to fossil fuels. Since the first proof of recharge ability by Abraham and Jiang [1], much research has been presented in order to improve its overall performance, but many problems still arise from the stability of electrolytes and effective catalysts for oxygen evolution (OER) and oxygen reduction (ORR) [2,3].

We studied of Manganese-, Iron- and Palladium- octaethyl porphyrins as catalysts for OER and ORR in Li-Air cells with non-aqueous electrolytes. Since the electrical conductivity of these materials is low, mixing with active carbon followed by thermal treatment was performed to obtain the final catalyst composite. On chosen materials SEM and XRD as well as EIS, LP and CV investigations were conducted. The obtained electrochemical results show good catalytic properties for ORR and OER activity with Mn and Pd species. SEM images show the formation of a complex surface after the heat treatment of the composite materials. X-Ray diffraction revealed the formation of a graphite-like structure together with metal oxides particles in the case of MnClOEP and FeClOEP which results were supported by XPS data and will be presented at this conference.

**Keywords:** lithium-air, porphyrins, bi-functional catalysts

**Acknowledgement:**

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**References:**


**Investigating Thermal Kinetics Involved in the Formation of Lithium Titanate through Thermogravimetry Technique**

Piyush Sharma, O.P. Pandey

School of Physics and Materials Science, Thapar University, Patiala, India

Corresponding author’s email id: piyush.sharma@thapar.edu

**Abstract**

Thermal kinetics of solid state reactions involved in the formation of lithium titanate has been studied through thermogravimetry (TGA) technique. The powder mixture of lithium carbonate and titanium dioxide was heated to 1000 ºC at different heating rates, in nitrogen environment. Various kinetic parameters such as activation energy, pre-exponential factor and co-relation factor were evaluated by employing Flynn-Wall-Ozawa method. It was observed that the average activation energy and pre-exponential factor were 214.52 kJ mol⁻¹ and 4.96×10⁻¹⁰ min⁻¹, respectively. In order to determine reaction mechanism, master curves corresponding to different mechanism were generated though Criado method and compared with experimental curve. The results revealed that the reaction mechanism was three-dimensional diffusion D3 mechanism. The details of these are presented in this work.

**Electrochemical Behaviour of ‘Ultra-fine’ Electrode-Active Nanoparticles Decorating/Coating Carbon Nanotubes for Li-Ion and Na-Ion Batteries**

Hem Shruti Bhardwaj, Mahesh K. Satam, Peter C. Joseph, Thrinathreddy Ramireddy, Amartya Mukhopadhyay

High Temperature and Energy materials Laboratory, Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay (IITB), Mumbai 400076, India

†Presenting author: Corresponding author’s email id: hemshruti@gmail.com
Corresponding author’s email id: amartya_mukhopadhyay@iitb.ac.in

**Abstract**

Some of the major drawbacks of ceramic electrode materials for Li/Na-ion batteries include inferior rate capability and cyclic stability. Incorporation of conducting reinforcements like multi-walled carbon nanotubes (MWCNTs) is a promising avenue to address such issues.
However, physical mixing of electrode-active particles with MWCNTs leads to inhomogeneous distribution and poor contact; whereas vapour deposition routes are not scalable. Against this backdrop, we have developed electrode materials characterized by electrode-active ‘ultra-fine’ nanoparticles uniformly decorating/coating MWCNTs via innovative, but facile, wet-chemical synthesis based route (sans physical mixing). The superior electrochemical performances of such electrodes based on LiFePO$_4$, SiO$_2$ and Na$_2$Ti$_3$O$_7$ nanoparticles (decorating MWCNTs), primarily in terms of enhanced rate capability, cyclic stability and reduced voltage hysteresis, will be presented. The discussion will lead to better insights into some of the fundamental aspects (including first-order phase transformations during charge/discharge) at such ‘ultra-fine’ nanoscaled dimensions (~5-10 nm), in the presence/absence of MWCNTs (in intimate contact).

**PXX**

**Facile and Sustainable Synthesis of Different Shapes of Zinc Oxide Nanoparticles: Morphology Dependent Photocatalytic Activity**

Sameer Ulhas Hadkar, Sayyed Arzuman Nisa, Santosh A. Mani, Pradip Sarawade

Department of Physics, University of Mumbai, Mumbai, India

Corresponding author’s email id: sameer.u.hadkar@gmail.com

**Abstract**

This works report a cost-effective and sustainable protocol for synthesis of two different shaped zinc oxides nanoparticles. We observed that two different shapes of zinc oxide can be synthesized using exactly same synthetic protocol by simply changing the precursor zinc salt. Simply changing the precursor zinc salts, we have synthesized nano-sized ZnO nano-rods. The crystalline structure of the prepared material was confirmed by X-Ray powder Diffraction (XRD) technique. The surface morphology of ZnO nanoparticle were imaged with the help of Scanning Electron Microscopy (SEM) technique. The surface area was analyzed using Brunauer – Emmett - Teller (BET) technique, and Fourier Transform Infrared Spectroscopy (FTIR). The ZnO$_2$/UV photocatalytic degradation of methylene blue (MB) has been investigated in aqueous heterogeneous suspensions. These results suggest that ZnO$_2$/UV photocatalysis may be useful method for treatment of diluted waste waters in textile industries.

**Keywords:** zinc oxides nanoparticles, ZnO nano-rods, XRD, SEM, FTIR, BET, photocatalysis
pH Dependent Morphology of Ammonium Vanadium Oxide and its Electrochemical Performance on Sodium Ion Battery

Ananta Sarkar, Sagar Mitra

Electrochemical Energy Laboratory, Department of Energy Science and Engineering, Indian Institute of Technology Bombay, Powai, Mumbai 400076, Maharashtra India
Corresponding author’s email id: Ananta2k10@gmail.com, Sagar.mitra@iitb.ac.in

Abstract

Sodium ion battery is most alternative advanced technology for portable electronics devices. In our present study, we are dealing with a suitable cathode material for sodium ion battery which can deliver high capacity as well as good stable cyclic performance. Now a day’s vanadium based compounds has great attention towards sodium ion battery due to layer structured, huge inter-planner spacing, its rich chemistry (multiple oxidation reduction stage), cheap and easily available, high capacity, good structural stability. Not only that this vanadium based cathode materials can easily prepared to our desired nano-dimensional shape and size. Layer structure ammonium vanadium oxide was prepared by single step hydrothermal process at different pH. It was clearly observed that as the pH of the solution was increased up to 3 the pure phase of monoclinic ammonium vanadium oxide (NH₄V₄O₁₀) was form. Again further increased of pH of the solution the the initial peaks at 9 degree 2 theta was disappeared and a new sharp peaks at 15 degree 2 theta position is appear. The growth of the materials at lower Ph (<3) is observed in (00l) direction whereas at higher Ph the growth of the materials is happen in (h0l) or (hkl) direction also conform by FEG-SEM.

From the charge/discharge profile, it is observed that at very low pH ~1.5 and at higher pH, the materials does not show any discharge capacity. At ph 4, the initial discharge capacity of the materials was only about 50 mAh g⁻¹, however upon cycliny the specific discharge capacity is increased up to 100 mAh g⁻¹ after 45 cycles. Where as at pH 2, we observed stable cycling performance, having initial discharge capacity ~ 100 mAh g⁻¹ and after 45th cycle ~ 93 mAh g⁻¹. However we observe the best cycling performance of the materials made at pH 3. The initial discharge capacity was ~ 142 mAh g⁻¹ and after 45th cycle almost 92% (130 mAh g⁻¹) capacity was retained with out any addition of expensive electrolyte.
Abstract

Controlled and local release of drugs (growth factors and therapeutics) from three dimensional (3D) porous scaffolds are important for maintenance of cell survival, cell proliferation and promotion of tissue regeneration. Porous scaffolds of controlled pore structure were developed as an efficient and robust material platform for homogeneous tissue regeneration. Furthermore, precise integration of controlled drug delivery structures such as microspheres or microgels or therapeutic nanoparticles into those porous scaffolds can yield a designer scaffold of specific interest that can be useful for cocktail of functions such as tissue regeneration, drug therapy, drug (nanomedicine) screening and so on. In this talk, I will introduce the preparation of few kinds of micro-and-nano therapeutic impregnated designer polymer scaffolds of controlled pore structure for application in controlled drug delivery in tissue engineering and nanomedicine screening. Porous scaffolds were prepared using freeze-drying technique by precise integration of drug loaded microspheres/microgels/therapeutic nanoparticles in polymer/hybrid polymeric matrix of controlled pore structure. The pore structure was controlled using pre-prepared ice microporogen prepared from deionized water or natural polymers. The scaffolds were studied for regeneration of skin, cartilage and bone defects as well as an in vitro tumor model for drug (nanomedicine) screening.

Keywords: controlled release; drugs, porous designer scaffold; ice microporogen; nanomedicine
Invited Lecture:

**Tribocorrosion and Electrochemical behaviour of Nanocrystalline Diamond coated Ti based alloys for Orthopaedic Application**

Vasanth Gopal\textsuperscript{a}, Maneesh Chandran\textsuperscript{b}, M. S. Ramachandra Rao\textsuperscript{b}, Stefano Mischler\textsuperscript{c}, Shoufan Cao\textsuperscript{c}, Geetha Manivasagam\textsuperscript{d}*

\textsuperscript{a}Department of Physics, School of Advanced Sciences, VIT University, Vellore 632014, India
\textsuperscript{b}Materials Science Research Centre, Department of Physics, IIT Madras, Chennai, India
\textsuperscript{c}Tribology and Interface Chemistry Group, Ecole Polytechnique Federale de Lausanne (EPFL), Switzerland
\textsuperscript{d}Centre for BioMaterials Science and Technology, School of Mechanical & Building Sciences, VIT University, Vellore 632014, India.

Corresponding author’s email id: gmv1225@yahoo.com

Abstract:
The unique properties of nanocrystalline diamond (NCD) coatings such as high wear resistance and chemical inertness makes it a potential candidate for coatings on orthopaedic implants such as hip and knee implants. However, the tribocorrosion behaviour of NCD coatings is unexplored till date. In this work, we studied the tribocorrosion behaviour of NCD coatings deposited on Cp-Ti, Ti-6Al-4V and Ti13Nb-13Zr using hot filament chemical vapour deposition (HFCVD) method. Surface topography, phase, surface roughness and hardness of the NCD coatings were characterized by scanning electron microscope, X-ray diffraction, Raman spectroscopy, atomic force microscopy and nanoindentation respectively. The experiments were conducted at open circuit potential using a reciprocating sliding tribocorrosion apparatus. In addition, the electrochemical behaviour of the NCD coatings was assessed by the potentiostat. The obtained results demonstrate that all the NCD coated samples exhibited nobler potential and lower corrosion rate than the bare Ti alloys. The open circuit potential of NCD coatings under tribocorrosion condition shows minimal potential drop at the start of sliding and then decreased progressively with time during sliding.

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**Effect of Water and Bone Marrow Removal on Tensile and Compressive Strength of Cortical Bone**

Praveer Sihota, Ram Naresh Yadav, Navin Kumar#

Nano-Bio-Mechanical Characterization laboratory, Department of Mechanical Engineering, Indian Institute of Technology Ropar, Rupnagar-140001, India

Corresponding author’s email id: nkumar@iitrpr.ac.in

Abstract

Mineral and collagen are two important constituents of bone; each play a significant role towards load bearing strength such as mineral confirms the strength and collagen is responsible for
toughness. Water is the third important constituent in bone which interacts with mineral and collagen both, but the quantitative analysis for this effect is still under research. To determine the bone strength, toughness and stiffness, tensile and compressive test have been performed on hydrated and dehydrated cortical bone. This study is useful to understand the role of water in bone strength and toughness.

**Key words:** Bovine bone, mechanical properties, tensile test, compressive test, water

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**AC magnetic heating study of water dispersible Fe₃O₄ magnetic nanoparticles**

S. K. Sharma, R. S. Ningthoujam, R. K. Vatsa

Chemistry Division, Bhabha Atomic Research Centre, Mumbai-400085, India

Corresponding author’s email id: rsn@barc.gov.in

**Abstract**

Magnetic nanoparticles (MNPs) have been used in many biomedical applications such as separating agents for biomolecules, magnetic resonance imaging (MRI) contrast agent, drug delivery vehicles and hyperthermia for cancer therapeutic agents. Fe₃O₄ particles coated (particle size ~ 15 nm) with oleic acid was dissolved in dichloromethane and cooled to the 0 °C. On it, dried 3-chloroperbenzoic acid in dichloromethane is added drop wise to the cooled solution with stirring. The particles were stirred at the room temperature for 3 hours. This process converts the double bond of oleic acid to epoxide. The particles are removed by centrifugation and washed with methanol. The particles are dissolved in dimethyl formamide and then aspartic acid is also added. It is heated to 60 °C for 4 hours. This converts epoxide to two functional groups (OH and amino succinic acid), which help in dispersion of particles in water. Particles are collected by centrifugation and washed with methanol. The particles are characterized by IR spectroscopy. Heating effect of the particles on application of an external AC magnetic field is measured for 10 mg/mL aqueous solution. The particles (5-10 mg/ml) are able to reach the hyperthermia temperature (43 °C) within 5-10 minutes in 400 A (335 Oe) and at 265 kHz. The heating efficiency can be increased after increase of particle size as well as by increasing applied currents/magnetic fields.
Peptide Dendrimer-Fe$_3$O$_4$ Nanoparticles as Contrast Agents in Non-Invasive Magnetic Resonance Imaging

Saumya Nigam$^1$, D. Bahadur$^2$

$^1$IITB-Monash Research Academy, IIT Bombay, Powai, Mumbai, India
$^2$Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology
Bombay, Powai, Mumbai, India
Corresponding author’s email id: dhirenb@iitb.ac.in

Abstract

Superparamagnetic iron oxide (Fe$_3$O$_4$) nanoparticles have recently found an appreciable place as $T_2$ contrast agents in the magnetic resonance (MR) imaging of cancer. Their surface modification by dendritic macromolecules affect their relaxation times and play an important role in their efficacy as MR contrast agents. This work demonstrates the fabrication of a peptide dendrimer used in subsequent engineering of Fe$_3$O$_4$ nanoparticles as an effective $T_2$ contrast agent for non-invasive MR imaging \textit{in vitro}. The fabricated system was evaluated for its comparative $r_1$ and $r_2$ relaxivity under different environmental parameters using 9.4 Tesla small animal MRI. The effect of temperature, buffer solutions and iron concentration was assessed by investigating the magnetic properties and induced MR signals. The MR measurements indicate a significant increase in the transverse relaxation and relaxation ratios of the dendritic nanosystem. Both $T_1$- and $T_2$- weighted phantoms showed significant change in signal intensity with an increasing amount of Fe concentration. This indicates that these dendritic Fe$_3$O$_4$ generate MR contrast on both longitudinal ($T_1$) and transverse ($T_2$) proton relaxations-times weighted sequences due to the dipolar interaction of magnetic moment of the particles and protons of the water. The relaxation ratio ($r_2/r_1$) of was seen to be 100.7 in an aqueous environment as against 62.3 in buffer environment at 25 °C. This difference in the contrast properties and relaxivity was seen to be largely dependent on the temperature and buffer ions in the surrounding microenvironment. These dendritic Fe$_3$O$_4$ were also employed for the $T_2$-weighted MR imaging of human cervical cancer (HeLa) cells \textit{in vitro} after various time intervals over a period of 24 h.

Figure 1. (a) $T_2$ relaxation of dendritic-Fe$_3$O$_4$ in simulated body fluid (b) dependence of transverse relaxivity ($r_2$) of dendritic-Fe$_3$O$_4$ on iron concentration and temperature
Surface Functionalized Prussian Blue Encapsulated Nickel Oxide Nanospheres on Flexible Substrate for Bio-Sensing Application

Appan Roychoudhury 1, Arneish Prateek 2, Suddhasatwa Basu 2, D. Sakthi Kumar 3, Sandeep Kumar Jha 1

1 Centre for Biomedical Engineering, IIT Delhi, New Delhi 110016, India
2 Department of Chemical Engineering, IIT Delhi, New Delhi 110016, India
3 Bio-Nano Electronics Research Centre, Toyo University, Saitama 350-8585, Japan

Corresponding author’s email id: appan.roychoudhury@gmail.com

Abstract

A dopamine biosensor has been developed using prussian blue (PB) modified nickel oxide (NiO) nanoparticles (NPs) and tyrosinase enzyme conjugates. Dopamine is one of the most important catecholamine neurotransmitter that shows significant role in the functions of central nervous, renal and hormonal systems. Abnormal concentrations of dopamine in body can lead to Parkinson’s disease, schizophrenia and other neurological disorders.

A wet chemical sol-gel method was used to synthesize NiO NPs using sodium dodecyl sulphate as anionic surfactant and the surface of the synthesized NiO NPs was modified with prussian blue to enhance electrocatalytic activity. The structural and morphological studies of the developed NPs were carried out using X-ray diffraction (XRD), high resolution transmission electron microscopy (HR-TEM), Raman and UV-Vis spectroscopy. The average crystallite size of the prepared PB-NiO NPs was found as 15 nm from XRD studies and average particle size was varied from 20 to 25 nm obtained from HR-TEM micrographs. The purity of the sample was confirmed through X-ray photoelectron spectroscopy (XPS), energy dispersive X-ray spectroscopy (EDX) and Fourier transform infrared spectroscopy (FTIR) studies and deposited onto flexible screen printed carbon electrode (SPCE) substrate to use as a matrix to immobilize enzyme and to explore its bio-sensing characteristics. The tyrosinase enzyme molecules were immobilized onto PB-NiO NPs functionalized SPCE surface by covalent immobilization for selective detection of dopamine and the enzyme immobilization was confirmed through scanning electron microscopy (SEM) and FTIR studies. The electrochemical detection and estimation of dopamine was conducted using fabricated Tyrosinase/PB-NiO/SPCE electrode by cyclic voltammetry and chrono-amperometry methods and the electrochemical response studies reveal linearity in broad detection range of 0.5-100 μM and high sensitivity of 0.91 μA/μM with linear regression of 0.99. The developed dopamine sensor also shows a fast response time of 24 seconds, decent selectivity in presence of interferents ascorbic and uric acid, long shelf life of 45 days with good reusability and validated with real samples.
**Ultrasensitive biosensing of antimalarial drug using polypyrrole doped gold nanorods modified Au electrode**

Utkarsh Jain, Nidhi Chauhan

Amity Institute of Nanotechnology, Amity University, Noida - 201303, Uttar Pradesh, India
Corresponding author’s email id: karshjain@gmail.com

**Abstract**

Bioactive electrode of polypyrrole doped gold nanorods (GNRs) was electrochemically fabricated onto Au electrode for pharmaceutical application. Synthesized GNRs were characterized using Dynamic light scattering (DLS) and UV spectroscopy for the absorption peaks. Electrochemical interfacial properties and immobilization of enzyme onto the GNR-Ppy Au electrode have been evaluated and confirmed with the use of cyclic voltammetry and electrochemical impedance spectroscopic techniques. The peroxidise/GNR-Ppy based biosensor was further used for sensing artemether a potent antimalarial drug. The biosensor shows linearity of 0.02–5.0 ng mL\(^{-1}\) of artemether. The procedure was applied to the assay of the drug in dosage form. The limits of detection for parenteral artemether was 0.010 ng mL\(^{-1}\). The mean percentage recoveries obtained were in the range from 95.2% to 97.5% for parental drug. The resultant precision and accuracy as evidenced have shown a promising selectivity in their application.

**Keywords:** Antimalarial drug, peroxidise, gold nanorods, polypyrrole, biosensor

**Mesoporous Cu-Substituted Zno Nanoassembly with Enhanced Photocatalytic, Antibacterial and Anticancer Performances under Visible Light**

Jagriti Gupta, D. Bahadur*

Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Mumbai – 400076, India
Corresponding author’s email id ID: dhirenb@iitb.ac.in

**Abstract**

Visible-light driven photosensitizers have been recognized for their potential utility for various applications especially in nanomedicine. The aim of this study is to investigate a new strategy to substitute Cu ions in ZnO nanoassemblies for induction of photodynamic effect under visible light irradiation. Here, we report the synthesis of Cu substituted ZnO nanoassembly (Cu-ZnO NAs) with optimized Cu concentration, required for enhanced photosensitive performance for sustained anti-bacterial and anti-cancer activity under dark conditions as well as visible light irradiation. It is noted that the substitution of Cu in ZnO nanoassemblies remarkably improves its...
absorption properties, charge separation efficiency as well as ROS level that make it more appropriate for photodynamic therapy under visible light irradiation for killing of bacterial and cancerous cells. The generated ROS causes a significant decrease in cell viability as well as mitochondrial membrane potential. Moreover, Cu-ZnO NAs shows ROS induced cellular apoptosis, DNA fragmentation, and depolarization of mitochondrial membrane and cell cycle arrest in S phase. These characteristics enable the use of these NAs as a photosensitizer in cancer therapy. To the best of our knowledge, this is the first report on bacterial killing and cancer therapy using Cu-ZnO system under visible light irradiation.

**Keywords:** Nanostructure, Photocatalysis, Antibacterial activity, Photodynamic therapy, Cu-ZnO.

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**Surface Modification of Bio-Implant Material for Enhanced Corrosion Resistance**

Gopi Perumal, H. S. Grewal, H. S. Arora

Department of Mechanical Engineering, Shiv Nadar University, Gautam Budh Nagar, India

Corresponding author’s email id: harpreet.arora@snu.edu.in

**Abstract**

Stainless steel (SS316L) is widely used in health-care as a bio-implant due to its excellent biocompatibility, superior wear resistance and high resistance to localized corrosion in chloride environments. However, degradation by wear and corrosion is still reported to be one of the prime reasons for failure of an implant material. Therefore, improving the corrosion resistance of implant materials can significantly enhance their performance and likely to have large economic implications. Surface modification is a viable technique for addressing degradation problems in materials. In the current work, Friction stir processing (FSP), was used to tailor the surface characteristics of SS316L. FSP was done under controlled cooling conditions with strain rates being varied over a wide range. Microstructural studies were done using electron back scatter diffraction which revealed ultra-fined grain structure for samples processed under rapid cooling conditions. Corrosion studies were done using electrochemical techniques comprising potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) with ringer solution as an electrolyte. The increased breakdown potential, reduced hysteresis loop and current density indicate improved pitting and crevice resistance for processed samples. The study provides a platform for design and development of advanced implant materials for various clinical applications.

**Key Words:** Friction Stir Processing, Microstructural refinement, Corrosion
Detection and Application of Glycated Hemoglobin with Voltammetric Sensing Amplified by 3D-Structured Nanocomposites

Nidhi Chauhan, Shaivya Gupta, Utkarsh Jain
Amity Institute of Nanotechnology, Amity University, Noida - 201303, Uttar Pradesh, India
Corresponding author’s email id: nidhichauhan2007@rediffmail.com

Abstract
Glycated hemoglobin (HbA1c), a good marker for glycine level in blood, has shown consistency over a very long period of time (up to two to three months). Hence HbA1c has been mostly used and is indeed an established test for monitoring the glycaemic control in persons suffering from diabetes. A 3D-structured reduced graphene oxide (rGO), multiwalled carbon nanotubes (MWCNT) and platinum nanoparticles (NPs) composite (rGO–MWCNT–Pt) was synthesized by a facile and green way and used as interface for the development of an electrochemical biosensor for HbA1c detection. The network structure of rGO–MWCNT nanocomposite provides more active sites for Pt deposition and the synergistic effect of rGO, MWCNTs and PtNPs significantly improved the electrochemical performance of the modified electrode. PtNPS can combine HbA1c and enhance the conductivity of working electrode. The structure of rGO–MWCNT–Pt nanocomposite was characterized by scanning electron microscopy (SEM) and electrochemical Impedance Study (EIS). Fructosyl valine oxidase (FAO) is immobilized onto the nanocomposite via amide bonds. Cyclic voltammetry was used to characterize the recognizability of HbA1c. Under optimum experimental conditions, the proposed biosensor exhibited a low detection limit, a wide linear range as well as good stability and reproducibility.

Keywords: Biosensor, glycated hemoglobin, graphene oxide (rGO), multiwalled carbon nanotubes (MWCNT) and platinum nanoparticles

Enhanced Anticancer Efficacy of Folate-Grafted Lipid Modified Dual Drug Loaded Nanoassemblies to Reduce Drug Resistance in Ovarian Cancer

Lina Pradhan, Rohit Srivastava, D. Bahadur

Abstract
Ovarian cancer is a major cause of global gynaecological cancer-related mortality. Platinum-based drugs (cisplatin and carboplatin) are limited in their clinical effectiveness for drug resistant
ovarian cancer. Cisplatin resistant cancer can in concept be treated with paclitaxel, doxorubicin and etoposide drugs, but prove toxic to many patients. Recently, researchers have focused on nanotechnology based targeted therapies, representing a new therapeutic approach, intended to maximize intracellular drug uptake and minimize drug resistance. In this regard, folic acid (FA) conjugated lipid coated mesoporous magnetic nanoassemblies (FA-LMMNA) synthesized for targeted delivery of Doxorubicin and Paclitaxel (DOX:TXL-FA-LMMNA) has been investigated herein. This formulation provides specific targeting effect by folate conjugation, improving the anti-cancer efficacy on cisplatin sensitive and resistant ovarian cancer cells (A2780\textsuperscript{S} and A2780-Cis-Res). The lipid coating and surface conjugation of FA on mesoporous magnetic nanoassemblies (MMNA) has been confirmed by TEM and zeta potential analyzer. We observed that DOX:TXL-FA-LMMNA significantly enhanced anticancer efficacy as compared to dual drug encapsulated lipid modified mesoporous magnetic nanoassemblies (DOX:TXL-LMMNA). Through ICP-AES, fluorescent microscopy and flow cytometry studies we have confirmed a significantly higher in vitro cellular uptake and targeting efficacy of DOX:TXL-FA-LMMNA for the A2780\textsuperscript{S} and A2780-Cis-Res (folate receptor positive (FR\textsuperscript{+ve})) cells due to overexpression of FR on these cell lines. In case of FR negative (FR\textsuperscript{-ve}) prostate cancer (PC3) and normal fibroblast (L929) cells, no significant difference owing to FA was observed. These results suggest that DOX:TXL-FA-LMMNA has the potential activity for targeted delivery of dual drugs for treating cisplatin sensitive and resistant ovarian cancer.

**Keywords:** Magnetic, Mesoporous, Dual drug, Drug resistance, Folic acid, Targeting chemotherapy

Schematic diagram demonstrating folate mediated targeting drug delivery to FR-positive (FR\textsuperscript{+ve}) cisplatin resistant ovarian cancer (A2780-Cis-Res) and FR-negative (FR\textsuperscript{-ve}) prostate cancer (PC3) cells using dual drug encapsulated folate grafted lipid coated mesoporous magnetic nanoassemblies (DOX:TXL-FA-LMMNA). In this paper, the proof of formulation and in vitro targeting affinity of DOX:TXL-FA-LMMNA against FR\textsuperscript{-ve} and FR\textsuperscript{+ve} cancer cells are reported.
References:

