2009 Functional and Structural Nanomaterials: Fabrication, Properties, and Applications: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Nanomaterials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Gregory Thompson, University of Alabama; Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute; Jiyoung Kim, University of Texas at Dallas; Seong Jin Koh, University of Texas at Arlington; Wonbong Choi, Florida International University; Alexander Howard, Air Force Research Laboratory

Sunday, 6:00-8:00 PM Room: 3018
February 15, 2009 Location: Moscone West Convention Center

Session Chairs: Gregory Thompson, University of Alabama; Jiyoung Kim, University of Texas at Dallas; Alexander Howard, Air Force Research Laboratory; Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute; Seong Jin Koh, University of Texas at Arlington; Wonbong Choi, Florida International University

Bending Strength of Single Crystal Silicon Micro Beams Fixed on Both Ends: *Guangping Han*[1]; Gaoping Wang[2]; Xiuhong Wang[1]; Kai Liu[3]; [1]Zhengzhou Institute of Aeronautical Industry Management; [2]Henan University of Technology; [3]Xi'an University of Technology

MEMS is a rapidly growing multidisciplinary technology, in which understanding of the mechanical behavior of materials and micro structures lags far behind micro-fabrication and application of micro devices, thus, accurate evaluation of the mechanical properties of material is one of the most challenging issues. Six kinds of single crystal silicon micro beams fixed on both ends with trapezoidal cross section were fabricated using photolithography technology, with dimensions of 150-1000 μm long, 16-60 μm wide, 6 and 20 μm thick. The micro beam specimens were used in bending test by nano indentation method. Results show that the mean values of bending strength increase with the ratio of surface area to volume, varying from 3.24 to 10.15Gpa; based on weibull analysis, shape parameters are 4.21-10.54 while scale parameters under the fracture probability of 65.4% vary from 3.26 to 10.26Gpa; both the average bending strength and weibull parameters display strong size effects.

Characteristic of the Liquid Sodium by the Dispersing Nanoparticles: *Jun-ichi Saito*[1]; Kuniaki Ara[1]; [1]Japan Atomic Energy Agency

The purpose of this study is to suppress the high chemical reactivity by dispersing nanoparticles into liquid sodium. An atomic interaction between the nanoparticle atom and sodium atom is harnessed to suppress the chemical reactivity. The theoretical calculation showed the atomic bonding between nanoparticle atom and sodium atom was stronger than that between sodium atoms. The charge transfer occurred to the nanoparticle atom from the sodium atom. It suggests that the fundamental and reaction properties change by the atomic interaction. The fundamental property of sodium dispersing nanoparticle changed compared to sodium. The reaction behavior with water or oxygen of the sodium dispersing nanoparticles also changed compared to sodium. The reaction heat of the sodium dispersing nanoparticles reduced. It means that there is the possibility of suppression of reactivity of liquid sodium by the atomic interaction.

Characterization of Noble Metal Core Shell Nanostructures via Scanning Probe Microscopy and Ab Initio Calculations: *Aniketa Shinde*[1]; Juexian Cao[1]; Sangyeob Lee[1]; Chulsu Jo[1]; Ruqian Wu[1]; Regina Ragan[1]; [1]University of California, Irvine

Self assembled rare earth disilicide nanowires are used as templates for Pt and Au nanostructures on Si(001). We performed experimental and computational studies to investigate the adsorption of rare earth atoms on the Si(001) surface and the onset of disilicide nanowire formation. These results pave a way for understanding and eventually controlling the growth of rare earth disilicide nanowires on the Si(001) substrate. Simulated scanning tunneling microscopy (STM) and charge density difference images agree with experimental STM and Kelvin Probe microscopy contact potential difference data. Dipoles induced by RE adatoms are predicted to decrease the substrate work function, as confirmed by KPFM. We have also explored calculations for the adsorption of noble metal atoms on YSi2 nanowires. Significant charge transfer from Y to Pt drastically lowers the Pt-d band and hence new chemical and optical behaviors are expected from the Pt atom.

Controlling the Self-Assembly of Silica-Capped Silver Nanoparticles through Hydrophobicity: *Yong-Jae Choi*[1]; Tzy-Jiun Luo[1]; [1]North Carolina State University

Recent progress on spontaneous metallization process associated with aminosilane was utilized to synthesize silver nanoparticles with a narrow size distribution, which were later confirmed by TEM, XRD, and UV-Vis spectroscopy. The as-synthesized nanoparticles were capped in silica structures through amine groups, and their surface charges can be modified through mole ratios of silane and silver ions. Self-assembly of silica modified silver nanoparticles was observed on hydrophobic surfaces such as polypropylene and polydimethylsiloxane. The deposited nanoparticle layer, once initiated, was found to induce accelerated growth of nanoparticles at the surface, resulting in a thicker film with a reflective metallic colors. In contrast, surface treated O2 plasma was found to significantly reduce the surface deposition of nanoparticles. This surface controlled self-assembly of silver nanoparticles was later utilized to produce patterns of silver nanoparticles layer. SEM, AFM and cyclic voltammetry were also utilized to characterize the structures and properties of the film.

Development of a Simulation Method for the Formation of Nano-Porous Anodic Aluminum Oxide: *Eun Cheol Do*[1]; Byeong-Joo Lee[1]; [1]POSTECH

Since the nano-porous anodic aluminum oxide is used as a template for fabrication of nanostructured materials, it is important to be able to control the structural factors of AAO such as pore diameter, interpore distance and pore alignment. As a means to provide a guide to control the structural property of AAO, we developed a simulation scheme for the formation and growth of nano-pores during the anodizing. The dissolution of oxide layer on oxide/electrolyte interfaces which is the rate-determining step in pore growth was mainly focused, assuming that the strength and distribution of electric field which have effects on the pore growth rate depend on the curvature and thickness of oxide layer on the pore bottom. By calculating the direction and rate of dissolution as a function of the electric field distribution, pH and temperature, the growth and rearrangement of pores could be well reproduced in good agreement with experiments.

Direct Synthesis of Straight SiO2 Nanorods: *Guang Zhu*[1]; [1]Beijing Information Technology Institute

The straight SiO2 nanorods with a diameter of about 200nm and smooth surface have been directly synthesized by high temperature vapor deposition method at 1300°. The as-synthesized samples were characterized by means of scanning electron microscopy, energy dispersive x-ray, and transmission electron microscopy. The results show that as-synthesized silica nanorods have a uniform size, well-defined shape, and smooth surface. However, the morphologies and microstructures of silica nanorodsare affected by synthesis conditions, such as the concentration of the SiOx and the the deposition temperature. On the basis of these experimental results, a possible growth mechanism of silica nanorodsin this process is proposed.

Effect of Ingot Microstructure on Magnetic Properties of Nd2Fe14B/α-Fe Nanocomposite Magnets: *Junhua You*[1]; [1]Northeastern University

In this text, Metallographic microscope, Scanning electron microscopy(SEM) and X-ray diffraction(XRD) have been used to analyze the microstructures and phases of the cast ingots. The effect of cast ingot microstructure on magnetic properties of the bonded magnets is also investigated. The results indicated that: Both of the ingot I(1kg)and the ingot II(60g) are composed of Nd2Fe14B matrix phase, α-Fe particle phase and Nd-rich phase, the microstructure of the ingot II is much finer because of its faster cooling velocity; The magnetic properties of the bonded magnets made from the ingot II are higher than that made from the ingot I, meanwhile the magnetic properties difference of the magnets made from different parts of the ingot is about 6%, it indicates that the magnetic properties of bonded magnets are not sensitive to the ingot microstructure.

Enhanced Photoelectrochemical Degradation of Methyl Orange Using Anodized Ti Rods: *Archana Kar*[1]; Vaidyanathan Subramanian[1]; [1]University of Nevada, Reno

Titanium dioxide (TiO2) is widely used for heterogeneous photocatalytic waste treatment. Several studies have reported the application of TiO2 slurry as well as immobilized TiO2 but they are limited by difficulties in post-treatment recovery and reduction in active surface area. One approach to minimize this problem is

to utilize TiO2 nanotubes formed by anodization of Ti foil backbone. To increase the photoefficiency of the TiO2 nanotubes with respect to geometrical surface area we utilized Ti rods of diameter 0.5 mm for anodization. TiO2 nanotubes were prepared by anodizing Ti rods in an Ethylene glycol and Ammonium fluoride electrolyte. The length and diameter of the nanotubes were found to be 700 - 800 nm and 100 - 170 nm respectively. Photodegradation experiments confirmed that anodized Ti rod shows 43% Methyl orange degradation whereas anodized Ti foil shows 20% MO degradation under the same conditions.

Modeling the Electrochemical Interactions of Nano-Particulate Systems in Medical Devices: *Jonathan Guyer*[1]; David Saylor[2]; James Warren[1]; [1]National Institute of Standards and Technology; [2]Food and Drug Administration

Nano-particulate silver systems are widely used in wound dressings, surgical masks, and catheter coatings as anti-microbial agents. We previously developed a phase field model of the electrochemical interface and demonstrated that a simple set of assumptions gives rise to a rich set of behaviors, including electrocapillary phenomena, differential capacitance curves that resemble experimental measurements, and non-linear kinetics consistent with the empirical Butler-Volmer relation. Despite these successes, numerical constraints limited the applicability of the model to dimensions of a few nanometers. Fortunately, however, the model is capable of making predictions at precisely the spatial and temporal scale that we are interested in for studying medical applications of silver nano-particles. We will discuss the impact of particle size, solution concentration, and particle aggregation on ion release and surface charge, which not only impact the anti-microbial efficacy and system stability, butmay also affect biocompatibility.

Multilayer Optical Filters Withstand Extreme Strain: Thad Druffel[1]; Matt Lattis[1]; Omar Buazza[1]; *Scott Farmer*[1]; [1]Optical Dynamics

Nanocomposites composed of UV cured polymers and metal oxide nanoparticles offer highly engineered mechanical and optical properties for transparent, flexible systems. Because nanoparticles can offer high hardness and a wide range of refractive indices, their inclusion in a polymer matrix can dramatically increase wear resistance and significantly alter refractive index, while the polymer binders are allowed to control overall mechanical flexibility. We have successfully built sharp cut optical filters composed of more than 30 discrete layers that easily withstand large strains induced by mechanical loading and thermal cycling. We demonstrate a UV cured, spin applied thin-film system that can undergo strains in excess of 20 percent without failure. This novel coating system allows sophisticated thin-film filters to be used in applications and environments that were previously impractical.

Palladium Doped TiO2 Thin Films with Antibacterial Properties: *Mehdi Rezaiyan Deloei*[1]; Mohammad Ghorbani[1]; Mohammad Mohsenzadeh[2]; [1]Sharif University of Technology; [2]Ferdowsi University of Mashhad

Thin film of TiO2 and it's palladium doped sample were prepared from a titanium isopropoxide precursor by particulate sol-gel processing on 316 stainless steel substrate. FTIR analysis was used to identify the chemical changes which occurred in the solution. It shows absorption peak at about 576 Cm-1 correspond to Ti-O-Ti bands. The morphology of the coatings was characterized by scanning electron microscopy (SEM). X-Ray diffraction pattern showed that Palladium presence increases the transformation temperature of anatase to rutile. It was found that Pd addition contributes to an increase in the activity of thin film by the aid of UV-Vis spectroscopy and antibacterial tests against E.coli.

Phase Formation and Mechanical Properties of Cu-Zr Based Glasses and Glass Matrix Composites: *Simon Pauly*[1]; Jayanta Das[1]; Jürgen Eckert[1]; [1]IFW Dresden

The crystallization behaviour of $Cu_{50}Zr_{50-x}Ti_x$ (0 = x = 10) metallic glasses is investigated. Higher Ti contents promote the formation of metastable phases and the crystallization proceeds in multiple steps. However the phase evolution upon quenching the melt is different also indicating a distinct dependence on the Ti content. Therefore, different TTT or CCT diagrams have to be considered. Kinetic parameters like fragility, activation energy of crystallization are compared with data of Cu-Zr-Al alloys.Cu-Zr based alloys can undergo an austenite-to-martensite transformation (MT). This transformation is believed to enhance the ductility of partially crystalline Cu-Zr based bulk metallic glasses. These composites show high yield strength (up to 1753 MPa) and large plastic strain (over 15%). The high strength scales with the volume fraction of glassy matrix. The MT was investigated in a high-energy x-ray beam with respect to compositional influences and the stress levels at which the transformation occurs.

Preparation and Characterization of Nano-Sized Polyhedron Co3O4 Powder by Spray-Oxidation: *Xueyi Guo*[1]; Qiusong Guo[1]; Qingming Feng[1]; Qinghua Tian[1]; [1]School of Metallurgy

Nano-sized Co3O4 powder with polyhedron morphology were prepared by using single step spary-oxidation. The precursor solution was prepared by using cobalt chloride (CoCl2•6H2O) as raw material. Precursor solution was sprayed by using inner mixed air-nozzle and oxidized in a pipe resistance furnace with compressed oxygen as the carrier gas. The products were characterized by scanning electron microscope(SEM), x-ray diffraction(XRD), infrared spectrum(IR) and brunauer-emmett-teller(BET) surface area method. SEM results show that the reaction temperature strongly influencs the morphology of the particles. Nano-sized powders polyhedron-Co3O4 can be successfully prepared at temperature of about 800º.XRD studies and infrared spectrum(IR) revealed that the product is pure Co3O4 with normal-spinel structure. The specific BET surface area was found to be 5.3m2/g, indicating that the particles have a high activity and a good prospect of application.

Preparation of Antimicrobial Colored Sheets with Modified Silver-Doped Titanium Dioxide Nanocrystals: Guoliang Li[1]; *Bing Peng*[1]; [1]School of Metallurgical Science and Engineering, Central South University

Silver-doped titanium dioxide nanocrystals were treated with several dispersant agents by wet activated technology, and sodium stearate showed the superior property to boost the hydrophobicity of silver-doped TiO2 nanocrystals. Antimicrobial colored sheets were prepared with modified silver-doped TiO2 nanocrystals which were treated with sodium stearate by dry activated technology. The properties of treated colored sheets were significantly affected by concentration of silver-doped TiO2 nanocrystals in polyester coatings and the treatment conditions. The treated colored sheets showed fine photocatalytic activity to enhance the decolorization and degradation of methyl orange under UV light irradiation, good antimicrobial activity against Escherichia coli, and strong time effectiveness of photocatalytic and antimicrobial properties after water flushing. The optimal property was obtained when the colored sheets were coated with paint including 3% modified silver-doped TiO2 nanocrystals which were treated with 3.5% sodium stearate.

Preparation of Nanorods and Diamond like Carbon by High Temperature Dissociation of Silicon Carbide in a Plasma Heated Special Reactor: *Bijan Nayak*[1]; B. K. Mishra[1]; [1]Institute of Minerals and Materials Technology

Silicon carbide nanorods have excellent scope in several future applications such as field emission TV, flexible thin film computers, atomic force microscope tips, quantum devices, micro and nano composites to name a few. Like wise, diamond like carbon (DLC) finds wide use in wear resistant surface coatings and electronic component packaging. The present work reports an ingenious arc plasma method to dissociate silicon carbide at temperatures above 2200°C by taking advantage of its typical non-melting property. A special plasma reactor has been designed and developed to prepare silicon carbide nanorods and DLC by controlling the retention and exit of Si vapour in the reactor. In the first case, when the dissociated Si in the form of vapour is allowed to recombine with C inside the reactor, SiC nanorod formation is observed. Assembly of rods are found to pin to SiC grains at different points. In the second case, when the Si vapour is let out of the reactor zone, the left out C on the dissociated SiC surface, grows epitaxially on the underlying SiC surface to produce DLC layer. The SiC nanorods and DLC layer were characterized by micro Raman spectra, optical microscopy, TEM and AFM for structural evaluation. The paper envisages to discuss further details about the work and results at the time of presentation.

Preparation of Nanosized Zinc Ferrite Particles in the System of Fe(III)-Zn(II)-NH3-CO32—H2O: *Qinghua Tian*[1]; Xueyi Guo[1]; Jun Li[1]; Dong Li[1]; [1]Central South University

Based on the review of technical literatures, the co-precipitation-drying-thermal decomposition was determined for the preparation of nanosized zinc-ferrite. The ammonium bicarbonate was chosen as the co-precipitation agent, and the thermodynamic analyses were done for the solution system of Fe(III)-Zn(II)-NH3-CO32—H2O. The double-jet precipitation process was proposed based on the thermodynamic analyses results. Considering the heavy aggregation among nano-sized particles, the measures were adopteed by addition of dispersant in the process of co-precipitation and washing by organic solvent or azeotropic distillation. By TG-DTA analysis, the suitable thermol-decomposition temperature of the zinc ferrite precursor was determined at about 450°.Kept at this temperature for 2 hours, the pure and well crystalized ZnFe2O4

was obtained. SEM Photos of the obtained powder shows that the particles are uniform in size distribution (20nm-50nm) with good dispersivity.

Surface Modification of Silver-Doped Nanometer Titania with Stearic Acid: Yunchao Liu[1]; Bing Peng[1]; *Liyuan Chai*[1]; Liqiang Liu[1]; [1]School of Metallurgical Science and Engineering, Central South University

Organic surface modification of silver-doped TiO2 with stearic acid was investigated by varying reagent amount, reagent concentration, time, temperature and pH value. The lipophilic degree was analyzed using a 37 factorial design to obtain the optimal condition. The prepared samples were characterized by FT-IR and SEM, and the antibacterial property was determined. The results showed that the lipophilic degree reached up to 64.3% under optimum condition of stearic acid amount 1%, stearic acid concentration 0.0002mol·L-1, modification time 90 min, temperature 80° and pH 6. The stearic acid was bonded on the surface of silver -doped TiO2 by chemical bond. The dispersancy of silver-doped TiO2 was improved after modification. Although antibacterial rate decreased slightly, it still achieved 99.77%.

Synthesis and Characterization of SiO2 and SiC Micro/Nanostructures: *Guang Zhu*[1]; [1]Beijing Information Technology Institute

Silica-based nanowires, straight nanorods, straight Y-shaped silica nanorods, flower-like microstructures, and SiC/SiO2 core-shell coaxial nanocables have been generated through a simple thermal evaporation method. The synthesized samples were characterized by means of scanning electron microscopy, transmission electron microscopy, high resolution transmission electron microscopy, energy dispersive X-ray spectroscopy, and Raman spectrum. Generated silica nanowires with a diameter of about 100nm and length of up to several tens of micrometers, straight silica nanorods and Y-shaped nanorods with a diameter about 50-200nm, and novel flower-like silica microstructures all are amorphous and consist only of silicon oxide, and have a neat smooth surface. Generated SiC/SiO2 core-shell coaxial nanocables have a crystalline core and a surrounding amorphous layer. The results show that the present method should be possible to synthesis various micro/nanostructures under appropriate experimental conditions. These nanostructures may find applications as building blocks in nanomechanical or nanoelectronic devices.

Synthesis and Processing of Cu-CNT Nano-Composites: Martín Mendoza[1]; *Guillermo Solórzano Naranjo*[1]; Eduardo Brocchi[1]; [1]PUC-Rio

This work presents some structural characteristics of a Copper–2%CNT nanocomposite synthesized by chemical method. Single wall Carbon nanotubes(SWCNTs), with diameters between 5-10 nm were used. The nano-composites powders were produced by dissociation of a homogeneous suspension containing Cu(NO3)2.3H2O, SWCNT an anionic tensoactive; followed by hydrogen reduction of the obtained CuO-SWCNT product. Thermodynamic studies provided support to the experimental procedure. X ray diffraction and Transmission Electron Microscopy have been used as characterization tools. The former confirmed the presence of metallic copper with carbon. The later allowed the observation of a good dispersion and adherence between Cu particles onto CNT. The obtained Cu powder particles were observed to be in the 150-300nm range. Bulk nano-composite pellets were produced by issostatic pressure under 150MPa. Sintering studies show a heterogeneous grain growing of copper matrix reaching a polycrystalline product of 150nm- 3µm grain size. Mechanical and transport properties measurements are currently in progress.

Synthesis of Single-Crystalline Silicon Nitride Nanowires with Controlled Diameters by Nitriding Cryomilled Nanocrystalline Silicon Powder: Fei Chen[1]; Ying Li[2]; Wei Liu[2]; Qiang Shen[1]; Lianmeng Zhang[1]; Qing Jiang[3]; Enrique Lavernia[2]; *Julie Schoenung*[2]; [1]Wuhan University of Technology; [2]University of California, Davis; [3]Jilin University

In the present work, silicon nitride nanowires (SNNWs) have been synthesized via nitriding cryomilled nanocrystalline silicon powder. The silicon powder exhibits a fine polycrystalline structure after the cryomilling process, with an average grain size of 25 to 125 nm at various cryomilling times. The SNNWs that form after the nitridation of the cryomilled silicon powder exhibit single crystal structure and are 20 to 100 nm in diameter and ~ 10 µm in length. The diameter of the nanowires is in agreement with the grain size of the cryomilled Si powder. Microstructural characterization reveals that the as-synthesized nanowires have a hexagonal structure and their primary growth direction is along the [0001] direction. The formation of the Si-N-Si bond during the cryomilling process, as investigated theoretically with density functional theory, promotes the subsequent synthesis of the a-Si3N4 nanowires. The mechanism for nanowire formation appears to be a vapor-solid (VS) reaction.

Synthesis of Straight Si3N4 Nanowires: *Guang Zhu*[1]; [1]Beijing Information Technology Institute

The novel straight Si3N4 nanowires have been directly synthesized by thermal evaporation of the mixture powders of silica and carbon nanofibers at 1300°C without assistance of any metal catalyst. The as-obtained Si3N4 nanowires are generally 30-50 nm in diameter and several tens of micrometers in length, and have a smooth surface. The characteristics of the products are analyzed by various methods, results of which indicating that temperature and ambience are two key factors for the formation of Si3N4 nanowires, and the possible growth mechanisms is also discussed.

Aluminum Reduction Technology: Light Metals Division Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Gilles Dufour, Alcoa Canada, Primary Metals; Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Alcan; Jayson Tessier, Alcoa Deschambault

Sunday, 6:00-8:00 PM Room: 2001
February 15, 2009 Location: Moscone West Convention Center

Detecting Abnormalities in Aluminium Reduction Cells Based on Process Events Using Multi-Way Principal Component Analysis (MPCA): *Brent R. Young*[1]; John Chen[1]; Nazatul Aini Abd Majid[1]; Mark Taylor[1]; [1]University of Auckland

In the aluminium industry optimal production and quality products are major process targets. One way to achieve these targets is by improving the process control of aluminium reduction cells, and this is the aim of this research. This research proposes to apply an advanced multivariate control chart to aluminium reduction cells in a manner which provides new insights into process abnormalities and their diagnosis. The proposed approach uses multi-way principal component analysis to observe the movement of data towards abnormality after process events. Preliminary results showed that using the proposed approach could detect anode spikes after anode changing or tapping. Data with anode spikes present moved in a different direction than the data with anode spikes absent. An anode spike trajectory could be set up based on this discrimination. Data which move towards the anode spike trajectory have a high possibility of having anode spikes. Therefore based on this trajectory, the cell could be monitored ahead of time for spikes, and operations may take action to search for them much earlier. This will lead to a real-time fault detection system and is expected to assist process engineers in improving the process control of aluminium reduction cells.

Biological Materials Science: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Ryan Roeder, University of Notre Dame; John Nychka, University of Alberta; Paul Calvert, University of Massachusetts Dartmouth; Marc Meyers, University of California

Sunday, 6:00-8:00 PM Room: 3014
February 15, 2009 Location: Moscone West Convention Center

Application of Small Angle Neutron Scattering to Quantitatively Analysis of Bony Canliculus of Human Compact Bone: *Yong Choi*[1]; Eun J. Shin[2]; Baik S. Seong[2]; Doo J. Paik[3]; [1]Sunmoon University; [2]KAERI; [3]Hanyang University

Small angle neutron scattering (SANS) was applied to quantitatively analyze of human compact bone, especially fine bony canaliculus to get important information about growth and degradation of the human bone. Two types of jaw-bone with different physiological histories such as normal and osteoporosis bones were selected. Bone density was determined by bone densitometer.

Microstructure of the compact bone was observed bytransmission electron microscopy. The specimen was cut to fit and tested by SANS of HANARO in KAERI. Directional distribution of bony lacuna of the compact bone and nano-sized canaliculus interconnected in all direction were observed by SANS. The amount of canaliculus of lacuna was larger in normal bone than in osteoporosis bone. Microstructure observation by transmission electron microscopy and measurement of bone density also support the fact that SANS is one of the useful techniques to study in-situ quantitative evaluation of the very fine bony canaliculus of compact bone.

Development of a Vascular Occluder for Use in Liver Resection Surgery: *Michael Ashbrook*[1]; Prince Anyalebechi[1]; Timothy Fitzgerald[2]; John Hall[3]; Ken Jonkman[3]; [1]Grand Valley State University; [2]Saint Mary's Hospital; [3]Avalon Laboratories

Liver resection surgery is the procedure by which a cancerous region of a liver is removed. Unfortunately the surgery has the potential to cause high levels of blood loss. This is because nearly all of the blood returning from the lower extremities to the heart flows either through the liver or through the major vein that the liver connects to such as the inferior vena cava. Current techniques cannot seal the larger blood vessels in the liver and so do not completely eliminate bleeding. A device known as hepatic vein occluder has been designed to completely isolate the liver from blood flow during liver resection. It seals off the point where the liver joins into the inferior vena cava, while still allowing blood to flow through and into the heart. In this paper, the design, construction, and results of preliminary testing of the device are discussed.

Electrochemical Study of Titanium Behaviour and Semiconducting Properties of Anodic Oxide Films Formed on Titanium in PBS Solutions with Different pH: *Piotr Handzlik*[1]; Krzysztof Fitzner[1]; [1]AGH University of Science and Technology

The first aim of this study was to investigate the electrochemical corrosion behavior of titanium and the corrosion rate in the PBS solutions with pH=8.9, and pH=2.9. Potentiodynamic curves and Tafel plots were used to estimate E_{cor} and i_{cor} at t=21°C. Electrochemical Impedance Spectroscopy (EIS) confirmed high corrosion resistance of Ti under imposed conditions. To obtain information about electronic properties of passive oxide films formed at various potentials Mott-Schottky plots were constructed and the flat band potential and number of donor densities were derived. However, the change of temperature to 36.6°C showed that the corrosion current increased significantly in both solutions. EIS experiments indicated that equivalent circuit must be changed which speaks for the change of the properties of the protective layer at the titanium surface. Calculated donor densities confirmed also this observation: donor densities increased. Anodic oxide films were studied by XPS technique to identify composition of the layer.

Femtosecond Laser Micromachining of Bone Mechanical Test Specimens: *Katrina Altman*[1]; Katharine Flores[1]; Dave Farson[1]; Elise Morgan[2]; [1]The Ohio State University; [2]Boston University

The mechanical properties of bone are highly statistical due to its anisotropic and hierarchical microstructure. For other engineering materials, testing at the microscale has been shown to provide data for individual microstructural components in an effort to understand macroscopic mechanical behavior. The application of such microscale testing to bone will permit modeling of the aggregate material to predict effects of age, disease, or injury on the mechanical properties. The femtosecond laser is presently used to produce microscale specimens in bovine cortical bone, which will be used for mechanical testing. The femtosecond laser is advantageous for micromachining of biological materials because it may be used in ambient, non-vacuum environments, making it a flexible tool for machining the bone surface while preserving its microstructure. The short pulse duration minimizes thermal diffusion and damage to the surrounding material. Microcompression pillars with diameters ~10µm have been produced. Processing and experimental results will be discussed.

Fluoridated Hydroxyapatite Bioactive Coatings on Ti-Alloy Substrate Deposited by RF Magnetron Sputtering: Dongyang Lin[1]; Xiaoxiang Wang[1]; Xiaoyan Liu[2]; [1]Zhejiang University; [2]Jiangsu University

A pure and dense hydroxyapatite [Ca10(PO4)6(OH)2, HA] coating and a fluoridated HA [Ca10(PO4)6(OH)2-xFx, FHA] coating are deposited on Ti6Al4V substrates by Radio frequency magnetron sputtering. Researches have been carried out in the phase composition, microcosmic appearance and growth pattern of sputtering coatings, based on XRD, SEM, FTIR and AFM. The result indicates that magnetron sputtering coating appears in amorphous state, which could be transformed into crystalline state after annealing treatment; the microscopic surface of the sputtered coating is uneven, forming network structure. The growth pattern of the coating is lamellar accompany with island way. There isn't any pore or crackle on the coating/substrate interface and the interfacial binding strength is higher than 50Mpa. Fluorine-incorporation in HA does not generate significant influence on interfacial binding strength. However, FHA bioactive coating implant has better mechanics stabilities than HA coating implant in Simulated Body Fluid (SBF) experiment, which is significant in extending the period of validity of the coating.

Micro-Arc Oxidization of a Commercial Purity Titanium for Biomedical Applications: Cemil Isiksacan[1]; Mert Gunyuz[1]; Hakan Bermek[1]; Pinar Huner[1]; Murat Baydogan[1]; Eyup Kayali[1]; *Huseyin Cimenoglu*[1]; [1]Istanbul Technical University

Commercial pure titanium is an attractive material for dental implant production. Conventional titanium implants do not chemically connect to bone or actively induce bone growth compared with calcium phosphate coated implants. In this respect, micro-arc oxidation is an effective surface modification technique to enhance their bioactivity by forming functional, adherent and porous titanium oxide surface layer. In this study, the surface properties including the morphology, roughness and wettability a commercially pure titanium was investigated after. Micro-arc oxidation process was performed in "(CH3COO)2Ca.H2O + Na3PO4" electrolytic solution with addition of silver inorder to achieve antibacterial surface layer. Micro-arc oxidation formed a porous titanium oxide layer on the surface with small precipitates containg phosporous, calcium and silver. Biocompatibility of the titanium was then determined by simulated body fluid and cell culture tests as well as antibacterial tests.

Temperature Effect on the Structure and Mechanical Properties of Nacre: Zaiwang Huang[1]; *Xiaodong Li*[1]; [1]University of South Carolina

Structural and mechanical characterization has been performed on nacre heat treated at various temperatures. We show that, after treated for 10 minutes at 500° and 1000° in air respectively, two phase transformations with aragonite to calcite and aragonite to calcium oxide (CaO) occur. Scanning electron microscopy and atomic force microscopy images clearly demonstrate the microstructure evolvement process: the survival of sandwiched structure and occurrence of holes instead of nanoasperities on aragonite platelet surface treated from 500°, eventually, the formation of micro-scale CaO particles at 1000°. The nanoindentaion testing results exhibit that nacre at high temperature has a sharp loss in elastic modulus and hardness comparing with those at room temperature. Nanoscale structural and mechanical characterization for nacre heat treated at different temperatures therefore may provide great benefits in bioinspired materials and open new avenues for exploring the origin of its unique mechanical properties.

The Effect of Dialysis Environment on the Structural Properties of the Membranes Used for High Flux Dialysis: *Mehmet Aksoy*[1]; Metin Usta[2]; A. Hikmet Ucisik[3]; [1]Istanbul City Health Management; [2]Gebze Yuksek Teknoloji Enstitusu; [3]Bogazici University

High flux dialysers containing new generation of dialyser materials have been widely used for patients with chronic renal failure within the last decade. Dialyser membranes are more prone to damage to to the harsh environment during high flux dialysis. Reuse of dialysers has advantages like better biocompatibility and lower cost, any damage of the dialysers membrane during reuse of the dialysers can also cause very serious clinical complications. Therefore reuse of the dialysers is an issue that has to be approached more cautiously. In this study polysulphone and polyamide dialyser membranes were being investigated in terms of mechanical properties and changes in crystallinity before and after dialysis sessions. Dialysis sessions were performed on five patients with dialysis age less than two years and without any other accompanying disease at the Hemodialysis Department of Istanbul HaydarpasaNumune State Hospital.

The Evaluation of Hysteresis Loop of Nickel-Titanium Orthodontic Wires: S. Mohamad S. Aghamiri[1]; *Mahmoud Nili Ahmadabadi*[1]; [1]University of Tehran

NiTi wires have been used in orthodontic application because of their unique characteristics. The mechanical properties of wires should result in achieving continuous optimal forces and rapid tooth movement. Magnitude of hysteresis loop attributes to energy dissipative processes and the stress hysteresis specifies the limit of irreversible phenomena that happen during stress induced martensite (SIM) transformation. This investigation was carried out to examine stress

hysteresis behavior of nickel-titanium archwires in different temperatures. Two different brands of NiTi archwires including TrueFlex (superelastic wire) and 3M Unitek (shape memory wire) were studied by three-point bending test in 4 mm deflection. Each test was done in three clinically relevant temperatures of 22, 37 and 50°C. Differential scanning calorimetry (DSC) analysis was performed to determine the phases and transformation temperatures in wires. The results show that there are considerable and meaningful differences between the hysteresis of wires in each situation.

Characterization of Minerals, Metals and Materials: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee, TMS/ASM: Composite Materials Committee

Program Organizers: Toru Okabe, University of Tokyo; Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF

Sunday, 6:00-8:00 PM Room: 3009
February 15, 2009 Location: Moscone West Convention Center

A New Method of Cutting Blast for Vertical Shaft Excavation and Its Experimental Study: *Zhang Yiping*[1]; [1]Guizhou University

Based on cutting principle and technology development of vertical blasthole cut by stage and deck in vertical shaft excavation, combined with the merits of middle space charging and toe space charging, the reinforced cutting effect of central large-diameter blasthole and the method of cutting blast by stage and deck toe space charging for the vertical large-diameter blastholes is put forward and analyzed theoretically. This new cutting blast method is provided with the advantages of high blasthole using ratio, big cavity bulk, low rate of chunk, even lumpiness and relatively high energy using ratio. The parameters choice and practical effects of this cutting method were discussed after in-situ experiment. It shows that the decked delay time of 75ms-100ms is applicable.

Analysis of the Contacting State of Specimen with Supports in Dynamic Fracture Tests by Modified Hopkinson Pressure Bar: Chunhuan Guo[1]; *Ruitang Liu*[1]; Yang Yang[1]; Yongdong Wang[1]; [1]Harbin Engineering University

Presently, problems existing in dynamic fracture experiments which are tested in Hopkinson bar are that the contacting state of specimen with supports is not very clear. In this paper, Experiments with standard Charpy specimens (10mm× 20mm× 100mm) of a structural steel are tested using modified Hopkinson pressure bar and strain gage techniques. The initial results indicate that the bouncing of specimen from supports in impact loading instant is observed, i.e. the contact of specimen with supports is lost and that the pre-crack in specimen is initiated under the condition of one-point-bending. The effect of span(S) on the bouncing behaviors of the specimen is important, and the critical span size in the test is obtained, that is, when $S \leq 67.5$mm, the specimen always keeps in contact with supports during loading process. In addition, the studies about the size effect of specimens in this test are ongoing.

Carbon Compound as Anode Material Electrode in Super Lithium Ion Capacitor: Li Jie[1]; *Yang Juan*[1]; Hao Xin[1]; Zhang Zhian[1]; Lai Yanqing[1]; Zhou Xiangyang[1]; [1]Central South University

Series of carbon compounds as anode materials were prepared for super lithium ion capacitor using graphite and active carbon (AC) as raw materials. Their electrochemical properties were investigated by constant current charge-discharge test. The results showed that the compound anode had good capacitive performance as well as Li-ion battery performance. The potential of the capacitor could be as high as 3.5V vs Li/Li^+ when compared with 2.5V vs Li/Li^+ in the AC-AC capacitor, accordingly, the energy density increased from 21.7Wh/kg to 40.3Wh/kg. The compound anode also had excellent rate performance that as the current density increasing from 0.1A/g to 1A/g,the capacitance decreased only 1.3F/g, and good cycle performance that the capacitance holding remained 96.7% after 10 times cycles even at the highest potential of 3.5 V vs Li/Li^+.

Characterization of the SnO2:F/CdS:In Structures: *Shadia Ikhmayies*[1]; Riyad Ahmad-Bitar[2]; [1]Applied Science Private University; [2]University of Jordan

SnO2:F/CdS:In bilayers were produced by the spray pyrolysis technique on glass substrates. The structures were characterized by recording and investigating their transmittance curves, I-V plots, x-ray difractograms (XRD) and by observing their scanning electron microscope (SEM) images. From the I-V plots it was found that the SnO2: F forms an ohmic or quasi-ohmic contact with CdS:In. XRD patterns show the polycrystalline nature of the films and show that there is a small shift in the position of the (200) line of SnO2:F without the appearance of new peaks. The morphology of the structures are compared with those of SnO2:F alone and CdS:In alone on glass substrates.

Coating $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ with ZnO Nano-Particles by Mechanical Solid-State-Chemistry-Reaction: *Ping Yang*[1]; Jing Zhan[1]; Chuan-fu Zhang[1]; Xi Dai[1]; You-qi Fan[1]; [1]Central South University

To improve the electrochemical performances, $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ cathode materials have been coated with ZnO nano-particles by mechanical solid-state-chemistry-reaction. The structures and morphologies of the synthesized materials were investigated by XRD, SEM and TEM and the electrochemical performances of materials were studied within a voltage window of 2.75-4.3 V at current density of 170mAh/g. The results show that the surface of $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$ particles is coated with very fine ZnO composite but its structure is not affected by coated with 3% ZnO. The presence of a thin ZnO layer could suppress the reaction between the cathode and electrolyte, and remarkably decreases the charge transfer resistance, which is attributed to the improvement in the cyclic performance comparing the bare $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$. It is proposed that surface treatment by mechanical solid-state-chemistry-reaction is a simple and effective method to improve the electrochemical performance of $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$.

Defects in Deformed Zircaloy 2: An Effort to Couple Observations from Different Analytical Techniques: *S. Sahoo*[1]; V. Hiwarkar[1]; I. Samajdar[1]; P. Pant[1]; P. Pujari[2]; G. Dey[2]; D. Srivastav[2]; R. Tiwari[2]; S. Banerjee[2]; [1]IIT Bombay; [2]BARC, Mumbai

Defect evolution during uniaxial cold compression and cold/warm rolling of fully recrystallized zircaloy 2 was extensively studied. An attempt has been made to correlate the observations obtained from various techniques; positron annihilation spectroscopy (PAS), x-ray diffraction (XRD), transmission electron microscopy (TEM) and electron backscattered diffraction (EBSD). Deformation twinning in compression samples had shown strong influence on positron lifetimes. Samples, where twinning was maximum had lowest positron lifetime and where twin decay was maximum had highest positron lifetime. In case of cold/warm rolled samples, the positron lifetimes decreased with increase in deformation temperature. This drop was relatively higher up to a deformation temperature of 200°C and thereafter it was lower to 400°C and 600°C. XRD estimated dislocation density and lattice strain increased with progressive compression. However for rolled samples, the results of dislocation density showed exactly similar trend as lifetime measurements and lattice strain gradually decreased with increase in deformation temperature.

Electrical, Optical and Structural Properties of Vacuum Evaporated CdTe Thin Films: *Shadia Ikhmayies*[1]; Riyad Ahmad-Bitar[2]; [1]Applied Science Private University; [2] University of Jordan

Polycrystalline CdTe thin films were prepared by vacuum evaporation on glass substrates. The I-V plots which were linear were used to find the resistivity. A value of 2.10×106 O.cm was obtained. The transmittance was measured in the wavelength range 400-1100 nm. The bandgap energy was found to be 1.48 eV. X-ray diffraction pattern shows that the material deposits in the zinc blend structure with one strong reflection from the C(111) plane. The scanning electron microscope image shows a uniform surface with a small density of large ( ) grains scattered on the surface.

Influence of Particles Shape Characteristics of Galena on their Floatability Ground by Ball and Rod Mills: *Mohammad Reza Aslani*[1]; Bahram Rezai[2]; Esmail Jorjani[1]; [1]Islamic Azad University, Tehran Science and Research Campus, Technical and Engineering Department; [2]Amirkabir University of Technology, Mining, Metallurgical and Petroleum Engineering Department

Crushing and grinding are factors that cause creation some changes in physical and chemical characteristics of processing materials such as distributions of sizes and shapes. In this research, experimental studies to determine the shape properties and floatability of galena were performed on the products of ball and rod mills. Shape properties have been stated in terms of common shape descriptors such as elongation, flatness, roundness, and relative width by measuring on the projections of particles using scanning electron microscope. The floatability characteristics of galena were determined by flotation technique using the laboratory flotation cell. Finally, some correlations were found between the shape properties and recovery rate of particles. The results have shown that

the recovery rate decreases with increasing roundness and relative width, that these particles are slow floatable, i.e., elongated and flatted particles having higher recovery rate indicated more hydrophobicity and are fast floatable.

Liquid Metal Embrittlement of AISI 4340 Low Alloy Steel by Ga-In Eutectics: *Refael Levy*[1]; Eugen Rabkin[2]; David Gorni[1]; Shimshon Bar - Ziv[1]; [1]RAFAEL Ltd.; [2]Department of Materials Engineering, Technion – Israel Institute of Technology

We studied the Liquid Metal Embrittlement (LME) of 4340 martensitic steel by liquid In-Ga eutectics. LME reduces the steel strength only in high strength notched specimens that were deformed in tension at low strain rates. The nucleation stages of LME were investigated using notched specimens that were deformed in liquid metal environment under sustained load, close to their fracture stress. After load was removed, the near-notch region was examined using SEM and AFM. A pre-fracture penetration (~10 μm) of liquid alloy along the grain boundaries associated with changes in the martensitic microstructure was observed. A significant decrease of notch tensile strength with decreasing deformation rate was found in the case of LME. The corresponding value of the deformation activation volume was in a good agreement with predictions of Nam-Srolovitz atomistic mechanism of LME [PRL vol. 99, No. 025501 (2007)].

Mechanisms of Bioleaching of Phosphor from Phosphorous Iron Ore: Li Qian[1]; *Jiang Tao*[1]; [1]Central South University, School of Minerals Processing and Bioengineering

The investigation on bio-dephosphorization from iron ore was conducted. The results show that there are two ways for bio-dephosphorization from iron ore, one is that thiobacillus ferrooxidans directly derive phosphorus from ore, the other is acid that metabolizing by thiobacillus ferrooxidans leaching, which is the more impotent one. Adding suitable pyrite can strengthen the process through providing nourishment and direct or indirect producing acid. From the analysis of E-pH graph of Fe-H2O system, it was concluded that Fe2+ wouldn't be oxidized under sterile system in this potential and pH ranges. It obviously can conclude that the Thiobacillus ferrooxidans reduced the potential for the oxidization of Fe2+ and made the oxidization would occur in the above pH and potential zone, so the bio-leaching process can proceed. From the analysis of E-pH graph of P-H2O system, it was concluded that H3PO4 can exist steadily.

Microstructure and Creep Properties of T6 Treated Ti-6Al-4V Alloy: *Bao Xianyu*[1]; Tian Sugui[1]; [1]Shenyang University of Technology

By means of T6 heat treatment, creep properties measuring and microstructure observation, an investigation has been made into the influence of T6 treatment on the microstructure and creep properties of Ti-6Al-4V alloy. Results show that the deformation feature of the isothermal forged Ti-6Al-4V alloy during creep is that a large number <a> dislocation slipping are activated on the basal planes, and the slipping of <a+c> dislocation is activated on pyramidal planes. After T6 treatment, the initial a phase displays an equiaxed morphology, and some of the ß phase is distributed the around region of a phase, in which distributes some needle-like martensite phase, which enhances obvious the creep lifetimes of the alloy at 575 MPa and 400°C. The deformation mechanism of T6 state alloy during creep is that dislocations occur the single orientation slipping, namely, the slipping of <a+c> dislocation is activated on the pyramidal planes.

Organic Radical Battery: Nitroxide Polymers as a Cathode-Active Material: *Yan Yuan*[1]; Baizhen Chen[1]; Hui Xu[1]; Xichang Shi[1]; [1]Central South University

Stable nitroxyl polymers, such as poly(2,2,6,6-tetramenthyl-1-piperidinyloxy-4-yl methacrylate) (PTMA), are known to be effective as cathode active materials for lithium rechargeable betteries. The nitroxide radicals displayed a reversible and very rapid redox performances. We present a new synthesized method to get PTMA powder, then a doctor-blading method was used to prepare the PTMA composite electrodes,enabling successful production of homogeneous electrodes. It was characterized through FT-IR and SEM methods. The organic radical battery consists of lithium metal anode and PTMA cathode with an active material content of 65wt.%. The best performance is achieved with a thin cathode that shows nearly 100% utilization of the active material (-111mAh/g) at 0.1C. Through the electrochemical tests, it was found that there was a stable plateau at 3.65V while discharging. Besides, the cycling stability and rate capability were measured in detail. Although they showed a certain quantity of irreversible capacity on the first cycle, the capacity stabilized after the second cycle.

Preparation and Characterization of Porous Copper Powder by Thermal Decomposition of Complicated Copper Oxalate: Chuanfu Zhang[1]; *Youqi Fan*[1]; Jianhui Wu[1]; Jing Zhan[1]; Ping Yang[1]; [1]Central South University

Based on Thermogravimetric and Differential Thermal Analysis (TG-DTA), porous copper powder is prepared by thermal decomposition of complicated copper oxalate. The effects of various conditions on the morphology, crystalline grain size and specific surface area of copper powder were investigated, including temperature, time, mixing ratio and flow rate of gas, and the heating rate. Furthermore, composition and morphologies of the products were characterized by X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and BET adsorption isotherm. It is found that porous copper powder was produced under optimal conditions. The specific surface area is over 5.74 $m^2 \cdot g^{-1}$, and average pore size in particles is about 30.3 nm. After comparison of SEM images, the final product Cu well inherits the morphology of the precursor, thus a mechanism of in-situ decomposition is proposed.

Study on Deeply De-Magnesium Oxide from Phosphate Ores by Floatation: *Zhang Qin*[1]; Chen Wei[1]; Qiu Yueqin[1]; Mao Song[1]; Liu Zhihong[1]; Tang Yun[1]; [1]Guizhou University

Dolomite is main impurity mineral in phosphate ores. In practice of phosphate ores production, the grade of concentrates is only containing P2O5 31-32%. In this study, even the increasing of floatation reagent consumption and decreasing of floatation recovery, economic performance is estimated high according to the decreasing of H2SO4 consumption in subsequent production of phosphate fertilizer and the increasing of products value. In floatation stage, influence factor include the synergism impacts of floatation collector, the consumption of H2SO4 and other factors such as structure of flow sheet, stages, time, concentration of ore pulp, which were discussed respectively in this paper. The samples including raw material, concentrates and tailings were characterized by mineralogy, chemical analysis, the X-ray diffraction (XRD), scanning electron microscopy (SEM). The results show that the grade of concentration can reach to P2O5 36% by deeply de-magnesium oxide from phosphate ores, which yield a high added value.

Study on Radioactive Contamination of Fly Ash in Guizhou: *Qiu Yue Qin*[1]; Zhang Qin[1]; Cao Jianxin[1]; [1]Guizhou University

In Guizhou Province, most radionuclide and content of fly ash did not analyzed and the safety requirement did not restricted. In this study, the composition and content of the natural radionuclide of the main fly ash in Guizhou Province were determined and analyzed. The results show the scope and severity of radioactive contamination of fly ash in Guizhou. The scientific and feasible countermeasures were exhibited in this paper.

Synthesis and Characterization of Complicated Copper Oxalate Precursor Powder by Complexing Precipitation Method: Chuanfu Zhang[1]; *Youqi Fan*[1]; Jianhui Wu[1]; Jing Zhan[1]; Ping Yang[1]; [1]Central South University

Complicated copper oxalate precursor powder was prepared using ammonium oxalate as precipitating agent in the Cu(II)-$C_2O_4^{2-}$-NH_3-NH_4^+-H_2O system. The composition and morphology of the powder are characterized by chemical analysis, X-ray Diffraction(XRD), Scanning Electron Microscope (SEM), Infrared spectroscopy(IR), Thermogravimetric and Differential Thermal Analysis(TG-DTA). The effects of temperature, copper ion concentration, pH value, addition of ethanol are investigated. It's indicated by the experimental results that precursor of copper oxalate is prepared below a critical pH value, whereas the precursor turns to be a complicated copper salt while over the pH value. Conclusively, homogeneous belt aggregation powder is synthesized under the optimized conditions: 50~60°, 0.4 $mol \cdot L^{-1}$ copper ion concentration, pH value 6.4~6.8.

Synthesis and Electrochemical Characteristics of $Li(Ni_{1/3}Co_{1/3-x}M_xO_2$(M=Ti, Mg) Cathode Material by Oxalate Precursor: Chuan-fu Zhang[1]; *Ping Yang*[1]; Jing Zhan[1]; Xi Dai[1]; Yin-liang Zhang[1]; [1]Central South University

$Li(Ni_{1/3}Co_{1/3-x}Mn_{1/3})M_xO_2$(M=Ti, Mg) cathode materials were prepared from $LiOH \cdot H_2O$ and oxalate precursor. The physical and electrochemical properties were studied by XRD, SEM, cyclic volt-ampere (CV), AC impedance and constant current charge-discharge. The results show that crystal volume of Ti^{4+} or Mg^{2+} doped sample is increasing and the electrochemical reaction resistant R_{ct} is decreased at high rate, improving the cyclic performance and rate capability. The effect of Ti^{4+} doped is better than Mg^{2+}. The sample is well crystallized and simple pure phase with a-$NaFeO_2$ layered structure when doping quantity x=0.025. The second specific discharge capacity of $Li(Ni_{1/3}Co_{1/3-0.025}Mn_{1/3})Ti_{0.025}O_2$is

143.2mAh/g at 1C,128.0mAh/g at 2C in the voltage of 2.75¬4.3V,and still has 140.7mAh/g and 121.7mAh/g after 20 cycles, respectively, keeping 98.25%, 95.07% capacity.

Electrode Technology for Aluminum Production: Light Metals Division Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Barry Sadler, Net Carbon Consulting Pty Ltd; John Johnson, RUSAL Engineering and Technological Center LLC

Sunday, 6:00-8:00 PM Room: 2001
February 15, 2009 Location: Moscone West Convention Center

Boron Salt Inhibitors of Air Reactivity of Prebaked Carbon Anodes – Literature Review and Laboratory Studies: Rafael Tosta[1]; *Evelyn Inzunza*[1]; Luisa Delgado[2]; [1]CVG Aleasa; [2]Universidad Simón Bolívar

The anode carbon consumption in a reduction cell for the aluminum production depends on many factors among those that are included the raw materials, the factory processes,the design of the cell, the current efficiency and the operations of the cell. In the protection of prebaked carbon anodes, they are something well known the kindness of the boron against the oxidation for air and although the contamination could exist in the electrolytic reduction cells on the part of the boron, the one impregnated selective of carbon anodes will reduce to the minimum the presence of boron in the molten metal. The use of a modifier of the structure of the film inhibitor will reduce even more the proportions of boron to use without reducing the resistance to the oxidation. This allowed assuring the efficiency of the boron in the protection of carbon anodes against the oxidation for air.

Empiric Mathematical Models for Real Density of Calcined Coke Based on Industrial Data: *Edinaldo Silva*[1]; Deovaldo Júnior[2]; Aldo Santos[2]; Marco Giulietti[3]; Silas Derenzo[3]; [1]Petrocoque S.A. Indústria Comércio; [2]Santa Cecília University; [3]IPT - Intitute for Technological Research

The production of green coke provides additional gain to refineries due to the increase of light fractions such as LPG, gasoline, diesel oil and by reduction of sulphur and metals in these fuels. After calcination, green coke is used by aluminum, iron, re-carburizing and titanium dioxide industry. The high quality of the coke is indicated by the consistency of the real density, which is, according to the literature, function of calcining conditions of green coke. The following work has aimed at making mathematics models based on industrial data for the prediction of real density of calcined petroleum coke. For the confection of the mathematical model, it was applied the software "Table Curve 3D". The results for the proposed models indicated that the highest coefficient of correlation (r=0.30) was obtained under following process condition: a) calcining temperature and b) feeding rate of the calcining kiln.

Analysis of Sodium and Cryolite Bath Penetration in the Cathodes Used for Aluminum Electrolysis: *Jilai Xue*[1]; Wenli Ou[1]; Jun Zhu[1]; Qingsheng Liu[1]; [1]University of Science and Technology Beijing

Quantitative analysis of penetrated sodium and bath is very important in evaluating carbon cathode property and service life. In this work, sodium and bath components in the cathode samples used for aluminum electrolysis were analyzed using quantitative XRD, chemical titration, SEM-EDS techniques. The samples were sliced along their axis and each piece was analyzed to obtain a concentration profile of the penetrated sodium and bath components against the penetration depth. A sodium front was found ahead of the penetrated NaF and Na3AlF6, which formed a peak at 3.5% in the curve of Na concentration vs. penetration depth. The penetrated NaF and Na3AlF6 also showed the peaks at 2.5 % and 20%, respectively, in their concentration curves following the Na front. Various chemical reactions within the cathode materials are discussed against their possible effects on the Na and bath penetration.

Numerical Analysis of the Anode Voltage Drop of a Reduction Cell: Wangxing Li[1]; JieMing Zhou[2]; Yiwen Zhou[1]; [1]Zhengzhou Research Institute of CHALCO; [2]School of Energy and Power Engineering, Central South University

About 7~9% of the overall cell voltage of a modern Hall-Heroult cell is the anode voltage drop. It contributes a significant fraction of the cell's overall power consumption. The paper presents Finite Element Method simulation results of anode voltage drop. The purpose of the work is to determine the constituents of the anode voltage drop and to consider possible design modifications to lower the anode voltage drop. The influences of anode carbon material, carbon shape, stub shape, and contact condition on anode voltage drop were analyzed. The paper presents the ideal design of the anode.

Magnesium Technology 2009: Poster Session - Magnesium and Its Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Sunday, 6:00-8:00 PM Room: 2006
February 15, 2009 Location: Moscone West Convention Center

Session Chair: Eric Nyberg, Pacific Northwest National Laboratory

Age Hardening Response of Mg-Ce-Al Alloys: Harpreet Brar[1]; *Michele Manuel*[1]; [1]University of Florida

The aim of this paper is to characterize the age hardening response of permanent mold cast Mg-Ce-Al alloys containing various strengthening precipitates. Several compositions were designed using the PANDAT software system and a proprietary Mg alloy database to precipitate various phase fractions of Al3Ce and Al11Ce3 strengthening phases. Of particular interest is the Al3Ce phase having a DO19-type ordered crystal structure, which has been shown to have a high lattice coherency with the HCP lattice. The coherency of the DO19 precipitates promotes greater strengthening of the alloy. The alloys were aged for different times and their hardness was measured to determine solution and precipitation strengthening contributions to the peak hardness.

Crack Behavior of Magnesium Alloys Studied Using a CamScan Microscope: *Dafei Kang*[1]; Martin Crimp[1]; [1]Michigan State University

Magnesium alloys are receiving increasing attention as they are potentially good candidate materials for a range of applications, particularly in the automotive industry. Their promising properties, for instance, high specific strength and low density, make them likely substitutes for conventional heavier materials such as cast iron, steel, and at some cases even aluminum. In this paper, the crack behavior of some magnesium alloys are examined in a CamScan FE microscope eqiuipped with EBSD/OIM capacity.

Determination of Mushy Zone Mechanical Properties of a Magnesium Alloy: *Partha Saha*[1]; Srinath Viswanathan[1]; [1]University of Alabama

The mushy zone tensile behavior of magnesium alloy MRI 206 was determined using a Gleeble 1500D thermo-mechanical simulator. A protective atmosphere of CO2 + 0.5% SF6 mixed gas was used during the tests. Various specimen geometries were investigated in order to develop an isothermal zone along the specimen gauge length. It was observed that grip materials and contact area have a profound effect on the temperature profile distribution along the specimen free span. Tensile deformation tests were conducted at varying strain rates and fraction solid. The results are compared with data obtained previously for aluminum 3004 alloy.

Effect of Grain Size on Corrosion Behavior of Squeeze Cast AJ62 Magnesium Alloy in Salt Solution and Automotive Coolant: *Lihong Han*[1]; Xueyuan Nie[1]; Qiang Zhang[1]; Zhizhong Sun[1]; Henry Hu[1]; [1]University of Windsor

AJ62 magnesium alloy was prepared by squeeze casting. The fine grain structure formed in the thin skin layer close to the surface of the castings while the central region of the casting exhibited coarse microstructure. The potentiodynamic polarization and the electrochemical impedance spectroscopy (EIS) experiments were performed by using EC-LAB SP-150 electrochemical apparatus to investigate the corrosion resistances of the AJ62 alloys with different grain sizes in a salt solution and automotive coolant. The electrochemical behavior of fine microstructure was compared with that of coarse-grained AJ62 alloy. The effect of grain size on the corrosion behavior including the inter-granular corrosion rate and pitting corrosion resistance was analyzed, and the mechanisms of corrosion were discussed.

Effect of Thermo-Mechanical Treatment on Texture Evolution of Twin-Roll Cast Mg Alloys with Various Second Phase Particles: *Kyung Hun Kim*[1]; G. T. Bae[1]; J. H. Bae[1]; D. H. Kang[1]; Nack Kim[1]; [1]Pohang University of Science & Technology

Recent development of twin-roll casting technology has shown that it can efficiently produce low cost, high performance Mg alloy sheet products. They are usually subjected to thermo-mechanical treatment (TMT) such as warm rolling to modify the microstructure so that optimum combination of mechanical properties can be obtained. Among various microstructural features, grain size and texture would be mostly affected by TMT. It is of common knowledge that the poor ductility of wrought Mg alloys is due to the strong basal texture developed during TMT and thus it is important to modify the texture of Mg alloys by various TMTs. In the present study, effect of TMT on the texture evolution has been investigated. Alloy systems investigated are ZM, ZMA, and ZE alloys which have different types of second phase particles. Correlation between the texture and tensile properties will be made and the effect of TMT conditions.

Effects of KCl on Electrical Conductivity of BaF2-LiF-MgF2 Electrolyte: *Ying Nie*[1]; Shaohua Yang[1]; Zhaowen Wang[1]; Xianwei Hu[1]; Linzhi Ma[1]; [1]Northeastern University

This paper studies on the preparation of Al-Mg alloy from MgO by molten salt electrolysis method. BaF2-LiF-MgF2 is taken as electrolyte. The CVCC method was used to measure electrical conductivity of the electrolyte. The experimental results indicated that KCl as additive can obviously improve the electrical conductivity of the electrolyte. The electrical conductivity of the electrolyte was increased with the increase of temperature and also the KCl content. The electrical conductivity of electrolyte increased from 1.4500S•cm-1 to 2.0272S•cm-1 with the mass percentage of KCl from 0 to 11% under the temperature of 850°C, the increased value is 0.5772S•cm-1.

Influence of Strontium Addition on Tensile Properties of Squeeze Cast AM60 Alloy: *Shuping Wang*[1]; Henry Hu[1]; [1]University of Windsor

The effect of strontium content on the tensile properties of squeeze cast Mg-Al-Sr alloy was investigated. Three different strontium contents, 0, 1.5, and 3.0 wt%, were added to Mg-6 wt.% Al alloy (AM60 alloy) and squeeze casting under the applied pressure of 30 MPa. The results of tensile testing indicate that the ultimate tensile strength (UTS), yield strength (YS) and elongation (Ef) of the squeeze cast Mg-Al-Sr alloy decreased with increasing strontium content. Microstructural analysis indicated that Sr content influences a number of phases present in the squeeze cast Mg-Al-Sr alloys. Also, Sr addition decreased the grain size of the alloys with increasing the strontium content. However, the increase in porosity level by Sr addition, which seems to offset its grain refinement effect, should be responsible for the decrease in tensile properties.

Microstructure and Properties of Mg-Al-Zn-(Sm) Alloys: *D. H. Xiao*[1]; [1]Central South University

Mg-9.0Al-0.8Zn alloys with 0.3%Sm were prepared by casting. The effects of scandium addition on the microstructure and mechanical of the alloys before and after heat extrusion have been investigated using microscopy analyzing and mechanical properties testing. It has been shown that the based alloys structure is mainly composed of α-Mg matrix, Mg17Al12 phase and MgAlSm phase when samarium was added. The samarium addition improves the morphology and distribution of Mg17Al12 phase. Such improved microstructure is accompanied by the improvement of mechanical properties of the extruded alloy at room temperature and high temperature. At this condition, the tensile strength and elongation of the alloy with Sm are 325 MPa and above 11% at the room temperature.

Microstructures and Mechanical Properties of the Recrystallized Mg-Zn-MM-Sn Alloy Sheets: *Beomsoo Shin*[1]; Heon Kang[1]; Donghyun Bae[1]; [1]Yonsei University

Microstructures and mechanical properties of the recrystallized Mg-Zn-MM-Sn alloy sheets, fabricated via rolling after gravity casting, were investigated. Dynamic recrystallization(DRX) occurs during hot rolling and static recrystallization(SRX) is achieved by heat treatment after cold rolling. The final grain size developed after SRX and DRX was significantly varied; the average grain sizes are measured to be 4~6 μm and 15~20 μm after SRX and DRX, respectively. Furthermore, the SRXed sample exhibits weaker crystallographic texture than the DRXed one. To investigate effects of recrystallization methods on mechanical properties, uniaxial tension tests were performed on the alloy sheets at varied temperatures and strain rates. As a result, the recrystallized Mg-Zn-MM-Sn alloy shows superior elongations at high strain rates of $10^{-1}s^{-1}$, $10^{-2}s^{-1}$ and elevated deformation temperatures, i.e. 150 ~ 250 °C than those of AZ31 magnesium alloys. The relation between ductility and grain morphology will be also presented in detail

Relationship between Internal Porosity and High Cycle Fatigue Property of Die-cast Magnesium AZ91D Alloy: Won-Guk Kang[1]; Jeoung-Han Kim[2]; Sang-Bok Lee[2]; Jung-Chul Park[3]; *Kee-Ahn Lee*[1]; [1]Andong National University; [2]KIMM; [3]RIST

High cycle fatigue properties for two different die-cast magnesium AZ91D alloys with different amount and size distribution of internal porosity were investigated. Mechanical fatigue tests were conducted under R=0.1 and 80Hz frequency condition at room temperature. The fracture surfaces and grip regions of selected specimens were examined with scanning electron microscopy. It was found that internal porosity highly influences the mechanical and fatigue properties, reducing significantly the values of elongation, tensile strength and especially high cycle fatigue strength. The difference in cycles to fatigue failure was mainly attributed to a drastic difference in nucleation site size, with ranged from several hundred μm's to several mm's. Fatigue cracks initiated at internal porosity cluster within the specimen and near the surface, depending on the relative cluster size and propagated primarily through the ß-$Mg_{17}Al_{12}$ particle laden interdendritic regions. Effects of size distribution and amount of porosity on fatigue property were also discussed.

Tensile and Compressive Deformation behavior of Ca-containing AZ31 Extrudes: *Chang Yim*[1]; Na Eun Kang[2]; Jeoung Han Kim[1]; Bong Sun You[1]; Hyeong Kyu Park[3]; [1]Korea Institute of Materials Science; [2]Metals Bank; [3]Korea Institute of Geoscience and Mineral Resources

The deformation behavior of Ca-containing AZ31 extrudes during tensile and compressive loading parallel to extrusion direction was characterized experimentally under various strain rates and temperatures. The ultimate tensile strength and maximum compressive strength were increased with increasing of strain rate and amount of Ca and decreasing of temperature. The shape of compressive flow curves was different from the shape of tensile flow curves. Tensile flow curves showed a typical convex shape showing strain hardening after yielding, but during compressive loading concave flow curve was observed in initial plastic deformation region due to activation of deformation twin as additional deformation mode. After peak stress, the flow stress was decreased gradually with continuing the deformation under tensile loading, but the flow stress was sustained constantly under compressive loading.

Tracing Nucleation and Grain Growth during Static Recrystallization of Pure Mg by EBSD: *Jianxin Zou*[1]; Jayant Jain[1]; Chadwick Sinclair[1]; [1]University of British Columbia

The "nucleation" of new grains and their subsequent growth during static recrystallization of uniaxially compressed, commercially pure Mg have been investigated using Electron backscattered diffraction (EBSD). The pure Mg samples were uniaxially compressed at room temperature and specific regions were selected from the as-deformed microstructure to follow during isothermal annealing at 573K. Our results illustrate the heterogeneous nature of recrystallization within pure Mg. This heterogeneity can be linked to the underlying deformation mechanisms operative in individual grains. As such, the recrystalization response appears to depend strongly on factors such as crystallographic orientation and starting grain size. The results gathered here clearly show the importance of local deformation state on the recrystallization process and final recrystallized grain structure within pure Mg.

Very High Cycle Fatigue (VHCF) of Thixomolded® AZ91D Magnesium Alloy: Effect of Porosity and Aging Heat-Treatments: *Raghavendra Adharapurapu*[1]; Andrew Sharp[1]; Chris Torbet[1]; J Jones[1]; Tresa Pollock[1]; [1]University of Michigan

The very-high-cycle-fatigue behavior of Thixomolded® magnesium alloy AZ91D in the 10^6-10^9 cycles regime has been studied. Fatigue experiments were conducted in an ultrasonic fatigue testing machine at ~19kHz in materials with varying casting porosity and aging heat-treatments. Fatigue properties of as-cast, naturally-aged (5 years, 20° C), hot-isostatically pressed (HIP-ed)+solution-treated (ST) and HIP+ST+peak-aged material have been characterized. The HIP process removed only solidification-induced porosity, while the gas porosity remained. The porosity in the as-cast alloy was measured to be 2.33±0.30% compared to 1.64±0.76% for HIP-ed samples and 2.14±0.50% for HIP-ed+ST samples. A comparison of the fatigue behavior of AZ91D produced by different

routes, viz., die-casting and Thixomolding®, indicated a higher endurance limit for the latter due to lower internal porosity of the castings. Fractographic analysis was carried out in all cases to correlate the porosity to the overall fatigue behavior and to examine the fatigue fracture surfaces.

Materials for High Temperature Applications: Next Generation Superalloys and Beyond: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS: Refractory Metals Committee
Program Organizers: Joseph Rigney, GE Aviation; Omer Dogan, National Energy Technology Laboratory; Donna Ballard, Air Force Research Laboratory; Shiela Woodard, Pratt & Whitney

Sunday, 6:00-8:00 PM Room: 3010
February 15, 2009 Location: Moscone West Convention Center

Aging Effects on the High Temperature Tensile Behavior of Inconel 718 Superalloy: Hui-Yun Bor[1]; Chao-Nien Wei[1]; *Chen-ming Kuo*[2]; Yan-Tang Yang[2]; Chao-Chung Tai[2]; [1]Chung-Shan Institute of Science & Technology; [2]I-Shou Univ

Standard heat treatment (HT1) for Inconel 718 superalloy is solid solution at 1095°C, 1h/AC, then aging at 955°C, 1h/AC + 720°C, 8h/FC 48°C/h to 620°C, 8h/AC. In order to study the aging effects of delta phase, two more conditions were studied in this research, namely, HT2 (no aging condition 955°C, 1h/AC) and HT3 (955°C, 3.5h/AC). Tensile tests using servohydraulic MTS system were performed at room temperature, 350°C, 450°C, 550°C, and 650°C. Since HT2 produces no delta phase, elongation to failure are the largest among these three aging conditions. Increasing the 955°C aging time, the UTS and yield stress raise, because platelet delta phase is more uniformly nucleated and more direction oriented at grain boundaries. The fractographics of tensile specimens show that both inter-granular and trans-granular fractures are observed in HT2 specimens, nevertheless, only inter-granular fracture is observed in the other two cases.

Comparative Oxidation Study of High Temperature Superalloy Fibers for Turbomachinery Sealing Applications: *Huseyin Kizil*[1]; Mahmut Aksit[2]; [1]Istanbul Technical University; [2]Sabanci University

Demand for increased power and efficiency placed superalloys in the cornerstone of most high temperature engineering applications. Use of such materials in fiber form at elevated temperatures is not desirable due to high surface to cross-section ratio. Recently, fiber components find use in contact applications in turbomachinery due to their superior wear performance. Although oxidation behavior of cobalt and nickel based alloys is well characterized in bulk form, aggravation caused by high surface to cross-section ratio requires detailed oxidation evaluation of these fibers. Present study investigates oxidation performance of eight different cobalt and nickel based superalloy fibers. Although both cobalt and nickel based fibers show similar oxidation performance at lower temperatures, nickel based fibers exhibit superior performance as exposure temperature is raised to 1100°C. The results indicate that application of cobalt based fibers should be limited by 900°C due to poor oxidation performance.

Crack Growth Behavior of Alloy 276 as Functions of Temperature and Load Ratio: *Joydeep Pal*[1]; Muhammad Hasan[1]; Ajit Roy[1]; [1]University of Nevada, Las Vegas

Austenitic nickel-base Alloy 276 has been considered as a structural material for nuclear hydrogen generation using a thermochemical cycle. In view of fluctuations in temperature during operations, this alloy may undergo thermal cyclic loading and experience crack propagation in the presence of minute flaws present in this alloy. This paper presents the results of a crack propagation study under cyclic loading (da/dN) at ambient and elevated temperatures under different load ratios (R). The data indicate that the magnitude of da/dN under a steady-state condition at all three R values (0.1, 0.2 and 0.3) was maximum at higher temperatures. Further, temperature did not influence the magnitude of threshold stress intensity range (ΔK_{th}) at a constant R value. However, the magnitude of ΔK_{th} was reduced at higher R values, irrespective of the testing temperature. The overall da/dN vs. ΔK data will be substantiated with the results of fractographic evaluations.

Effect of Rare Earth Elements on the Isothermal Oxidation Behavior of CM247LC, a Polycrystalline Ni-Base Superalloy: *Krishna Ganesan*[1]; Gerhard Fuchs[1]; Cynthia Klein[2]; Allister James[2]; [1]University of Florida; [2]Siemens Power Corporation

Minor additions of Rare Earth Elements (REE) to superalloys can lead to dramatic improvements in high temperature oxidation properties by retarding oxide scale growth rate and increasing the scale adherence under thermal cycling conditions. In this work, effect of ppm level additions of REE on the isothermal oxidation behavior of CM247LC was investigated. Fully heat treated superalloy samples were exposed to oxidizing conditions at elevated temperatures for an extended time. Comparison between different REE additions was based on weight gain data which was also used to establish the oxidation kinetics. Examination of oxide scales, internal oxidation stringers and inter-diffusion zone (IZ) was done using SEM and EPMA line scans on samples exposed for varying oxidation times. XRD was performed to identify the oxide phases while detailed analysis of IZ was done using TEM for understanding the role of REE in the oxidation mechanism.

Effects of Cyclic Loading, Temperature and Load Ratio on Plastic Deformation of Alloy 617: *Muhammad Hasan*[1]; Joydeep Pal[1]; Ajit Roy[1]; [1]University of Nevada, Las Vegas

Hydrogen generation is currently being considered under both Nuclear Hydrogen Initiative and Next Generation Nuclear Plant programs. The structural materials to be used in both programs will experience elevated temperatures approaching 950°C. Nickel-based austenitic Alloy 617 has been identified to be a suitable material by the United Stated Department of Energy for such applications. Substantial data have recently been generated on this alloy with respect to the effect of temperature and load ratio (R) on its crack propagation rate under cyclic loading (da/dN). The results indicate that the magnitude of da/dN was enhanced with a reduction in R value within the steady-state-region for all three tested temperatures. The magnitude of stress intensity range at constant R values remained identical despite a variation in testing temperature. Fractographic evaluations revealed a combination of striations and dimples on the fracture surfaces of the tested specimens. The comprehensive results are presented in this paper.

High Temperature Oxidation Behavior of SiO_2 Protective Layer Coated IN738LC Superalloy Using Combustion CVD (CCVD): *SeungKeun Oh*[1]; Youngman Kim[1]; Sang Ryu[1]; YangHong Kim[1]; [1]Chonnam National University

IN738LC is a Ni-based superalloy for high temperature applications such as gas turbine blades for generator in power plants. The oxidation behavior of IN738LC is a major factor in determining the life time of material when it is exposed in air of high temperature. Protective coatings may be a solution to improve the stability of parts made of the alloy. The processing methods, such as thermal CVD, PECVD, and the combustion CVD (CCVD), may be applied for protective coatings on IN738LC. In this study, SiO_2 protective coating is applied to an IN738LC alloy by using CCVD. TEOs($C_8H_{20}O_4Si$) and HMDS ($C_6H_{19}NSi_2$) were selected for the source material of the SiO_2 layer during CCVD. The oxidation resistance of the alloy was evaluated through TGA.

Hot Cracking of Ni Based Superalloys: *Joel Andersson*[1]; Göran Sjöberg[1]; Aurélien Alboussière[2]; [1]Volvo Aero Corp; [2]ENSICA Engineering Institute

The demand for aerospace materials that could sustain their desirable properties at even higher temperature together with processing characteristics that are better or comparable to that of alloy 718 is increasing not at least due to environmental aspects. Waspaloy which is used in the hotter sections in an aero engine because of its higher working capability compared to alloy 718 face some drawbacks as cost and processing are not as good. However, a newly developed alloy, Allvac 718Plus is claimed to have a working capability in between 718 (650C) and Waspaloy (750) with processing characteristics similar to that of alloy 718. In this study the susceptibility towards hot cracking of highly restrained welds in alloy 718, Waspaloy and Allvac 718Plus are investigated. Repair welding of machined grooves together with dsc and microscopy techniques for metallurgical investigation are performed.

Interface Structure and Chemical Stability of Continuous Mo Wire Reinforced NiAl Composites: Jia Song[1]; *Weiping Hu*[1]; Günter Gottstein[1]; [1]Institute of Physical Metallurgy and Metal Physics

Refractory metal Mo has a high melting point (2617 °C), a high strength (about 500~700 MPa) associated with a good ductility (> 15% at fracture elongation) at room temperature as well as good thermal conduction and good

thermal stability at high temperatures. Due to these valuable properties it has been tried in the present study to reinforce NiAl with continuous Mo wires in order to improve the ductility at RT and enhance the creep resistance at high temperatures of NiAl. The diameter of the used Mo wire is 125 µm. Mo wire reinforced NiAl composites were produced as following: at first, the Mo wires were coated with NiAl by PVD. The coating thickness is about 20~50 µm that is corresponding to about 30~50% volume fraction of Mo wire. The matrix-coated Mo wires were then put into a channel die for diffusion bonding. The hot pressing parameters were at 1300°C under 40 MPa in vacuum for 1 hour. Interface structure and chemistry of the composites were characterized by means of electron microscopy (HRTEM, SEM) and microanalysis (EDX, EELS and electron diffraction). Results of micro-characterization demonstrated that a continuous brittle Mo3Al reaction layer with a thickness of about 5 µm has been formed between Mo wire and NiAl matrix during diffusion bonding. Behind Mo3Al reaction layer inner Mo wire the Al concentration reached to about 1.6 at% that indicated an over-saturated dissolution of Al in Mo matrix. The formation of Mo3Al reaction layer and diffusion of Al into Mo caused Al-dilution in NiAl near interface and led to a deviation of matrix composition from the region far away from interface. Possible influence of the interface structure and chemistry on mechanical properties of Mo wire reinforced NiAl composites are discussed.

Lattice Misfit Measurements of Ruthenium-Bearing Nickel-Base Superalloys: *Jestine Ang*[1]; Hon Tong Pang[1]; Vassili Vorontsov[1]; Howard Stone[1]; Catherine Rae[1]; [1]University of Cambridge

Under intermediate creep conditions, a superalloy with a highly negative misfit will spontaneously develop a 'labyrinth' structure, rafting in all six <001> directions, irrespective of the direction of the applied stress; consequently, premature creep failure occurs. As there is generally a positive correlation between the refractory content and lattice misfit, we seek to quantify the effect of lattice misfit on creep, as this will enable us to determine the threshold of useful refractory element additions. A series of eight ruthenium-containing superalloys were designed using DoE to evaulate the effects of Co, Mo Ru and W on mechanical properties. Alloys with representative low, intermediate and high misfit values will be compared. Specimens used were interrupted at the end of primary creep. Misfit values calculated using the lattice parameters of γ and γ' measured at the high energy x-ray synchrotron facility, ESRF, will be compared to misfit values estimated by JMatPro®.

Microstructural Analysis of Nickel Base Super Alloy, IN 738LC in Different Time Temperature Exposure: *Babak Jahani*[1]; [1]Toos Gashtavar

Nickel base superalloys are widely used for gas turbine blading and other related parts at high temperatures. These alloys are vacuum melted and investment casted for high quality casts and heat treated to get excellent mechanical properties at high temperatures. The alloy used in this investigation was IN_738LC and heat treatment adopted for this alloy is solution treatment at 1120 C for two hours and air cooled to the room temperature and then aged for 24 hours at 845 C. By means of scanning electron microscopy (SEM), hardness testing and EDS, the changes in characteristics during different aging times, the size of particles and characteristic of carbides have been studied. Room temperature hardness of the superalloy decreases with increasing in aging temperature. In the a specified temperature, the hardness of the alloy increases at first but after reaching to a peak, the hardness begings to decreasing.

Microstructure and Mechanical Properties of Direct Aged 718Plus® Alloy: *Erin McDevitt*[1]; James Bentley[2]; Wei-Di Cao[1]; [1]Allvac; [2]Oak Ridge National Laboratory

ATI 718Plus® alloy is a new gamma-prime strengthened Ni-based superalloy that has a 100ºF increase in temperature capability compared to 718 alloy, good hot working characteristics and relatively low cost. Direct aging has been demonstrated to be effective at providing an increase in strength and a corresponding improvement in stress rupture performance. The fine scale microstructure of 718Plus in the direct-aged and solution-treated and aged conditions was characterized using analytical and conventional transmission electron microscopy. The elemental partitioning among the gamma-prime, delta, and austenite phases, the composition of the grain boundary delta-phase, and grain boundary segregation of Mo and Nb, will be discussed and compared to alloy 718. A portion of this research was conducted at the ORNL SHaRE User Facility, which is sponsored by the Division of Scientific User Facilities, Office of Basic Energy Sciences, U.S. Department of Energy.

Oxidation Behavior of Inconel 617 Surface Treated by Al-Pack Cementation: *Tae Sun Jo*[1]; Sang Gil Park[1]; Dong-Seong Kim[1]; Ji Yeon Park[2]; Young Do Kim[1]; [1]Hanyang University; [2]Korea Atomic Energy Research Institute

Inconel 617 is a candidate material for the high temperature applications such as turbine blades, structural materials for nuclear reactors, and high-temperature gas-cooled reactors. In this work, the oxidation behavior of Inconel 617 after Al-pack cementation was studied by exposure to air for 1000 hr at 950°C. Al-pack cementation was carried out at 800°C ~ 1000°C for 1 h in Ar using an Al2O3 crucible containing the specimen and a powder mixture of Al : Al2O3 : NH4Cl = 15g : 83g : 2g. The coating layer phase after Al-pack cementation was confirmed as NiAl and Ni2Al3 by EPMA and XRD. The thickness of coating layer was increased with increasing temperature. After exposure at 950°C, the phase analysis of coating layer was carried out by TEM, SEM, and EPMA. The hardness of coating layer was measured by nanoindentation. The oxidation resistance and hardness of surface after Al-pack cementation have improved.

Rapid Synthesis and Consolidation of Nanostructured WSi2-SiC from Mechanically Activated Powders by Pulsed Current Activated Heating: In-Jin Shon[1]; *Jeong-Hwan Park*[2]; Kee-Do Woo[2]; Jin-Kook Yoon[3]; [1]Division of Advanced Materials Engineering, the Research Center of Industrial Technology, Chonbuk National University; [2]Division of Advanced Materials Engineering and the Research Center of Industrial Technology, Engineering College, Chonbuk National University; [3]Advanced Functional Materials Research Center, Korea Institute of Science and Technology

WSi2 has an attractive combination of properties, including high melting temperature, high modulus, high oxidation resistance in air, and a relatively low density. To improve on its mechanical properties, the approach commonly utilized has been the addition of a second phase to form composite and to make nanostructured materials. Dense nanostructured WSi2-SiC composite was synthesized by pulsed current activated heating within 2 minute in one step from mechanically activated powders of WC and 3Si. Highly dense WSi2-SiC with relative density of up to 99.9% was simultaneously synthesized and consolidated under simultaneous application of a 80 MPa pressure and the pulsed current. The average grain sizes of WSi2 and SiC were about 47 nm and 38 nm, respectively. The average hardness and fracture toughness values obtained were 1698 kg/mm2 and 4.8 MPa•m1/2, respectively. The present fracture toughness and hardness are higher than those(3.3 MPa·m1/2, 1375 Kg/mm2) of monolithic WSi2.

Study of the Effects of Fe and Ti Additions on the Microstructure of Nb-18Si-5Sn Based Alloys: *Panayiotis Tsakiropoulos*[1]; Nikos Vellios[2]; [1]The University of Sheffield; [2]University of Surrey

In developmental Nb silicide based alloys improvement of their environmental behaviour has been reported when alloying with Fe, Sn and Ti. In the presence of Cr and Ti and the absence of Sn in the aforementioned alloys, the addition of Fe is claimed to enhance the formation of Laves phase, and thus to be beneficial regarding oxidation. However, there is very limited literature regarding the role of Fe and Sn in the microstructure of Nb silicide alloys in the absence of Cr. The motivation for this work was to study the synergistic effects of Sn and Fe in the presence of Ti on the microstructure of Nb-18Si silicide based alloys. The paper will discuss the role of Fe in phase selection with particular reference to the niobium solid solution, Nb3Sn, the niobium Nb3Si and 5-3 silicides, alphaNb5Si3 and betaNb5Si3, and Laves phase as well as the hardness of the silicides.

TEM Observation of Ti-47Al-2Cr Alloy, Refined by Cyclic Heat Treatment: Hesam Shakoorian[1]; Saeed Heshmati-manesh[1]; *Mahmoud Nili-ahmadabadi*[1]; Hassan Ghassemi Armaki[2]; [1]University of Tehran; [2]Tohoku University

TiAl intermetallics are excellent candidates for high temperature applications for their unique properties. However, they suffer from severe embrittlement and low formability at room temperature. Grain refinement could be a solution for this problem and could improve ductility and formability at low temperatures. In this research, the grain refinement of Ti-47Al-2Cr alloy was achieved by cyclic heat treatment. In each heat treatment cycle Massive transformations was formed. The microstructure was investigated by optical and transmission electron microscopy. Some defects such as stacking faults were observed in the microstructures resulted from massive transformation. It is supposed that formation of these faults play an important role during grain refinement. Finally, the optimum heat treatment route was selected and the mechanism of the grain refinement was discussed by means of electron microscopy.

Temperature Dependence of the Lattice Misfit of Rhenium and Ruthenium Containing Nickel-Base Superalloys: *Steffen Neumeier*[1]; Sigrid Schwub[1]; Florian Pyczak[2]; Mathias Göken[1]; [1]University of Erlangen-Nuremberg; [2]GKSS Research Centre Geesthacht

Rhenium and ruthenium in 4th generation nickel-base superalloys increase the lattice misfit between the γ- and γ'-phase. The lattice misfit varies with temperature and its magnitude determines the evolution of the γ/γ'-microstructure during creep. The lattice misfit of several experimental alloys with systematically varied rhenium and ruthenium contents was investigated at temperatures up to 1100 °C by X-ray diffraction. It was found that the lattice misfit depends strongly on the chemical composition of the alloys and the partitioning behavior of the alloying elements. The hardness of the phases γ- and γ' measured by nanoindentation in an atomic force microscope both correspond well with the partitioning behavior and X-ray results. Also the temperature dependence of the lattice misfit is modified by rhenium and ruthenium. The change of the lattice misfit with temperature is significantly smaller in rhenium-containing alloys compared to rhenium-free alloys and even less pronounced in ruthenium-containing alloys.

Temperature Dependent Elastic Constants of Directionally Solidified Superalloys: *Chen-ming Kuo*[1]; [1]I-Shou University

Directionally solidified superalloys have been extensively used as turbine blade materials to improve creep-rupture and thermal fatigue performances. Turbine blades are subjected to fluctuant temperature changes. Precise knowledge of material behavior at various temperatures is essential in design and service life evaluation of turbine blades. In this study, Wells' averaging method is extended to consider temperature dependent engineering elastic constants. Although no existing theory predicts the temperature dependence engineering elastic constants, these constants could be estimated based upon very limited experimental data of solidification direction specimens and other temperature dependent materials data. Excellent agreement is observed between estimations and experimental data of 45° and 90° off DS direction specimens. Temperature dependent moduli and Poisson's ratios of nickel-based superalloy DS plates are also proposed.

The Effect of Thermal Exposure on the Microstructure and Properties of a RENE-80 Superalloy: *Saeed Farahany*[1]; Mehrdad Aghaie-Khafri[1]; [1]K.N.T University of Technology

The Ni-base superalloy RENE-80 is widely used in manufactring of the first stage blades of gas turbine engines.The influence of long time heating at 800 and 850 °C for 100 ,500,750 and 1000 hr on the microstructure, hardness, stress-rupture and mechanical properties have been investigated. Test specimens for creep tests were prepared from the heat treated and aged materials according to the ASTM-E139. Creep behaviour of blade alloy are generally determined by means of a test in which a constant uniaxial load and temperatures, 191Mpa and 982°C according to GE standard. Results of microstructure study by means of optical and scanning electron microscopy showed the particle gamaprime coarsened according to LSW theory. This phenomenon caused to decrease of hardness, creep strength and creep life time.

Theories and Computational Models for Internal Oxidation: *John Morral*[1]; Ximiao Pan[1]; Yali Li[2]; Yunzhi Wang[1]; [1]Ohio State University; [2]Shell Global Solutions, Inc

Internal oxidation is a well known phenomenon that results from gas-solid reactions. An example is oxygen diffusing into a Ni-Al alloy to form sub-surface alumina particles. Recent attempts to revise the classical theory based on a local equilibrium (LE) approach have suffered from a lack of experimental or modeling evidence. In this work the results of DICTRA style and phase field models of internal oxidation will be presented and compared with both the classical and LE theories. The detailed information provided from such models are a better test than most previous internal oxidation experiments which failed to measure concentration and precipitate volume fraction profiles, which are critical to distinguishing between the theories.

Time and Temperature Dependent Deformation of Alloy 617: *Muhammad Hasan*[1]; Joydeep Pal[1]; Ajit Roy[1]; Sudin Chatterjee[1]; [1]University of Nevada, Las Vegas

The heat exchangers to be used in the proposed nuclear hydrogen generation under the Next Generation Nuclear Plant (NGNP) program must withstand a maximum operating temperature of 950°C. In view of its superior high temperature deformation resistance, Alloy 617 has been identified to be a suitable candidate material for such application. Due to a variation in temperature during operations this alloy may undergo time-dependent anelastic deformation (Creep) at different temperatures under a sustained loading condition. Classical creep curves have been generated involving Alloy 617 at 750, 850 and 950°C. While three stage plots were observed at 950°C within a short duration (300hr), testing at 750 and 850°C exhibited primary and secondary stages alone of creep deformation following 1000hr. A detailed analysis of the creep including the creep rate and activation energy will be presented in this paper.

Mechanical Behavior of Nanostructured Materials: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Xinghang Zhang, Texas A & M University; Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Sunday, 6:00-8:00 PM Room: 3024
February 15, 2009 Location: Moscone West Convention Center

3D Dislocation Dynamics Simulations of Thin Rods under Uniaxial Tension: *Caizhi Zhou*[1]; Richard Richard[1]; [1]Iowa State University and Ames Laboratory

High strength, high hardening rates and abnormal Bauschinger effects in thin films have been attributed to constraints on dislocation motion and dislocation interactions. To understand these phenomena, 3-D dislocation dynamics (DD) simulations have been used to investigate the dislocation interactions in single crystal FCC thin rods with the same dimensions and initial loading conditions as those in an experimental program employing nanoscale tensile testing. The full 3D simulations were carried out at multiple stain rates and with different initial dislocation densities under uniaxial tension in various loading directions. Simulations were compared directly with the experimental results both to validate the 3D DD simulations and to elucidate mechanisms of dislocation interactions in small-scale samples.

Adhesion and Cohesion of Hard Transparent Coatings on Polymer Substrates: *Ani Kamer*[1]; Reinhold Dauskardt[1]; [1]Stanford University

Acrylics and polycarbonates are tough and light and have replaced glass in many applications. However, the surface of plastics is prone to scratching and water absorption and hard transparent coatings with high adhesion are critical for reliable function. Sol-gel derived hybrid coatings based on polysiloxanes have proven very versatile in terms of high hardness, ease of additive incorporation and optical properties, although their adhesion to plastics is not well characterized or understood. We report on quantitative thin film techniques to characterize the adhesion energy of hard transparent coatings on elastically soft polymethylmethacrylate substrates. In addition, cohesive properties are reported using channel cracking methods. The subcritical adhesive and cohesive crack growth rates are reported for a range of temperatures and moist and chemically active environments. Implications for the reliability and service life of coated plastics are discussed.

Analysis of Deformation Induced Lattice Defects in SPD Processed fcc Nanometals by DSC: *Daria Setman*[1]; Michael Kerber[1]; Michael Zehetbauer[1]; [1]Physics of Nanostructured Materials

For the strength, ductility and stability of nanometals processed by Severe Plastic Deformation (SPD) the nature, distribution and density of deformation induced lattice defects is crucial. Methods of annealing resistometry and differential scanning calorimetry (DSC) have been proven well to determine these quantities, although in case of complex vacancy defects, comparisons with X-ray line profile analyses for the dislocation density are necessary. For a given peak, the variation of heating rate in DSC allows to derive the activation enthalpy which provides informations on the defect type and the diffusion mechanism involved. Measurements of the activation energy as a function of shear strain and hydrostatic pressure are presented for SPD processed Ni 99,998% and Cu 99,99%. The variation of the activation enthalpy of the dislocation peak can be interpreted by the differences in internal strains left in the samples after SPD processing, which markedly affect their ductility and stability.

Atomistic Computer Simulations of Plasticity in Faceted Nanoparticles during Compression Test: *Dan Mordehai*[1]; Eugen Rabkin[1]; David Srolovitz[2]; [1]Department of Materials Engineering, Technion; [2]Department of Physics, Yeshiva University

We report on a series of molecular dynamics simulations of the indentation process of faceted gold nanoparticles. Firstly, we employed the Winterbottom construction to determine the nanoparticle shape according to the calculated surface energies and the adhesion between the particle and the substrate, which is a tunable parameter in our simulations. Then, the particle was compressed by a rigid flat indenter at a constant velocity. For strong adhesions between the indenter and the particle, onset of plasticity occurred in tension, when the particle was attracted to the indenter as it approached. As the adhesion between them was decreased, the jump-to-contact became elastic and dislocation nucleation occurred only in compression. The first Shockley partials nucleated at the facet corners and at the topmost surface steps, propagated toward the substrate and then spread along it. We discuss our results for different nanoparticle geometries and various adhesions between the particle and the indenter.

Characterization of a Large Plate Consolidated from Cryomilled Al 5083 Powder: *Troy Topping*[1]; Byungmin Ahn[2]; Yonghao Zhao[1]; Steven Nutt[2]; Enrique Lavernia[1]; [1]University of California, Davis; [2]University of Southern California

Aluminum alloys with nanocrystalline (NC) and ultra-fine grain (UFG) size are of interest because of their high strength – typically 30% stronger than conventionally processed alloys of the same composition. But, scalability of the materials is a concern for potential commercial and military users. This study investigates the mechanical and microstructural properties of a round, 14.4 kg plate produced by quasi-isostatic (QI) forging and subsequent rolling of cryomilled Al 5083 powder. After rolling, final dimensions of the plate are ~ 60 cm diameter by ~ 1.9 cm thick. The plate exhibits ductility and strength superior to conventional Al 5083 in tensile tests conforming to ASTM E8 standards. Microstructural investigation confirms the UFG nature of the material with a grain size distribution that accommodates plastic deformation while retaining high strength. This grain size distribution allows further strengthening of the material via cold-rolling, with deformation accommodated by multiple mechanisms.

Comparing the Texture Development during Cold Rolling of Nanocrystalline Nickel and Coarse-Grained Nickel: *Andreas Kulovits*[1]; Jorg Wiezorek[1]; [1]University of Pittsburgh

We cold rolled fully dense nanocrystalline (NC) Ni (30-40nm average grain size) up to 85% thickness reduction. In this grain size regime dislocation glide mainly facilitates plastic deformation. Grains of coarse-grained (CG) metals generally exhibit simultaneous activation of multiple glide systems. For average size grains in NC metals glide activity is limited to single dislocation glide systems in the initial stages of plastic deformation and glide dislocations react with grain boundaries that act as sources and sinks. The different dislocation behavior of NC and CG metals should impact microstructure and property evolution during plastic deformation. Here we compare the microstructural responses of NC and CG Ni to cold rolling. We determined changes in texture, boundary character, grain sizes and hardness as a function of strain by combining XRD, TEM and hardness testing. Differences in the texture evolution in NC and CG Ni are discussed in relation to dislocation behavior.

Cryomilled Commercially Pure Titanium with High Strength and Ductility: *Osman Ertorer*[1]; Troy Topping[1]; Ying Li[1]; Enrique Lavernia[1]; [1]University of California, Davis

Commercially pure titanium (GradeII) was cryomilled in a liquid argon environment and consolidated at 1073K via quasi isostatic forging (commonly known as the CERACON process). A multi-modal microstructure with the grain size range of 50-2200 nm was attained, providing balanced mechanical properties in term of tensile strength and ductility. A yield strength of 840 MPa and ultimate tensile strength of 902 MPa with 27.5% elongation to failure was measured in room temperature tensile tests. Mechanical behavior was rationalized on the basis of processing history, microstructure (multi-modal grain distribution, high-angle grain boundaries, high dislocation density), and chemistry. Accordingly the obtained high strength was attributed to reduced grain size, high dislocation density and solid solution strengthening. High ductility in combination with high strength was attributed to existence of coarse grains and high angle grain boundaries. The authors acknowledge the financial support provided by the Office of Naval Research (Grant No. ARO W911NF-06-1-0230).

Deformation of a Nano-Precipitate Strengthened Superalloy: *E-Wen Huang*[1]; Peter Liaw[1]; Yee-Lang Liu[2]; Ji-Jung Kai[2]; Lee Pike[3]; Wei-Ren Chen[4]; [1]University of Tennessee; [2]National Tsing Hua University; [3]Haynes International Inc.; [4]Oak Ridge National Laboratory

The structural properties of a nano-precipitate strengthened alloy have been studied by the small-angle neutron scattering (SANS) and the transmission-electron microscopy (TEM). The alloy is selected because the strength of the alloy is doubled by these precipitates upon the aging treatment while keeping good ductility. The SANS patterns show pronounced inter-particle correlation peaks due to the nano-precipitates. By a stochastic phenomenological model, the structure and form factors of the precipitates are determined and used to fit the experimental SANS results. We first calculate the structural information of the undeformed alloy and then confirmed the validity by transmission-electron-microscopy experiments. The SANS results show an invariance of the precipitate size and inter-precipitate distance on the deformed alloy, which suggests the change of a precipitate shape after deformation. This microstructural information resolved by SANS is in good agreement with the results obtained from the quantitative transmission-electron-microscopy (TEM) image analysis.

Effect of Nanocrystallization Conditions on the Structure and Mechanisms of Deformation of Amorphous Alloys: *N. Noskova*[1]; A. Potapov[1]; [1]Institute of Metal Physics of UD RAS

The amorphous alloys of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$, $Fe_{64}Co_{21}B_{15}$, and $Fe_5Co_{70}Si_{15}B_{10}$ prepared by the fast melt quenching on rotational Cu disc in the ribbon form with width 6-12 mm and thick 25-40 μm were investigated. Toroidal specimens were wound of these ribbons with an outer diameter 30 mm and inner diameter 25 mm. The studies of specimens of $Fe_5Co_{70}Si_{15}B_{10}$ ($\lambda S \approx 0.5 \cdot 10^{-6}$) and $Fe_{60}Co_{20}Si_5B_{15}$ ($\lambda S \approx 30 \cdot 10^{-6}$) amorphous alloys with different magnetostriction have been carried out. The influence of annealing temperature, rate of cooling, magnetic field frequency under thermomagnetic treatment (TMT) on the structure and deformation of $Fe_5Co_{70}Si_{15}B_{10}$ and $Fe_{60}Co_{20}Si_5B_{15}$ amorphous alloys samples has been studied. Amorphous-nanocrystalline alloys $Fe_{73,5}$ $Nb_1Cu_3Si_{13,5}B_9$, $Fe_{63,5}Co_{10}$ $Nb_1Cu_3Si_{13,5}B_9$, $Fe_{53,5}Co_{20}$ $Nb_1Cu_3Si_{13,5}B_9$ and $Fe_{43,5}Co_{30}$ $Nb_1Cu_3Si_{13,5}B_9$ were studied under different nanocrystallization condition. The chemical compositions of the disperse phases were determined. This work was supported by RFFI (grant -07-03-00339).

High Strength Al-Cu Based Ultrafine Eutectic Composites with Enhanced Plasticity: *Sung Woo Sohn*[1]; Jin Man Park[1]; Tae Eung Kim[1]; Won Tae Kim[2]; Do Hyang Kim[1]; [1]Yonsei University; [2]Cheongju University

Recently, ultrafine eutectic composites have received increasing attention due to their remarkable mechanical properties. In the present work, a possible way to fabricate Al-Cu based nanostructure–dendrite composites with enhanced plasticity has been investigated. Our main focus is to investigate systematically the effect of microstructure evolution on mechanical properties and the role of the third alloying elements. We prepared in-situ bulk samples with eutectic structure with ultrafine scaled lamellar spacing via an injection casting method. As a result, addition of small amount (~3at%) of the third element (Ni, Ag, Be, Sn, In, Si, Ge, Ga etc.) in Al-14at%Cu alloy effectively endows larger plastic strain reaching ~ 10% in Al-Cu-Si/Ge and notable plastic strain of ~25% in Al-Cu-Ga alloy with a reasonably high strength of ~ 1GPa due to the heterogeneities with different length-scale. Possible criteria that govern the ductile deformation behavior in Al-based ultrafine eutectic composites have also been investigated.

Introduction of Nanotwinning Structure in Copper Thin Films by Ion Bombardment Treatment: *Tsung-Cheng Chan*[1]; Chien-Neng Liao[1]; [1]National Tsing Hua University, Department of Materials Science and Engineering

An air gap structure employed in interconnect technology of integrated-circuits requires interconnecting materials of high mechanical strength and low electrical resistivity. Recently, copper with nano-scaled twins has been intensively researched due to its high yield strength, good ductility and reasonably low electrical resistivity. It has been reported that copper thin films with nano-scaled twins can be prepared by magnetron sputtering technique [1]. In this study, we have proposed a method to fabricate copper thin films with nano-scaled deformation twins by an ion bombardment treatment. Mechanical properties and microstructures of copper thin films were analyzed by nanoindentation technique and transmission electron microscopy, respectively. The effect of ion bombardment treatment on the microstructures and mechanical properties of the copper film will be discussed. Reference: 1. X. Zhang, A. Misra, H. Wang, X. H. Chen, L. Lu, K. Lu, and R. G. Hoagland, Appl. Phys. Lett. 88, 173116(2006).

Low Temperature Strain Rate Sensitivity of the Nanocrystalline Ni-20%Fe Alloy: *Elena Tabachnikova*[1]; Aleksey Podolskiy[1]; Vladimir Bengus[1]; Sergey Smirnov[1]; Mikhail Bidylo[1]; Hongqi Li[2]; Peter Liaw[2]; Hahn Choo[2]; [1]B.Verkin Institute for Low Temperature Physics and Engineering of the National Academy of Sciences of Ukraine; [2]University of Tennessee

Cryogenic mechanical properties of nanocrystalline (NC) alloys attract considerable scientific attention in last years. However, the micromechanisms of low temperature plastic deformation in NC materials are not clear yet. One of the parameters characterizing the deformation micromechanisms is the activation volume V of the process of plastic deformation. In this connection, strain dependences of V have been found in this work by measuring strain rate sensitivity of the deforming stress at temperatures 300, 170 and 77 K during uniaxial compression of rectangular specimens at initial strain rates $3 \cdot 10^{-5}$ s^{-1} and $3 \cdot 10^{-4}$ s^{-1}. It was found that values of activation volume are rather small (V ~ 20 a^3 at 300 K), and practically no strain dependence of V has been registered. The analysis carried out on this basis allowed us to suppose that controlling mechanism of plastic deformation in the NC alloy is emission of mobile dislocations from grain boundaries.

Mechanical Properties and Nanocrystallization Bbehavior of Al-Ni-La Alloys: Rina Sahu[1]; S Chatterjee[2]; *Kanai Sahoo*[1]; [1]National Metallurgical Laboratory; [2]Bengal Engineering and Science University

The mechanical properties and nanocrystallization behavior of rapidly solidified ribbons of Al-Ni-La alloys have been investigated in both as-melt spun and annealed condition using nano-indentation technique, Vicker's microhardness, differential scanning calorimetry and transmission electron microscopy. Microhardness, tensile strength and modulus of ribbons were examined with the variation of temperature and subsequently correlated with evolved microstructure. Significant improvement in properties with nanocrystallization (up to around 30 vol. %) occurs which are attributed to changes in microstructure. Scanning electron microscopy images of indented ribbons show pile up of materials in a semi-circular shear band. With increasing indenting load or with increasing nanocrystallization the material flow pattern changes to a radial distribution. These changes in mechanical properties, material flow behavior and the corresponding evolution of microstructure will be discussed.

Mechanisms of Deformation of Nanocrystalline Materials: *N. Noskova*[1]; [1]Institute of Metal Physics of UD RAS

Results of recent original studies of structure and properties of nanocrystalline metals and alloys produced by severe plastic deformation. High resolution transmission electron microscopy, scanning electron microscopy, and in situ deformation in the column of an electron microscope were used to analyze the structures and the mechanisms of plastic deformation of nanocrystalline materials. Based on the results of the investigation of deformation of fcc, bcc, and hcp nanocrystalline materials in situ in the column of an electron microscope, we can apparently assume for all types of crystal structures that, as the nanograin size decreases in a nanocrystalline material, rotational deformation modes arise upon deformation by tension, which lead to the development of mesoscopic deformation shears because of their cooperative nature. In the hcp nanocrystalline a-titanium, unlike the fcc and bcc nanocrystalline materials, deformation. This work was supported by RFFI (grant -07-03-00339).

Microstructural Evolution and Mechanical Properties in Fe-Based Nanosturctured-Dendritic Composites: *Tae Eung Kim*[1]; Jin Man Park[1]; Sung Woo Sohn[1]; Won Tae Kim[2]; Do Hyang Kim[1]; [1]Yonsei University; [2]Cheongju University

In the present study, we investigated the effect of third alloying element addition on microstructural evolution and mechanical properties of Fe-Zr, Fe-Nb and Fe-Ti in-situ nano/ultrafine eutectic composites. The microstructure of suction-cast samples (diameter: 2mm) is changed significantly by choice of additional elements (Cr, C or Ni/Mn) in Fe-7Zr, Fe-11Nb and Fe-11Ti(wt%) alloys. With the addition of C, ZrC, NbC and TiC carbides form inside the primary dendrites. It is noticeable that remarkable compressive plastic strain of 23% in Fe-11Nb-0.3C and 27.7% in Fe-11Ti-0.3C together with the reasonably high strength of ~1.4GPa is obtained presumably due to the homogeneous deformation behavior with the presence of carbide. On the other hand, with the addition of Ni/Mn, lath martensitic transformation occurs in the primary phase. As a result, Fe-Nb-Ni-Mn and Fe-Ti-Ni-Mn alloys exhibit high strength (~1.5GPa), however plasticity is significantly low (~8%) due to the presence of hard primary phase.

Nano Crystal Surface Modification Technology and Its Effects on Fatigue, Wear and Friction Characteristics: *Young Pyun*[1]; Inho Cho[2]; Jin Park[3]; Chang Min Suh[4]; [1]Sun Moon University/DesignMecha; [2]Designmecha; [3]DesignMecha Inc.; [4]Kyungpook National University, Daegu

UNSM (Ultrasonic Nano Crystal Surface Modification) technology will be introduced, which strikes the surface of a workpiece 20,000 or more times per second with 1,000 to 10,000 shots per square millimeter utilizing ultrasonic generating tool and thus brings severe plastic deformation to surface layers and induces nano crystal structure. The nano crystal structure of the surface layer is analyzed by XRD and TEM. UNSM also improves surface roughness and hardness and induces compressive residual stress in surface layers, which will in turn improve fatigue strength of the workpiece. UNSM creates micro dimples structure on surface, which will in turn also improve rolling contact fatigue strength and friction loss. The resuts of rotary bending test, tension and compression test, rolling contact fatigue test, pin-on-disc test, and friction coefficient test are carried out in order to show the UNSM effects.

Nanocrystalline Powder Consolidation in AA2124 Using Uniaxial Compaction and Severe Plastic Deformation: Hanadi Salem[1]; *Ahmed Sadek*[1]; Moataz Attallah[2]; [1]American Univ in Cairo; [2]University of Manchester

Hot and ambient compaction of AA2124 nanocrystalline powders was performed to produce bulk nanostructured materials, in combination with severe plastic deformation via warm equal channel angular processing (ECAP). Nanocrystalline powders of ~40µm particle size and ~700nm grain size were consolidated into hot and green compacts under various compaction conditions. Hot compacts with highest densities and hardness were achieved over pressure and temperature ranges of 375-450MPa and 420-480°C, respectively. Hot compaction resulted in coarsening of the initial grain structure to 2.2µm. Subsequent deformation via single-pass ECAP produced uniform fully densified bulk rods, with almost no coarsening in grain size, with subgrains 100nm in size. This processing route enhanced the hardness and compressive yield strength by 23% and 43%, respectively. Ambient compaction followed by single-pass ECAP produced macroscopically uniformly deformed rods. Nonetheless, due to lack of pre-consolidation, particle rotation under shear resulted in a significant degree of structural heterogeneity (grain size 78-500nm).

Performance Comparisons of Nanocrystalline Copper Fabricated by Room-Temperature-Molding and Vacuum-Warm-Compaction Method: *Wei Liu*[1]; Tianzu Yang[1]; Guang Chu[1]; Weifeng Liu[1]; [1]Central South University

Nanocrystalline Cu with average grain size of 20-25 nm was fabricated by room-temperature-molding method (RM) and vacuum-warm-compaction method (VWC) respectively. Scanning Electronic Microscopy (SEM), X-ray diffraction (XRD), Positron annihilation spectroscopy (PAS) and microhardness test were utilized to characterize these as-prepared nanocrystalline copper. The thermal stability, microhardness and micro-void distribution of the as-prepared nanocrystalline copper were compared and discussed in detail. The experimental results show that,compared with RM process, the increasing of density is unremarkable while the microstrain reduced during warm-compaction process. The microhardness of nanocrystalline copper prepared by VWC (2.7GPa) is higher than that prepared by RM (1.6-1.9GPa). Also, the nanocrystalline copper prepared by VWC has better thermal stability. Positron annihilation spectroscopy analysis indicates that, compared with the specimens prepared by RM, the average micro-void size and proportion of single vacancy is a little larger in nanocrystalline copper fabricated by VWC.

Plastic Flow Mechanisms in Ultra Fine Grained Pd and Pd-Ag Alloys Studied by In-Situ Tensile Tests: *Kejing Yang*[1]; Julia Ivanisenko[2]; Lilia Kurmanaeva[2]; Andrey Chuvilin[1]; Arnaud Caron[1]; Jürgen Markmann[3]; Ruslan Z. Valiev[4]; Hans-Jörg Fecht[1]; [1]University of Ulm; [2]Forschungszentrum Karlsruhe; [3]Universität des Saarlandes; [4]Ufa State Aviation Technical University

In-situ tensile testing, as a booming technique in materials analysis, expands the conventional understanding of mechanical properties, gaining insight into the deformation evolution. Its application to a systematic investigation of HPT Pd and Pd-x%Ag (x=5,20) alloys confirms enhanced strain hardening capacity by tailoring stacking fault energy of UFG materials. The plastic flow analyzed by grey scale correlation demonstrates that the Pd-20%Ag sample with the lowest stacking fault energy manifests not only largest uniform elongation but also the best resistance against strain localization after the onset of necking. Shear banding is the primary mechanism of plastic deformation after uniform elongation has been exhausted. However, unlike other samples that failed by development of existing shear bands, Pd-20%Ag surprisingly ruptured through

the catastrophical multiplication of newly-formed shear bands. Quantitative characterization of these shear bands and tentative explanations are provided.

Precipitation and Mechanical Behavior of an Al-Mg-Si Alloy Processed by ECAP: *Edgar Garcia-Sanchez*[1]; Marco Hernandez-Rodriguez[1]; Edgar Ortiz-Cuellar[1]; [1]UANL-FIME

The Equal Channel Angular Pressing (ECAP) is one of the most important SPD (Severe Plastic Deformation) methods for the production of ultrafine and nanostructured metals, and has been extensively utilized. In this work a commercial Al-Mg-Si alloy has been deformed at room temperature by multi-pass equal channel angular pressing (ECAP) to obtain submicron grained structures. The mechanical behavior was analyzed by nanoindentation tests and was associated with the microstructural state. The thermal stability of microstructure and the secondary precipitation were examined by scanning calorimetry (DSC) and transmission electron microscopy (TEM). The results showed the effect of the number of passes and post-SPD thermal treatment on the microstructural evolution and the mechanical properties.

Processing High-Strength Aluminum Alloys by ECAP at Room Temperature: *Zhichao Duan*[1]; Nguyen Chinh[2]; Cheng Xu[1]; Terence Langdon[1]; [1]University of Southern California; [2]Eötvös Loránd University

Because of the strengthening effect of precipitates, it is often difficult or impossible to process age-hardenable Al-Zn-Mg-(Cu) alloys by equal-channel angular pressing (ECAP) at room temperature. Processing at elevated temperatures is also not satisfactory because it leads to uncontrolled precipitation and/or significant grain coarsening. This paper describes alternative approaches which may be applied to successfully press these alloys at relatively low temperatures. The experimental results demonstrate that it is feasible to achieve a significant improvement in strength after only one pass in processing by ECAP.

Propagation of Buckling Delamination in Osmium-Ruthenium Films: *Wen Chung Li*[1]; Scott Roberts[2]; T. John Balk[1]; [1]University of Kentucky; [2]Semicon Associates

Due to their high thermal stability and low work function, osmium-ruthenium (OsRu) films are used as coatings for porous tungsten (W) dispenser cathodes. The grain morphology and microstructure of the films, which exert a significant influence on dispenser cathode lifetime, are affected by in-plane film stress. Electron microscopy reveals that the OsRu films consist of nanocrystalline (15 to 50 nm) columnar grains. Depending on deposition conditions, primarily the sputtering pressure and substrate biasing power, OsRu films exhibited in-plane stresses that varied greatly (between 20 MPa and 5 GPa in compression). The high residual stresses in as-deposited films led to extensive buckling delamination, in the form of telephone cords that spread over the entire film surface. This buckling propagation was observed in-situ using optical microscopy, and proceeded at an average rate of ~165 μm/s. The interplay of substrate biasing, in-plane stress and OsRu film microstructure will be discussed.

Properties and Consolidation of Binderless Nanocrystalline Tungsten Carbide by Rapid Sintering: In-Jin Shon[1]; *Byung-Ryang Kim*[2]; Min-Seok Moon[2]; Kee-Do Woo[2]; [1]Division of Advanced Materials Engineering, the Research Center of Industrial Technology, Chonbuk National University; [2]Division of Advanced Materials Engineering and the Research Center of Industrial Technology, Engineering College, Chonbuk National University

The attractive properties of WC are high melting temperature, high hardness, high thermal and electrical conductivities. Tungsten carbide find applications primarily in the cutting tool industries. In this work, we investigated the sintering of WC without the use of a binder by the high frequency induction heated sintering method. In addition, we also studied the effect of high energy ball milling on the sintering behavior, microstucture, and mechanical properties of binderless WC. The relative density of binderless WC sintered at 1240°C without high energy ball milling was about 72%, but increased with high energy ball milling time. Nearly full density (98%) of WC was obtained from high energy ball milled powder for 4 hours at the same sintering temperature. The grain size, fracture and hardness of binderless WC sintered from high energy ball milled powder for 10 hours were 87 nm, 8.1 MPa.m1/2 and 3020 kg/mm2, respectively.

Properties and Consolidation of Binderless Nanostuctured TiC from Mechanically Activated Powder by High Frequency Induction Heated Sintering: In-Jin Shon[1]; Byung-Ryang Kim[2]; *Min-Seok Moon*[2]; Kee-Do Woo[2]; [1]Division of Advanced Materials Engineering, the Research Center of Industrial Technology, Chonbuk National University; [2]Division of Advanced Materials Engineering and the Research Center of Industrial Technology, Engineering College, Chonbuk National University

Titanium carbide has a low density, relatively high thermal and electrical conductivity, high melting temperature (31000C) and high hardness. These properties have seen it used extensively in cutting tool applications and a hardening phase in composite materials.High frequency induction heated sintering is utilized to consolidate binderless nanocrystalline TiC within 3 minutes with the application of 80 MPa pressure. Nano-particle size(~25nm) of TiC is obtained by high energy ball milling for 10 hours. The relative density of TiC increases with milling time at the same sintering temperature. Nearly full density(98%) of binderless TiC is obtained using high energy ball milled powder for 1 hour at sintering temperature of 1335°C. The average grain size of the densified TiC decreases with milling time. The TiC sintered from high energy ball milled powder for 10 hours had grain size, fracture toughness and hardness values of 99 nm, 8.6 MPa.m1/2 and 2209 kg/mm2, respectively.

Reliability of Nano-Scale Au Thin Films on PDMS: *Onobu Akogwu*[1]; Marcus Eleruja[2]; Auxillia Munhutu[1]; David Kwabi[1]; Swaminathan Midthuri[3]; Wole Soboyejo[1]; [1]Princeton University; [2]The Obafemi Awolowo University; [3]University of Arkansas

This paper presents the results of a combined experimental and theoretical study of the reliability of nano-scale Au thin films on poly-di-methyl-siloxane (PDMS) substrates. The loading rate dependence and creep response of PDMS are investigated in stretching experiments, before using spring dash-pot models to characterize the observed deformation response. The mechanisms of deformation and cracking are then elucidated for nano-scale Au thin films on PDMS substrates deformed under monotonic or cyclic loading. The related changes in film resistance are examined before presenting Coffin-Manson approaches for fatigue life prediction. The implications of the results are discussed for design of robust flexible electronic structures.

Simulation and Mechanical Characterization of Open Celled Foams from MicroCT Scan Data: Bruno Notarberardino[1]; Brian Walker[2]; Philippe Young[1]; *Ash Harkara*[1]; [1]University of Exeter; [2]ARUP

Computational simulation is a very effective and valuable tool in investigating materials behavior at the micro and nano-scale level and in assessing its influence on the overall macro-scale properties. Well established computational techniques can now be used to simulate mechanical, fluid dynamics, thermal or any combined (multi-physics) phenomena at the micro and nano-scale level. Crucial to the success of such a simulation is the ability to represent the 'micro-architecture' accurately and efficiently - which has proved to be a very challenging task so far. This paper will present an innovative image-based mesh generation technique that converts 3D images of micro and nano-structures (as provided by typical Micro/NanoCT scanners) directly into high fidelity computational models. The approach provides a deeper understanding than experimental tests, and achieves more realistic model results than via analytical approaches. Real-life applications will be presented, including the densification analysis of open celled foam.

Study on Post Annealed Effect of SiOC(-H) Films by Inductive Coupled Chemical Vapor Deposition: *Teresa Oh*[1]; [1]Cheongju University

Low-k materials, low dielectric constant, inter layer dielectric material, organic thin films.

Surface Nano-Deformation of Gum Metal by In-Situ AFM Observation: *Yoshihisa Tanaka*[1]; Yang Jenn-Ming[2]; Liu Yu-Fu[3]; Yutaka Kagawa[3]; [1]National Institute for Materials Science; [2]University of California Los Angeles; [3]The University of Tokyo

Gum Metal is a newly developed beta titanium alloy which, in the cold work condition, exhibits a large non-linear elastic deformation and high strength at room temperature. This study was conducted to investigate the in-situ surface nano-deformation characteristics of a cold worked Gum Metal using an atomic force microscope (AFM). Tensile test and in-situ observations were conducted using a tensile device equipped with an AFM. Surface morphologies of the Gum Metal specimen were acquired before and after the deformation at various stages of the straining. The formation of slip bands within a grain was clearly observed

when the applied strain was approximately 2.1%. The amount of slip bands and average surface roughness was found to increase with increasing applied strain. The interaction of slip bands with the grain boundaries and mechanism of deformation of the Gum Metal based on nanoscale in-situ AFM observation will be discussed.

The Application of EBSD to Study Microstructural Development in Commercial Pure Ti Fabricated by Severe Plastic Deformation: Yongjun Chen[1]; *Hans. J. Roven*[1]; Yanjun Li[2]; Stephane Dumoulin[2]; John Walmsley[2]; [1]The Norwegian University of Science and Technology; [2]SINTEF

High resolution electron backscatter diffraction (EBSD) in conjunction with a field emission gun scanning electron microscope (FEG-SEM) has been used to study microstructural development during Equal channel pressing (ECAP) of commercial pure (CP) Ti. The use of EBSD allows one to quantitative measure in-grain orientation gradient, subgrains or cells, boundaries and microtexture. In the present paper, the commercially pure (CP) Ti with the average grain size of 22μm after rolling is refined towards a nanostructured microstructure after elevated temperature ECAP up to 8 passes. The evolutions of grain size, grain boundary structure, misoriention angles, subgrains or cells and microtexture were analysed in detail. The work also aims at revealing the dominating grain refining mechanism of HPC structured Ti during ECAP.

The Effect of Annealing on the Hardness of Electrodeposited Nanocrystalline Nickel: *Hsiao-Wei Yang*[1]; Anna Torrents Cabestany[1]; Manish Chauhan[1]; Farghalli A. Mohamed[1]; [1]University of California

The effect of annealing on the hardness of bulk electrodeposited (ED) nanocrystalline (nc) Ni having an average initial grain size of 20 nm was investigated. Hardness measurements were conducted at room temperature on specimens after annealing at different temperatures, ranging from 323 – 693 K for various annealing times. The results showed that the hardness of the material initially increased slightly with increasing annealing temperature and then decreased rapidly with increasing temperature above 500 K. It was suggested that the increase in hardness below 500 K was most likely due to the occurrence of substructural relaxation at non-equilibrium boundaries and the formation of annealing twins. Micrographs from tunneling electron microscope (TEM) show the evidence of annealing twin structure. Statistical analysis is also applied to determine the twin density and average grain size with twinning.

The Effect of HIP Temperature on a Cryomilled Al Alloy: *Troy Topping*[1]; Piers Newbery[1]; Byungmin Ahn[2]; Steven Nutt[2]; Enrique Lavernia[1]; [1]University of California, Davis; [2]University of Southern California

Al 5083 powder was cryomilled to obtain a nanocrystalline structure. Samples of the powder were hot vacuum degassed, to remove interstitial contaminants, and then consolidated by hot isostatic pressing (HIPping) at six different temperatures, before being forged at a high strain rate to produce plate material. The microstructure was characterized at the different processing stages using optical, scanning and transmission electron microscopy. The compressive properties of the as-HIPped material, plus tensile properties of the final product were evaluated. Despite grain growth as a result of HIPping, an ultra-fine grain structure was retained in the consolidated material, which consequently had increased strength over conventionally processed Al 5083. As the HIP temperature was increased, the density and grain size increased and the strength decreased, with near-full density being attained at 275°C (~0.64TM). Yield strength data indicate that both work hardening and Hall-Petch mechanisms are at work in the microstructure.

Microstructural Processes in Irradiated Materials: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Christophe Domain, Electricite De France; Gary Was, University of Michigan; Brian Wirth, University of California, Berkeley

Sunday, 6:00-8:00 PM Room: 2008
February 15, 2009 Location: Moscone West Convention Center

Session Chairs: Brian Wirth, University of California, Berkeley; Gary Was, University of Michigan; Christophe Domain, Electricite De France

A Three Feature Model of Irradiation Hardening in RPV Steels: *G. Robert Odette*[1]; Takuya Yamamoto[1]; Erik Mader[2]; [1]University of California, Santa Barbara; [2]EPRI

A new physical three-feature irradiation-hardening model (3FIHM), that is applicable over a wide range of fluxes and fluences, is described. In addition to stable matrix features (SMFs) and copper rich precipitates (CRPs), the 3FIHM treats both direct hardening and indirect sink effects of thermally unstable matrix defects (UMDs) that form and anneal under irradiation. High fluxes shift hardening to higher fluences, but also add the UMD hardening contribution. Thus the net effect of flux depends on all the embrittlement variables. The 3FIHM model is validated and calibrated by microstructural as microhardness data for both as-irradiated alloys and following low temperature post irradiation annealing to recover the UMDs. The 3FIHM model rationalizes observed hardening trends over several orders of magnitude of flux up to fluences in excess of $6x10^{19}n/cm^2$. New insights on the role of other hardening features, including late blooming Ni-Mn rich phases and dislocation loops are also discussed.

Anisotropy Changes in Pyrolytic Carbon Resulting from Proton Irradiation Induced Creep: *Anne Campbell*[1]; Rongsheng Zhou[1]; Gary Was[1]; [1]University of Michigan

High density pyrolytic carbon (PyC) is one of the structural materials used in the TRISO fuel particles for the Very High Temperature Reactor. Mechanical properties of PyC are dependent on the degree of anisotropy, so an understanding of the change in anisotropy caused by irradiation and irradiation-induced creep is imperative. Creep experiments were conducted on thin (<40μm) strip samples of high density PyC using a 2 MeV proton beam, with stresses of 20.7 and 13.8 MPa, and temperatures between 800 and 1200°C. The degree of anisotropy is characterized for samples without irradiation, after irradiation, and after irradiation creep, by analyzing the cross-sectional TEM diffraction patterns. Preliminary results show an increase in the degree of anisotropy from irradiation, and a greater increase from the addition of an applied stress. The dependence of the degree of change of anisotropy due to irradiation and stresses will be discussed along with possible mechanisms.

Assessing Composition Dependence in the Five-Frequency Model Using Kinetic Monte Carlo: *Benjamin Swoboda*[1]; Julie Tucker[1]; Dane Morgan[1]; Anton Van der Ven[2]; [1]UW-Madison; [2]University of Michigan - Ann Arbor

Diffusion of point defects play an important role in microstructural changes in irradiated materials. A widely used method for determining diffusion coefficients in materials with a dilute solute is the five-frequency model [1]. However, it is not clear over what range of compositions the dilute solute approximation is valid. Kinetic Monte Carlo is used to test the validity of the five-frequency model with increasing solute concentration. An effective Hamiltonian is defined which is equivalent to that used in the five-frequency model for dilute solute, but which can also be evaluated at all compositions. The effects of varying solute and solvent activation energies and solute-vacancy binding energies are explored. In addition, the compositional range of validity of an ab initio determined five-frequency model for the Ni-Cr and Ni-Fe system is discussed. [1] A.D. Leclaire, Solute Diffusion in Dilute Alloys, Journal of Nuclear Materials 69-70, 70 (1978).

Atomistic Simulation of Dislocation-Dislocation Loop Interactions in BCC Fe: *Hyon-Jee Lee*[1]; Brian Wirth[1]; [1]UC Berkeley

Ferritic steels exhibit large interstitial loops with Burgers vector ½<111> and <100>, which can be formed by agglomeration of point defects, either interstitials or vacancies, that are produced upon irradiation, or following

plastic deformation. Notably, recent transmission electron microscopy (TEM) experiments showed that nano-meter sized prismatic dislocation loops in body centered cubic (BCC) iron (Fe) exhibit fast one-dimensional motion. These dislocation loops along with other defects, such as voids and/or small precipitates, ultimately cause hardening, loss of ductility, and flow localization upon interaction with gliding dislocations. In order to understand the micro-structural process that controls the change in mechanical properties of BCC Fe, molecular dynamics (MD) and molecular statics (MS) simulations are used. The atomistic nature of interstitial loops, such as the formation mechanism, structure, mobility, and gliding behavior, as well, the interaction behavior of dislocation loops with screw dislocations will be discussed using both dynamic and static methods.

Characterization of Ion Irradiation Defects in Glassy Polymeric Carbon and Pyrolytic Graphite - A Comparison: *Malek Abunaemeh*[1]; Bopha Chhay[1]; Cydale Smith[1]; Claudiu Muntele[1]; Yanbin Chen[2]; R. Zhou[2]; Lumin Wang[2]; Gary Was[2]; Daryush Ila[1]; [1]Alabama A&M University; [2]University of Michigan

TRISO fuel that is planned to be used in some of the Generation IV nuclear reactor designs consists of a fuel kernel of UOx coated in several layers of materials with different functions. Pyrolytic carbon (PyC) is considered for two of these layers. In this study we are investigating the possibility of using glassy polymeric carbon (GPC) as an alternative to PyC. GPC is a type of polymer used for products where a lightweight material that can maintain dimensional and chemical stability in adverse environment and very high temperatures (up to 3000°C). We are looking at comparing the defects that appear in the structures of GPC and PyC after different doses irradiation with 2 MeV proton, 3 MeV N and 5 MeV Si bombardment at 600 and 800°C by using Fourier Transform Infrared spectroscopy, Raman Spectroscopy, Hall effect measurements, transmission electron microscopy and electron diffraction.

Comparison of the Stability of Different Oxides under Electron Irradiation in Martensitic ODS Steels: *Isabelle Monnet*[1]; Yann de Carlan[2]; Joël Ribis[2]; Philippe Dubuisson[3]; [1]CIMAP; [2]CEA/DEN/DMN/SRMA; [3]CEA/DEN/DMN

ODS ferritic materials are reinforced by a dispersion of oxides and nano-oxides within the matrix. Those materials are considered for different nuclear applications under irradiation but previous experimental studies have shown a partial dissolution of some oxides under neutron irradiation. In this work, electron irradiations were used to evaluate the stability of the oxides and to simulate the neutron irradiations. Four ferritic steels (Fe-9Cr-1Mo), reinforced respectively by Al2O3, MgO, MgAl2O4 and Y2O3, were studied. These materials were irradiated with 1MeV or 1,2 MeV electrons in a High Voltage Electron Microscope. This technique allows to follow one single oxide and to determine the evolution of its size during the irradiation. The results show that the dissolution rate of the oxides depends on the type of the oxide, on the temperature and on the irradiation dose.

Dilute Fe Alloys under Irradiation Modeled by an Ab Initio Based AKMC Model: Solute-Interstitial Clusters Properties: *Christophe Domain*[1]; Pär Olsson[1]; Raoul Ngayam-Happy[1]; Charlotte Becquart[2]; [1]EDF R&D; [2]LMPGM

The evolution of the microstructure of dilute Fe-CuniMnSiP alloys under neutron irradiation, has been modeled using a multiscale approach based on ab initio and kinetic Monte Carlo simulations. Neutron irradiation of pressure vessel steels lead to the formation of point defects and solute clusters that are responsible for the vessel embrittlement. Intrinsic properties of point defect clusters as well as their interaction with solute atoms have been studied using DFT. Our atomistic kinetic Monte Carlo (AKMC) model treats thus self interstitials in addition to vacancies. The parameters regarding the self interstitial atoms were based on ab initio predictions and adjusted on isochronal annealing data. Of special interest is the modeling of the solute – self interstitial clusters for which complex configurations has been obtained ab initio. AKMC results on irradiation using this improved parameterization will be presented and discussed.

Effect of Vacancy Supersaturation on Curvature Driven Grain Boundary Migration: *Moneesh Upmanyu*[1]; Branden Kappes[1]; [1]Colorado School of Mines

Considerable debate occurs on the effect of vacancy supersaturation on grain growth. While it is well known that grain coarsening results in vacancy generation and therefore opposes motion by mean curvature, the effect of an extrinsic non-equilibrium vacancy concentration, typically present in irradiated crystalline microstructures, on grain boundary properties is unknown. To this end, we have performed embedded-atom-method (EAM) based molecular dynamics simulations aimed at quantifying the effect of vacancy supersaturation on curvature driven grain boundary motion in Al. A bicrystal system is employed, consisting of an embedded cylindrical grain misoriented with respected to the matrix grain so as to form a high-symmetry, high angle <111> tilt grain boundary. Randomly dispersed vacancies are introduced into the simulation cell. The dynamics of the grain boundary allows us to extract the boundary mobility and stiffness. Results on the variation of these two quantities with vacancy undersaturation and supersaturation are presented and discussed.

Examining Lot-to-Lot Variability of HT-9 with Response to Irradiation: *Yong Yang*[1]; Kevin Field[1]; Alicia Certain[1]; Todd Allen[1]; [1]University of Wisconsin-Madison

This paper studies whether the lot-to-lot variability of HT-9 is a concern that would affect the potential application as the fuel cladding material in the next generation of nuclear reactors. Three different heats of HT-9 from EBR-II, ORNL and LANL were irradiated using a proton beam with various doses at two different temperatures of 300°C and 500°C, respectively. The post-irradiation examination includes the void swelling, dislocation structures, precipitates, as well as the radiation induced segregations along prior austenite grain boundaries and the martensite lath boundaries. The microhardness measurement was performed to study the irradiation hardening effects in the different heats, which can be correlated with the irradiated dislocation structures and the pre-irradiation microstructural differences.

Irradiation-Induced Precipitation and Dislocation Loop Formation in Ferritic-Martensitic Alloy HCM12A: *Zhijie Jiao*[1]; Janelle Penisten[1]; Gary Was[1]; [1]University of Michigan

Ferritic-martensitic (F-M) alloy HCM12A is under consideration as cladding and structural materials for advanced reactor systems. The copper content in this alloy could lead to irradiation-induced precipitation that may alter the mechanical properties of the alloy. However, little work has been done on this subject for this alloy. In this study, HCM12A was irradiated using 2 MeV protons to doses of 3, 7 and 10 dpa at 400 and 500C. The irradiation-induced precipitation was characterized using transmission electron microscopy (TEM) as well as atom probe tomography (APT). Preliminary results show that a significant amount of Ni- and Cu- rich precipitates, a few nanometers in size, were formed when the alloy was irradiated to 7 dpa at 400C. The evolution of precipitates and loops with dose and temperature will be discussed.

Lattice Strain and Damage Evolution of 9-12%Cr Ferritic/Martinsetic during In-Situ Tensile Test by X-Ray Diffraction and Small Angle Scattering: *Xiao Pan*[1]; Xianglin Wu[1]; Jonathon Almer[2]; Jan Ilavsky[2]; James Stubbins[1]; [1]UIUC; [2]Argonne National Laboratory

Radiation-induced defect structures are known to elevate material yield strength and reduce material ductility. These changes substantially reduce uniform elongation compared to the unirradiated material condition so that the small strains induce plastic instability. This process is commonly known as flow localization. Recent work by our group indicates that the tendency for embrittlement in face-centered cubic (FCC) materials is controlled by the critical stress, regardless of irradiation dose but strongly depending on temperature. It is believed that the critical stress, an intrinsic material property, is associated with the interfacial strength between particle and matrix which determines the void nucleation. In-situ tensile tests have been performed by high energy X-ray diffraction in Advanced Photon Source at Argonne National Laboratory. The lattice strain and stress evolution during deformation and corresponding damage evolution will be reported in the paper. In addition, the existed void nucleation criterion will be evaluated.

Material Selection for Spallation Neutron Windows - Application to ADS Prototypes: Manuel Perlado[1]; *Fernando Sordo*[1]; Alberto Abanades[1]; Jose Maria Martinez-Val[1]; Emma del Rio[1]; Enrique Martinez[1]; Antonio Lafuente[1]; Shalom Eliezer[1]; [1]Universidad Politécnica Madrid

High performance neutron sources are being proposed for many scientific and industrial applications: material studies, hybrids reactors and transmutation of nuclear wastes. In the case of transmutation of nuclear wastes, accelerator driven systems (ADS) are proposed as one of the main technical options for such purpose, being a high performance spallation neutron source a critical element for its operation. Inertial Fusion neutron sources will also be considered. The material selection for the window component become of paramount importance. We show an integral analysis of this spallation sources taking as reference the proposal in the framework of European projects. Our estimations show that

Titanium and Vanadium alloys are more suitable than steel as structural material for an industrial ADS beam window, mostly due to its irradiation damage resistance. An analysis of the microstructural knowledge from simulation and experiments of these materials will also close the estimation of this evaluation of materials.

Microstructural Development in Fe-Cr Alloys upon Ion Implantation: *Amuthan Ramar*[1]; Robin Schaeublin[1]; [1]CRPP - EPFL

Ferritic / Martensitic (F/M) steels shows good resistance to swelling and low defect accumulation upon irradiation relative to austenitic steels. A number of experimental studies in ferritic alloys, have shown the existence of large self-interstitial atoms loops with Burgers vector b =1/2 <111> and b =<100> in the bulk, which may provide a significant contribution to the hardening caused during irradiation at lower temperatures. Ab-initio simulation results shows that, initially loops with b =1/2<110> which is lower in energy should be formed. They would transform to loops with b =1/2 <111> with the increase in the Self-interstitial atoms and latter to loop with b =<100>. One of the challenge in this field is the observation of the loops with b =1/2<110> experimentally. The loop with b =1/2<110> could be analyzed in the microscope using weak beam g.b analysis, but their size is at the limit of the microscope's resolution.

Modeling of Carbon Diffusion in Ultrafine Grain Tungsten during Rapid Annealing: *Shahram Sharafat*[1]; Manmeet Narula[1]; Aaron Aoyama[1]; Nasr Ghoniem[1]; Nalin Parikh[2]; [1]University of California Los Angeles; [2]University of North Carolina at Chapel Hill

The High Average Power Laser (HAPL) project is pursuing development of an IFE power reactor with a solid First Wall. Typical operation exposes the FW tungsten armor to a variety of high energy ions including carbon at 10 Hz and will raise the surface temperature to about 2400 C within 10 ns between shots. Implantation of carbon can result in formation of WCs which decrease thermo-physical properties of tungsten. To estimate WC formation rates, carbon diffusion in polycrystalline tungsten must be determined. A carbon diffusion model was set up and solved for a 3-dimensional control volume consisting of ultra-fine grain structured tungsten. The diffusion of carbon during the rapid temperature transients were modeled and compared with diffusion in single crystals. It is shown that the temperature transient assists diffusion of carbon toward the implantation surface instead of pushing the carbon deeper into the tungsten armor.

Modeling of Point Defect Cluster Evolution under Pulse Irradiation: *Stanislav Golubov*[1]; Roger Stoller[1]; [1]Oak Ridge National Lab

Nucleation, growth and coarsening of point defect clusters or secondary phase precipitates are responsible for numerous changes that occur in the physical and mechanical properties of materials during irradiation. Most of the theoretical investigations of these phenomena have focused on continuous irradiation with little attention paid to pulsed irradiation conditions. However, the problem of pulsed irradiation is quite important when one considers that pulsing is an inherent aspect of current and proposed irradiation facilities such as the MTS (LANL), SNS (ORNL), IFMIF, and ITER. Thus, it is important to develop a fundamental understanding of material behavior under pulsed irradiation conditions, and the differences between this and the continuous irradiation characteristic of fission reactors in which a substantial amount of irradiation data has been obtained. Material response to pulsed and continuous irradiation conditions has been characterized using a new numerical solution to the kinetic equations describing point defect cluster dynamics.

On the Effects of Mechanical Alloying Ball Milling Parameters on the Grain Size and Nanofeature Distributions in Nanostructured Ferritic Alloys: *Nicholas Cunningham*[1]; G. Robert Odette[1]; Charles Eiselt[2]; Michael Salston[1]; Anton Meoslang[2]; Seward Gareth[1]; [1]University of California, Santa Barbara; [2]Forschungszentrum Karlsruhe

A previously reported extensive study of the effect of a large matrix of ball milling parameters on the homogeneity of Y-Ti-O enriched nanofeatures (NFs) in nanostructured ferritic alloys (NFA) was based on characterizing the grain size distribution by SEM and image analysis methods. The grain size distributions were assumed act as a surrogate to the NF distributions: small grains are stabilized by high NF concentrations, while larger grains contain fewer NFs. Larger grains and lower NF concentrations are believed to result in lower NFA strength and fracture toughness. In the current study a combination of electron back scattering diffraction (EBSD), nanoindentation, tensile and strain rate jump creep tests, small angle neutron scattering and transmission electron microscopy characterization techniques are used to refine the evaluation of the effects of ball milling parameters on the NFs and their spatial distributions, as well as the NFA grain sizes and strength levels.

Optical and Electrical Properties of Al Doped ZnO Transparent Film Irradiated by Co-60 Radioisotope: Ozge Ozdemir[1]; Huseyin Cimenoglu[1]; *Nilgun Baydogan*[1]; Hande Sengel[2]; Fehiman Akman[2]; Ates Parlar[2]; [1]Istanbul Technical University; [2]Sisecam

Al doped ZnO (Al:ZnO) thin film was prepared by sol-gel dip coating technique and deposited on soda-lime-silicate glass. After the annealing processes of Al:ZnO film, the specimens were cooled down to room temperature in air. Co-60 radioisotope was used to investigate gama radiation effect on Al:ZnO film. The color of the Al-doped ZnO is the same as that of the undoped ZnO film. However the doping of Al ions and irradiation of the film was effected on the defect chemistry of ZnO. The effects of Al dopant on the electrical conductivity of irradiated ZnO were investigated. The doping of Al increased the electrical conductivity of ZnO. The resistivity was measured by a four point probe. The variations of the resistivity, charge carrier concentration, and carrier mobility for the ZnO:Al film were investigated.

Point Defects as Effective Pinning Centers in High-Tc Single Crystals: *Yuri Petrusenko*[1]; Alexander Bondarenko[1]; Ivan Neklyudov[1]; [1]National Science Center - Kharkov Institute of Physics & Technology

The effect of low-temperature 2.5 MeV electron irradiation on the critical temperature T_c and transport critical current J_c in $YBa_2Cu_3O_{7-x}$ single crystals is reported. It is demonstrated that point defects generated by the ~MeV electron beam serve as effective pinning centers of the magnetic flux in high-T_c crystals, which give a substantial rise to the critical current density of irradiated superconductors. It is also found that irradiation to a dose of $3x10^{18}$ electrons/cm^2 slightly decreases the critical temperature, $\Delta T_c \approx -2$ K. At the same time, it substantially increases the J_c value (up to 30 times) and drastically changes the shape of the $J_c(\alpha)$–dependence, where α is the angle between the magnetic field vector and the *ab*-plane of the crystal. These changes are caused by the transition of the vortex lattice from the ordered state to disordered, this being due to an increase in the point defect concentration.

SANS Investigation of Irradiation-Induced Phase Separation in a Binary Fe-Cr Alloy: *Andreas Ulbricht*[1]; Frank Bergner[1]; Cornelia Heintze[1]; [1]Forschungszentrum Dresden-Rossendorf

Ferritic-martensitic chromium steels are candidate materials for future applications in both Gen-IV fission and fusion technology. Investigations of binary Fe-Cr alloys will contribute to the understanding of the behaviour of more complex alloys. The presented SANS results are focused on a Fe-9at%Cr alloy neutron-irradiated up to neutron doses of 0.6 and 1.5 dpa. We have observed a pronounced increase of scattering cross-sections for both magnetic and nuclear scattering. The A-ratio is about 2.8 for both irradiation conditions. This value is far from a value of 1.45 corresponding to nanovoids as scattering objects. This indicates that the irradiation-induced clusters are different from pure nanovoids and must contain Cr-atoms with the same or very similar average composition for both irradiation conditions. The composition of the clusters will be discussed in more detail. The size distributions of irradiation-induced defects have been calculated. The volume fraction of clusters increases slightly with neutron dose.

Temperature Dependence of Dislocation Loop Morphologies under Heavy-Ion Irradiation in Ultra High Purity Fe and Fe-Cr Alloys: *Zhongwen Yao*[1]; Mercedes Hernandez-Mayoral[1]; Mike Jenkins[1]; Mark Kirk[1]; [1]University of Oxford

Thin foils of pure Fe and FeCr alloys were irradiated with 150 keV Fe+ ions at temperatures 30-500°C in the Argonne IVEM-Tandem Facility. Dynamic observations followed the evolution of damage over doses 0-13 dpa. At low doses damage took the form of isolated dislocation loops with Burgers vectors b = <100> and ½ <111>. At temperatures ≥300°C and doses ≥1 dpa, complex microstructures developed in thicker regions of the foils, involving cooperative interaction and coalescence of smaller loops. In UHP Fe irradiated at 300°C, the damage took the form of large interstitial finger-shaped loops with b =½<111>. At 500°C, square-shaped interstitial loops with b = <100> nucleated and grew to large sizes. Damage structures in FeCr were similar but on a finer scale. Small voids were found at both 300°C and 500°C. In this paper we will describe experiments to investigate the transition in loop types.

The Change in the Hardness of LCAC, TZM, and ODS Molybdenum in the Post-Irradiated and Annealed Conditions: *Brian Cockeram*[1]; Richard Smith[1]; Lance Snead[2]; [1]Bechtel Bettis Inc; [2]Oak Ridge National Laboratory

Hardness measurements were performed on wrought LCAC, TZM, and ODS molybdenum in the post-irradiated and annealed conditions to determine the kinetics for defect mobility. Irradiations in HFIR at 270C to 600C were shown to result in relatively large increases in hardness (54% to 100%), while small changes in hardness (-11% to 18%) were observed for irradiations at 870C to 1100C. The kinetics for recovery for the alloys irradiated at 270C to 605C were determined by performing isochronous anneals. Recovery is observed to begin at about 600C and was completed at 1100C. The activation energy for recovery was determined to be about 4 eV for LCAC and ODS molybdenum, which is comparable to values reported in literature for molybdenum self-diffusion. TZM exhibits much slower recovery kinetics.

The Structure and Composition of Y-Ti-O Nanoclusters in Nanostructured Ferritic Alloys: *Barbara Wang*[1]; Hyon-Jee Lee[1]; Brian Wirth[1]; [1]University of California, Berkeley

Nanostructured ferritic alloys are distinguished by a high density of Y-Ti-O nanoclusters (NCs). The presence of these NCs stabilizes ultrafine grain sizes and subgrain dislocation structures, resulting in a material with remarkable tensile strength, creep strength, and irradiation resistance. In a study by Alinger et al., lattice Monte Carlo methods were used to determine the NC composition and structure, within a model of pair interactions on a rigid body centered cubic lattice. The pair interaction potentials were parameterized using ab-initio methods, while making several simplifying assumptions. In this work, a larger set of ab-initio calculations is utilized to determine the adequacy of the assumptions and to refine the interaction potentials. The results are used to define full potential energy functions for off-lattice relaxation Monte Carlo simulations of NC formation. The obtained NC composition and structure are compared with the Alinger et al. results, as well as with available experimental data.

Near-Net Shape Titanium Components: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS: Titanium Committee

Program Organizers: Rodney Boyer, Boeing Company; James Cotton, Boeing Co

Sunday, 6:00-8:00 PM Room: 2010
February 15, 2009 Location: Moscone West Convention Center

Comparison of Texture Evolution during the Extrusion of Zr-2.5Nb and CP Ti Tube: *Konstantinos Alevizos*[1]; Richard Dashwood[2]; Martin Jackson[3]; David Dye[1]; [1]Imperial College; [2]University of Warwick; [3]University of Sheffield

The texture and microstructure of Zr-2.5Nb pressure tubes used in structural nuclear applications is critical to the component life attained, and to the avoidance of costly re-tubing operations. Here, we compare the evolution of texture and microstructure obtained by tube extrusion in CP Ti and Zr-2.5Nb for both sub- and super-transus extrusion conditions. The effect of low temperature aging and heat treatment high in the alpha+beta field on the textures and microstructures obtained in the extrusions are also considered. The strain path and temperature evolution of the billet during extrusion have been modelled using constitutive data generated from isothermal compression and friction test samples. The model results are used to inform the discussion of the observations made using X-ray texture analysis, SEM and TEM.

The Effect of Plastic Strain and Applied Stress on Variant Selection during Transformation of Ti-6Al-4V: *Michael Glavicic*[1]; David Furrer[1]; David Rugg[1]; Lee Semiatin[2]; John Almer[3]; [1]Rolls-Royce Corporation; [2]Air Force Research Laboratory; [3]Argonne National Laboratory

The effect of plastic prestrain and applied stress on variant selection during the cooling-transformation of Ti-6Al-4V was established. For this purpose, the alpha-phase variants developed in tension samples comprising two coarse beta grains which were deformed to a finite strain in the beta phase field and then cooled with or without an applied load were determined by an electron backscatter diffraction. It was found that variant selection within a given grain and at the grain boundaries themselves is strongly affected by both prestrain and the applied load during cooling from the beta phase field.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee

Program Organizers: Sung Kang, IBM Corp; Iver Anderson, Iowa State University; Srinivas Chada, Medtronic; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Albert Wu, National Central University

Sunday, 6:00-8:00 PM Room: 2020
February 15, 2009 Location: Moscone West Convention Center

Session Chairs: Fu Guo, Beijing University of Technology; Iver Anderson, Iowa State University

The Influence of Ni Content on Cracking in IMC Reaction Layers between Sn-Cu-Ni Solders and Cu Substrates: *Kazuhiro Nogita*[1]; Christopher Gourlay[2]; Tetsuro Nishimura[3]; Shoichi Suenaga[3]; Stuart McDonald[1]; Hideaki Tsukamoto[1]; [1]University of Queensland; [2]Imperial College London; [3]Nihon Superior Co. Ltd.

IMC reaction layers between ball grid array (BGA) solders and Cu substrates has been investigated in Sn-Cu and Sn-Cu-Ni solders. The Ni content in the (Cu,Ni)6Sn5 reaction layer depends on the bulk solder alloy composition. In this paper, it is shown that the level of cracking in the (Cu,Ni)6Sn5 reaction layer varies with Ni content in the IMC. To explore this finding, DSC, SEM-EDS and XRD are used to study the influence of Ni content on phase transformations in (Cu,Ni)6Sn5. The findings are compared with a recent TEM study on the stability of hexagonal (Cu,Ni)6Sn5 in Sn-Cu-Ni alloys[1]. [1] K. Nogita and T. Nishimura, Scripta Materialia 59 (2008) 191.

Effect of Ionization Characteristics on Electrochemical Migration Lifetimes of Various Solder Alloys in NaCl and Na_2SO_4 Solutions: *Young-Bae Park*[1]; Ja-Young Jung[1]; Shin-Bok Lee[2]; Young-Sik Kim[1]; Young-Chang Joo[2]; [1]Andong National University; [2]Seoul National Univ

Higher density integration and adoption of new materials in advanced electronic package systems result in severe electrochemical reliability issues in microelectronic packaging due to higher electric field under high temperature and humidity conditions. Under these harsh conditions, metal interconnects respond to applied voltages by electrochemical ionization and conductive filament formation, which leads to short-circuit failure of the electronic package. In this work, in-situ water drop test and evaluation of corrosion characteristics for various solder alloys such as Sn, SnPb, SnAgCu alloys in NaCl and Na_2SO_4 solutions were carried out to understand the fundamental electrochemical migration characteristics and to correlate each other. The electrochemical migration characteristics of each solder in NaCl and Na_2SO_4 solutions will be discussed in detail.

Effects of Pad Open Size and Bump Height on the Electromigration Lifetimes and Failure Mechanism of Flip Chip Sn-3.5Ag Bump: *Young-Bae Park*[1]; Jang-Hee Lee[1]; Seung-Taek Yang[2]; Min-Suk Suh[2]; Qwan-Ho Chung[2]; Kwang-Yoo Byun[2]; [1]Andong National University; [2]Hynix Semiconductor Inc.

Electromigration of the flip chip Pb-free solder bump has recently been one of the most important reliability issues in flip chip packages. Effect of ever-decreasing size of solder bump on electromigration performance is very important not only in fundamental aspect but also technological point of view. In this work, the effects of pad open size and bump height on the electromigration lifetimes and failure mechanism of flip chip Sn-3.5Ag bump with Cu under bump metallurgy were systematically investigated by using both ex-situ electromigration test in an oven and in-situ electromigration test in a scanning electron microscope chamber, respectively. And, the line length effect on electromigration characteristics of Pb-free solder using the multi line-patterned specimens were correlated with the results from electromigration of Pb-free solder bump in flip chip package. Possible mechanism of size dependence of electromigration resistance will be discussed in detail.

Environmental Effect on Interfacial Debonding Energy of Inkjet-Printed Ag Interconnects on Polyimide Substrate for Flexible Electronics: *Young-Bae Park*[1]; Sung Cheol Park[1]; Hyun Chul Jung[2]; Jaewoo Joung[2]; [1]Andong National University; [2]Samsung Electro-mechanics

Inkjet printing technology is widely uses as a direct writing technique for fabrication of the electronic circuits on the flexible substrates. Even though there are many advantages including low cost, low temperature process, solution processing, and rapid prototyping, the interfacial adhesion between inkjet-printed Ag film and flexible polymer substrate is known to be very poor, which can be interfacial reliability issues for flexible electronics applications. In this work, the effects of CF_4 plasma treatment conditions of polyimide surface on the interfacial adhesion energy are evaluated from 180° peel test for various temperature/humidity conditions. Extensive surface analysis using AES, XPS, and AFM is performed to understand the fundamental adhesion enhancement mechanism due to CF_4 plasma treatment and degradation mechanism due to 85°C/85%R.H. temperature/humidity conditions, respectively.

Interfacial Adhesion and Reliability of Electroless-Plated Ni Film on Flexible Polyimide Substrate: *Kyoung-Jin Min*[1]; Sung-Cheol Park[1]; Kyu Hwan Lee[2]; Yongsoo Jeong[2]; Young-Bae Park[1]; [1]Andong National University; [2]Korea Institute of Materials Science

Flexible electronic substrates are increasingly adopted as future electronics packaging technology due to its merits of flexibility and performance. The applications of this technology needs high density integration process by using metal electrode on polyimide. Although these FPCB are widely used in flexible electronics application, poor interfacial adhesion between conductor metal and polymer substrate such as polyimide lead to bottleneck of its wide application due to lack of long-term interfacial reliability. In this study, we investigated the effects of the chemical pretreatment conditions of polyimide surface with KOH and Ethylenediamine combinations and also the post-baking treatment condition on the interfacial adhesion energy of electroless-plated Ni to polyimide systems in order to understand the interfacial bonding mechanism and enhance the adhesion. Extensive interface analyses using FE-SEM, AFM and XPS were performed to understand the fundamental interfacial bonding mechanism and to find the optimum conditions of wet treatment and post-baking, respectively.

Aging-Informed Constitutive Models for Steady State Creep-Plasticity in Sn3.8Ag0.7Cu Solder Alloy: *Kaushik Mysore*[1]; Dennis Chan[1]; Sri Chaitra Chavali[1]; Ganesh Subbarayan[1]; Indranath Dutta[1]; Vikas Gupta[1]; Darvin Edwards[1]; [1]Purdue University

Aging influences on microstructure and behavior of Sn3.8Ag0.7Cu solder alloy are shown to be significant. A modified viscoplastic Anand model and a simpler power law creep-plasticity model are developed to predict aging effects on behavior. Aging effects on primary and secondary creep under a range of applied loads and test temperatures are addressed. Procedures to compare alloys in terms of aging effects are discussed. Steady state creep strains, monotonic plastic strains and unified creep-plasticity theory are also discussed. Aging temperatures of -100 C, 250 C, 750 C and 1250 C, and aging times of 15, 30, 60 and 90 days (at each aging temperature) were selected as different levels of factors in a statistically designed experiment to study aging effects. Test specimens were selected with due pre-test considerations to joint-geometry, associated stress heterogeneity and joint-microstructures.

Inhomogeneous Consumption of Electroless Ni-P Layer at the Solder Joint Formed with Sn3.5Ag0.7Cu Alloy: *Yong Jun Oh*[1]; Sung Yong Oh[1]; [1]Hanbat University

The consumption behaviors of electroless Ni-P under-bump metallization (UBM) at the solder joint of Ni-P and Sn3.5Ag0.7Cu solder (SAC) alloy after reflowing and solid-state annealing are compared with those of electrolytic Ni UBM. Under the same reflow and annealing conditions, electroless Ni UBM is consumed more slowly than electrolytic Ni UBM, indicating that phosphorus (P) in Ni-P layer effectively retards the growth of intermetallic compounds (IMCs). However, the electroless Ni-P/IMC interface is highly irregular and serrated, and these irregularities become more severe with the aging time. Two reasons for this irregularity are proposed: 1) Ni-P UBM causes marked variations in the sizes and morphologies of the interfacial IMCs, and this results in different UBM consumption rates at different locations; 2) the coalesced Kirkendall voids in the Ni3P layer, particularly those formed during the annealing, facilitate the diffusion of Ni and Sn, thereby increasing the localized consumption of the Ni-P layer.

Creep Behavior of Sn-Zn Alloys: *Indrajit Charit*[1]; Srikant Gollapudi[2]; Triratna Shrestha; Korukonda Murty[2]; [1]University of Idaho; [2]North Carolina State University

Creep properties of three Sn-Zn solder materials (Sn-9Zn, Sn-20Zn and Sn-25Zn) were investigated using the impression creep technique in a temperature range of 323-413 K and at various stresses. Microstructures of these materials were examined using both optical and scanning electron microscopy. The Sn-Zn alloys show a typical M-type creep behavior implying the operation of a dislocation-climb controlled mechanism (n = 4-6). However, the activation energies for creep deformation in these alloys were calculated to be ~ 50-70 kJ/mol, which is less than the lattice self-diffusion activation energies of Sn or Zn. The creep results from the present study are compared with those of various Sn-Zn alloys and other solder alloys from various literature sources.

Failure Mode Characterization of Pb-Free Solder Joint with High-Speed Shear Test: *Sang-Su Ha*[1]; Jin-Kyu Jang[1]; Sang-Ok Ha[1]; Jong-Woong Kim[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University

During the solder process, the components, distribution and thickness of intermetallic compound (IMC) between solder and pad can strongly affect the strength of solder joints, which will eventually determine the brittles of joints. In this paper, failure behaviors of solder joints under various aging time of high-speed shear test were investigated with an experimental and nonlinear 3-dimensional finite element modeling work. A representative Pb-free solder alloy, Sn-3.0Ag-0.5Cu, was employed in this study. The shear force further increased with shear speed mainly due to the high strain-rate sensitivity of the solder alloy. Brittle interfacial fractures can be more easily achieved by high-speed shear test, especially in higher shear speed. This was discussed with the relationship between the strain-rate and work-hardening effect and resulting stress concentration at interfacial regions.

Solderability of Nanostructured Sn-Ag Solders: *Guangwen Zhou*[1]; Ying Sun[1]; Timothy Singler[1]; [1]State University of New York, Binghamton

The drive for green electronics has prompted a strong interest in the development of Pb-free nanosolders. However, the properties of even the most promising Pb-free nanosolders still do not meet the target goals, primarily due to the lack of understanding the fundamental processes governing the wetting reaction. In an effort to understand the solderability of Pb-free nanosolders, we have selected the reaction of Sn-Ag nanoparticles with Cu substrates as a model system to investigate the wetting reaction over different length and mass scales by employing a unique combination of in-situ microscopy techniques and multiscale modeling. An in-situ optical microscope is used to study the reaction of large quantities of nanoparticles at the micron scale, while the in-situ TEM provides visualization of the wetting reaction of individual nanoparticles and phase transformations of intermetallic compounds at the nanometer. The results obtained from the in-situ microscopy observations will be elucidated by multiscale simulations.

Microstructural Evolution of Interfacial Reaction Layers in Sn-3.5Ag-0.7Cu/ Electroless Ni–P Solder Joints: *Han-Byul Kang*[1]; Jee-Hwan Bae[1]; Jae-Wook Lee[1]; Min-Ho Park[1]; Cheol-Woong Yang[1]; [1]Sung Kyun Kwan Univ

We investigaed the interfacial reaction between Sn-3.5Ag-0.7Cu solders and an electroless nickel-immersion gold plated Cu substrate using analytical transmission electron microscopy (AEM). Cross sectional AEM samples were prepared by focused ion beam. Nano beam electron diffraction (NBED) technique is used for phase identification of interfacial reaction layers. The composition redistribution was analyzed by scanning transmission electron microscopy (STEM)/ energy dispersive X-ray spectrometry (EDS). The $(Ni,Cu)_3Sn_4$ IMC was formed at the Sn-3.5Ag-0.7Cu/Ni (P) interface. The Ni_2SnP ternary was formed between β-Sn and P-rich Ni layer and the dominant phase of P-rich Ni layer to be $Ni_{12}P_5$ and Ni_3P phase with a small amount of Ni_2P phase. In particular, Ni_2P phase was formed below the IMC. The Sn was found in the P-rich Ni layer, which indicates that Sn can diffuse through the P-rich Ni layer and react with the Ni-P compound to form a Ni2SnP ternary phase.

Effect of Sn Grain Orientation on the Electromigration Behavior of SnCu0.7 Solder: *Han-wen Lin*[1]; Chih Chen[1]; [1]NCTU

The requirement for smaller electrical devices but with much greater performance leads to a boost in current density and damage caused by electromigration. In this study, we use a sandwich structure of Cu|SnCu0.7|Cu of about 300μm wide to avoid the current crowding effect. Electron backscatter diffraction (EBSD) is used to acquire a grain orientation map before and after current stressing.

Due to the non-equiaxial crystal structure (body center tetragonal) of Sn, the electromigration behavior on grains with different orientation may appear different. It is expected that the electromigration damage prefers to occur along C axis, so the shrinkage of grain is more obvious in some particular grains. In this study, a lot of Sn grains with different orientations can be prepared. The effect of grain orientation on the electromigration behavior will be discussed in the conference.

Effect of Al-Trace Dimension on the Joule Heating in Flip-Chip Solder Joints under Current Stressing: *Hsiang-Yao Hsiao*[1]; Chih Chen[2]; [1]National Chiao Tung University ; [2]National Chiao Tung University

Joule heating effect may occur during accelerated electromigration tests in solder joints. It induces a temperature increase inside the solder joints, resulting in a much higher temperature than the ambient temperature and it may trigger thermomigration in solder. This work uses infrared microscopy to measure temperature distributions on the solder joints with various Al-trace dimensions. In order to investigate the thermal characteristics in solder bumps, lead-free solder joints with typical dimensions were adopted. Since the major heat source for the solder joints is the Al trace, we investigated the influence of Al-trace dimension on the temperature distribution in solder joints. Solder joints with Al-traces of 40 µm and 100 µm wide were examined. In addition, Joule heating effect in solder joints with 2550-µm-long, 1700-µm-long, and 850-µm-long Al traces were investigate. It is found that the dimension of the Al traces has significant influence on the temperature in solder bumps.

In-situ Observation of Electromigration Failure in Pb-Free SnAg2.6 Solder Joints with 2-µm Thick Nickel under-Bump-Metallization: *Wei-an Tsao*[1]; Chih Chen[1]; [1]NCTU

The failure due to electromigration is observed in Pb-free solder joints by in-situ observation. In this study, SnAg2.6 solder with 2-µm thick nickel under-bump-metallization (UBM) was stressed under 150°C with 0.8 Ampere, and the increase in resistance is monitored by using Kelvin probes. It is found that the different test condition may cause different failure mode and failure time of the solder joints. Furthermore, we can successfully obtain the details of microstructures changes during various stages of current stressing and the failure mode by in-situ observation. The detailed results will be presented in the conference.

Mitigation of Tin Whiskers Growth by Micro-Alloying of Tin Plating: *Aleksandra Dimitrovska*[1]; Radovan Kovacevic[1]; Dechao Lin[1]; [1]SMU

Sn serves as a key industrial material to make coatings on various components in the electronic industry. However, it was noticed that tin was prone to the development of whiskers even at room temperatures which is the leading cause for many electronic damages reported in the last several decades. One of the solutions developed was by applying Sn-Pb alloy as electroplated coating which has proven to have positive effect on the mitigation of Sn-whiskers. Unfortunately, with the European law imposed on July of 2006 all lead content is no longer permitted for use. In this study alloys such as Sn-Bi, Sn-Sb, Sn-Zn and Sn-Cu were electroplated onto the brass substrate by utilizing pulse plating technique, in order to achieve properties similar to the Sn-Pb alloy. Experimental results have indicated that this technique of the selected Sn-alloys can alter the microstructure of pure tin and could mitigate the growth of Sn-whiskers.

Size and Substrate Effects on the Mechanical Properties of Solder Joints: *Jenn-Ming Song*[1]; Guo-Wei Lee[1]; Cheng-Yi Liu[2]; [1]National Dong Hwa University; [2]National Central Univ

Due to the tremendous developments in IC and electronic packaging technologies, the size miniaturization of microelectronic interconnections has become an important trend. Since a reduction in joint size may give rise to complex mechanical and metallurgical responses, this study aimed to clarify the effect of gap size on microstructure and mechanical properties of pure Sn and Sn-Ag-Cu joints with Cu substrate. In addition, the influence of cross-sectional interaction of different substrates (Cu and Ni) on opposite side of joints was also examined. The results show that the shear strength increased and shear modulus descended with a shrunken joint gap size because of the constraining effect due to size miniaturization. Fracture mode was also affected by a change in joint thickness. For Cu/solder/Ni joints, the shear strength and it size dependence were weaker than those of Cu/solder/Cu joints. This could be ascribed to the asymmetrical structural feature.

Structure and Properties of Grain Boundary in Cu Affected by Nano-Twins: *Di Xu*[1]; Luhua Xu[1]; Vinay Sriram[1]; Jenn-Ming Yang[1]; K. N. Tu[1]; [1]UCLA

Cu with a high density of nanotwins have been shown to possess excellent mechanical strength and good electrical conductivity. This may lead to promising applications as interconnect material in Si microelectronic technology. The effect of nanotwin boundary on grain boundary structure and properties of Cu has been investigated. Nanoindentation study showed that hardness of copper near the grain boundaries which intersect with nanotwin boundaries is much higher than those of normal grain boundaries and bulk copper. Thermal stability study indicated that nanotwinned Cu thin films maintained good uniformity of grain size (absent of abnormal grain growth) while nanocrystalline Cu thin films with few twins underwent abnormal grain growth. We propose that the triple point where a twin intersects a grain boundary may play the key role in changing the structure and properties of the grain boundaries in Cu having a high density of nanotwins.

Effect of Design of Wiring Traces on the Current Crowding Effect in Flip-Chip Solder Joints: *Chien-Chih Kuo*[1]; Chih Chen[1]; [1]NCTU

The effect of different designs of Cu wiring traces on electromigration of flip-chip solder joints was investigated in this study using 3D finite element simulation. When flip-chip solder joints is stressed by currents, the currents flow along the path with the lowest resistance instead of distributing uniformly in the solder. The current crowding effect leads to electromigration damage and void formation in solder bumps. So how to relieve current crowding become an important issue. Four different designs of Cu wiring traces were adopted: slab trace, slit trace, 2-4 trace, 2-2-2 trace models. For the same stressing condition at 1A , the four different samples were simulated by ANSYS software. Take the slab trace sample as standard , we could find the other three models possess lower current density and better uniformity of current in solder bumps. Especially the 2-2-2 model has the lowest current crowding effect in solder joints. Thus, by changing the design of the wiring traces, we can get a uniform distribution of current in solder joints and relieve the current crowding effect.

In-Situ Observation of Electromigration Eutectic SnPb Flip-Chip Solder Joints under Current Stressing: *Dai-lin Wu*[1]; Chih Chen[1]; [1]NCTU

The eutectic SnPb flip-chip solder joints with under bump metallization(UBM) of Ti 1k / Cu 5k / Ni 2 µm has been used for this in-situ observation. The temperature increase during electromigration test of the eutectic SnPb was investigated by Infrared (IR) Microcopy, to make sure that the Joule heating effect is taken into consideration. The experiment was conducted under 130°C and 0.8A. The structure and chemical composition were determined by Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS). Kelvin probes are employed to measure the total resistance of a pair of bumps and the connecting Al trace. Detailed results will be presented in the conference.

Interfacial Reaction between the Electroless Nickel Immersion Gold Substrate and Sn-Based Solders: Ruihong Zhang[1]; Ran Zhao[1]; *Fu Guo*[1]; [1]Beijing University of Technology

The electroless nickel immersion gold (ENIG) surface finish is widely used in electronic packaging. The ENIG induced Au embrittlement has been investigated in SnPb/ENIG/Cu solder joint since several years ago. However, in Sn-based lead-free solder joint, discrepancies still exist about the influence of Au finish on the reliability of the solder joint. This study investigated the effects of ENIG surface finish on the interfacial reaction and thus the mechanical property of Sn-based solder joints. Experimentally, two types of ENIG with different thickness of Au layer were fabricated. The results indicated that the Au layer dissolved into the solder matrix readily in the soldering stage, and then affected the shear strength of the solder joint significantly. The Au migration occurred in the solder joint during service. The existence of Cu in the interfacial intermetallic compounds (IMCs) promoted the Au atoms to diffuse to the interface and made the solder joint immune to embrittlement.

Structural Analysis of Composite SnPb Whiskers Grown by Current Stressing: Cheng-Chang Wei[1]; *Tao-Chi Liu*[2]; Chih Chen[1]; [1]National Chiao Tung University; [2]National Chiao Tung University; Integrated Service Technology

The growth of whiskers from lead-free solder is the key concern for electronic components. It is well known that adding Pb into solder can mitigate Sn growth effectively due to stress relief. In this study, an accelerated test on whisker growth is achieved by current stressing in thin solder stripes. Composite SnPb whiskers can be grown at a current density above 5×10^4A\cm^{2}at 100°C. In some case, the diameter of a composite whisker is less then 600 nm; the length

could extend to several tens of microns. The microstructure of the composite whiskers is examined by a dual-beam focus ion beam and TEM technologies. EDS mapping is employed to examine the composition of the whiskers. The orientation relationship between the Sn and Pb whiskers were examined. The parent grains in which the whiskers grow from are also examined by TEM.

A Phase Field Model for the Study of Microstructure Evolution in Lead-Free Solder Joints: *An Serbruyns*[1]; Nele Moelans[1]; Bart Blanpain[1]; Patrick Wollants[1]; [1]KU Leuven

The presented phase field model is used to study the behavior of intermetallic compound (IMC) layers during soldering and subsequent thermal cycling in Sn-Cu systems. Thick IMCs are detrimental for the mechanical properties of the solder joint due to their brittle nature and therefore their growth has to be controlled. The microstructural evolution of IMCs is governed by diffusion, precipitation, grain growth and electromigration. All these concurrent processes are taken into account in the proposed phase field model. By varying diffusion coefficients, initial compositions, geometries, …, it is possible to study their effect on IMC growth. By using advanced modeling techniques, this can be done faster and on a larger scale than what is possible with experiments.

Accelerated Growth of Tin Whiskers from Evaporated Film: *Jing Cheng*[1]; Joe Subjeck[1]; Heather Howard[1]; Paul T. Vianco[2]; James C.M. Li[1]; [1]University of Rochester; [2]Sandia National Laboratories

An acceleration method was designed to apply compressive stresses on a 1 micron thick tin film evaporated on silicon wafer with an underlayer of 20nm thick chromium. By this method, the compressive stresses are known without the complication of intermetallic compounds when tin is deposited on copper. The loaded sample was annealed at 180°C in a vacuum oven for a week. SEM observations showed a number of unexpected tin depleted areas with whiskers grown in them. The sample was annealed again for another four weeks, after each week some depleted areas seemed to shrink and sometimes disappear but other new ones appeared. However, the whiskers remained intact. Another clamping experiment showed tin whiskers grew from the interface between the substrate and the tin film. Based on these observations, the authors propose an interface fluid flow mechanism for the transport of tin atoms in the formation of depleted areas and the growth of whiskers.

Microstructural and Mechanical Characterization of Solder Joints Fabricated by Explosively Reacting Nanolayers: *Michael Tong*[1]; Jenn-Ming Yang[1]; [1]UCLA

This study focuses on characterizing the microstructure and mechanical properties of solders fabricated by reactive soldering. The microstructure of Al/Ni explosively reacted nanolayers as well as post-joining reactive foil/solder interface is characterized with XRD, SEM, and TEM, which has never been done. Additionally, the mechanical properties of reactively soldered joints have been characterized by single lap shear and nanoindentation. Single lap shear testing is used to determine the effect of thermal aging on the joint shear strength. Furthermore, fracture surface analysis provided additional information regarding the integrity of the reactive foil/solder interface. Nanoindentation is used to clarify the mechanical behavior of individual layers and interfaces across the joints. Linear arrays of indents are used to capture data at or near the reactive foil/solder interface and the composite values of modulus and hardness are discussed.

Silver-Bismuth Alloys as High Temperature Lead-Free Solders: *Anthony Muza*[1]; Mark Cooper[1]; Carol Handwerker[1]; [1]Purdue University

Under pressure from RoHS, microelectronics companies are exploring possible high temperature lead-free solders to replace the 95Pb-5Sn tin-lead alloy used for chip interconnects and hierarchical soldering processes. This study investigated a range of Bi-Ag binary eutectic alloys as suitable high temperature lead-free solders and as partially molten joining materials. Results will be presented describing the wetting of these alloys as a function of alloy composition, temperature, and flux type, their microstructures, and their resulting electrical and mechanical properties. Of greatest importance for use in interconnects is that, as the Ag concentration increases, the Ag dendritic primary phase provides not only a more electrically conductive path than high Bi alloys, but also improved mechanical properties.

Constitutive Behavior of Mixed PbSn/SnAgCu Solder: *Jonathon Tucker*[1]; Carol Handwerker[1]; Ganesh Subbarayan[1]; [1]Purdue University

At this time, in reworking legacy components, there is a need to understand the reliability of Sn-Pb components reworked with Pb-free solder. Rate-dependent viscoplastic constitutive models for Pb-mixed solders are developed through extensive testing in this study. Three alloys of 1, 5 and 20 weight percent Pb were selected so as to represent reasonable ranges of Pb contamination expected from different Sn-Pb components reworked with Sn3.0Ag0.5Cu. Monotonic and creep tests were performed on specially designed assemblies at temperatures of 25°C, 75°C, and 125°C using a double lap shear test setup that ensures a nearly homogeneous state of plastic strain at the joint interface. The experimental data were used to fit for the parameters of the rate-dependent constitutive models.

Synergies of Computational and Experimental Materials Science: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee

Program Organizers: Katsuyo Thornton, University of Michigan; Henning Poulsen, Risoe National Laboratory; Mei Li, Ford Motor Co

Sunday, 6:00-8:00 PM Room: 3003
February 15, 2009 Location: Moscone West Convention Center

Calculation of Forming Limit Diagrams Using a Revised Hill's Nonquadratic Yield Criterion: *Hadi Noori*[1]; Reza Mahmudi[1]; [1]University of Tehran

Forming Limit Diagram (FLD) is a useful tool in analysing sheet metal formability. It has had a significant impact in both academia and industry on the determination of the maximum deformation that a material can withstand during a sheet metal stamping process. Jones and Gillis (JG) have proposed an analysis based on the idealization of sheet metal deformation into three phases: (1) homogeneous deformation up to maximum load, (2) deformation localization under constant load, and (3) local necking with a precipitous drop in load. In the present work, a revised Hill's nonquadratic flow law for sheets having planar isotropy with a high order of M=20 has been used in conjunction with the JG analysis. Calculated FLDs are successfully comparable with experimentally determined results for interstitial free steel sheet. Using the revised flow law eventuates in the FLD levels independent of the plastic anisotropy parameter, similar to what happens in reality.

Cellular Automaton Modelling of Grain Growth during Annealing of Alloy Fe-19Cr-8Ni: *Mirza Candic*[1]; Gerald Winter[1]; Baohui Tian[2]; Christof Sommitsch[1]; [1]University of Leoben; [2]Bohler Edelstahl GmbH & Co KG

Simulation and control of grain size during heat treatments are essential for tailoring the properties of polycrystalline materials. In the present work, for the description of grain coarsening process, a deterministic two-dimensional cellular automaton modell has been devoloped. The model simulates the grain coarsening process, from the point of variable initial microstructures and annealing temperatures with the computational time. For the validation of the presented model, the annealing of the austenitic stainless steel Fe-19Cr-8Ni with variable initial microstructures and annealing conditions was carried out. The results of the simulation have been validated by experimental results.

CFD Characterization of an Electrochemical Cell with a Rotating Electrode: Jesus Gonzalez-Trejo[1]; Rosalba Orduña-Martinez[1]; Manuel Palomar-Pardave[1]; Luis Hoyos-Reyes[1]; Mario Romero-Romo[1]; Miriam Aguilar-Sanchez[1]; *Cesar Real-Ramirez*[1]; [1]Universidad Autonoma Metropolitana - Azcapotzalco

Electrochemical cells with rotating electrodes (RE) are amply used to study the kinetics of charge transfer reactions. For this purpose, it is common to estimate the Reynolds number using geometrical parameters of the electrode, namely its diameter, and the applied angular velocity. Recent works have shown that many systems with the same Reynolds number but different cell boundary conditions, namely different electrode depth immersion or cell volume, are associated to totally different flow patterns. The main goal of this work is to obtain a hydrodynamic characterization of an electrochemical cell with RE using two different turbulence models (κ-ε and LES) to obtain with the former a general description of the system and with the latter further specificities. Moreover, we will compare the resulting turbulences models with physical simulations.

Deformation Mode Characterization and FEM Simulation of α-Titanium Deformed in a Bending: *Leyun Wang*[1]; Yiyi Yang[1]; Martin Crimp[1]; Philip Eisenlohr[2]; Darren Mason[3]; Thomas Bieler[1]; [1]Michigan State University; [2]Max-Planck-Institut für Eisenforschung; [3]Albion College

Deformation of polycrystalline α-titanium was investigated using a combination of electron microscopy characterization and crystal plasticity FEM (CPFEM) simulation. Commercially pure α-titanium samples were deformed by four-point bending. Microstructural patches displaying dislocation slip bands and deformation twinning were characterized using scanning electron microscopy (SEM). Grain orientations were determined using electron back-scattered diffraction (EBSD) before and after deformation. Based on this orientation data, activated slip and twinning modes were identified in the patches using trace and misorientation analysis. Using digital image correlation (DIC) analysis to compare images of the patches in the undeformed and deformed states, heterogeneous local strain distributions were determined. To assess the ability of the computational model to describe microscale deformation processes, the experimental results were directly compared with CPFEM simulations carried out on FEM meshes based on the orientation maps of the undeformed microstructural patches. This work was supported by NSF(DMR-0710570) and DFG (EL 681/2-1).

Dynamic Characterization of Granular Materials: *Gregg Fenton*[1]; Glenn Daehn[2]; Tracy Vogler[3]; Dennis Grady[1]; Anupam Vivek[2]; Yuan Zhang[2]; Geoff Taber[2]; [1]Applied Research Associates Inc; [2]Ohio State University; [3]Sandia National Laboratories

The dynamic behavior of granular materials such as granular silica (sand) and other ceramics has importance to a variety of engineering applications. Structural Seismic coupling, planetary science, earth penetration mechanics, and the performance of ceramic armors are some of application areas. Although the behavior of sand has been studied extensively for several decades, its dynamic behavior remains poorly understood. High-quality experimental data is needed for computational verification of dynamic events involving sand along with the need to improve our general understanding of granular material physics. This presentation will describe a series of experiments used to measure the dynamic behavior of sand in the partially compacted state as well as show how advanced instrumentation techniques, particularly the Photonic Doppler Velocimetry (PDV) can be used to obtain model parameters for a multi-component Equation of State (EOS) of dry sand. The EOS parameters are then available for use in computational simulations.

Femtosecond Laser Aided Tomography Applied to Titanium - Modified 4330 Steel: *McLean Echlin*[1]; Tresa Pollock[1]; [1]University of Michigan

Femtosecond laser aided serial sectioning is a novel tomographic technique that has been developed at the University of Michigan over the past 3 years. This system utilizes a femtosecond laser to machine a sample surface, capture successive images and create a 3D reconstruction. The technique has been applied to capture 3D data for a variety of different metallic material systems. This talk will primarily focus on the acquisition, image processing, and reconstruction of micron-scale particles in a 4330 steel. Of particular interest are titanium nitrides and 3D reconstructions of the nitrides and their surrounding microvoids which will be used as direct inputs for modeling the shearing process including shear banding and shear localization in ballistic armor applications of this material. To date, this reconstruction has allowed us to verify local and global spatial distributions of nitrides within the sampled material to resolutions of 1-10 microns within near cubic millimeter volumes.

Mechanical Behavior and Texture Evolution of AZ31 Magnesium Alloy during Uniaxial Compression: *Shiyao Huang*[1]; Mei Li[2]; John Allison[2]; Shaorui Zhang[1]; Yinghong Peng[1]; [1]School of Mechanical Engineering, Shanghai Jiao Tong University, Shanghai 200240, P.R. China; [2]Research and Advanced Engineering Laboratory, Ford Motor Company, Dearborn, MI 48121, USA

The mechanical behavior has been studied in compression at temperatures ranging from 200°C~250°C and constant strain rates of 0.01~1/s. Texture analysis was carried out by EBSD. A crystal model was implemented into ABAQUS/Explicit finite element code using VUMAT. The constitutive model was then used to calculate the flow stress curves and grain orientation distributions. Challenges in matching simulation and experimental results are mainly due to the insufficient knowledge of slip and twinning mechanisms, especially the initial critical resolved shear stresses (CRSSs). In this paper, reasonable values of initial CRSSs and hardening parameters under different temperatures were obtained when the simulation results offer best agreement with both experimental stress-strain curves and grain orientation distributions from texture analysis.

Microstructure Based Failure Analysis of Multiphase Steel Using Damage Mechanics Modelling: *Vitoon Uthaisangsuk*[1]; Ulrich Prahl[1]; Wolfgang Bleck[1]; [1]RWTH Aachen University

Multiphase steels have been developed for the automotive industry according to the purpose of the reduction of car body weight. These steels show excellent strength and ductility due to the coexistence of harder and softer phases in their microstructure. To describe the influence of the multiphase microstructures on mechanical properties and complex fracture mechanisms, an approach is presented using representative volume elements (RVE). Real microstructures were considered and cohesive zone was applied for the debonding of martensite-ferrite interfaces. Additionally, RVE simulations in combination with continuum damage mechanics were used to investigate local crack initiation in different sheet forming processes. At the failure moment, local strain distributions between different phases were studied, and correlated with macroscopic deformability results. The influences of material properties of the individual phases and the local states of stress on the failure behaviour were examined. A precise formability prediction based on microstructure for multiphase steels is aimed.

Microstructures and Mechanical Properties of Extruded AZ31 Magnesium Alloy: Weiqin Tang[1]; Xiaohui Fan[1]; Shaorui Zhang[1]; *Dayong Li*[1]; Yinghong Peng[1]; [1]Shanghai Jiaotong University

Hot extrusion is an efficient processing technique to manufacture magnesium alloy components. In this study, AZ31 rods were extruded from casting ingots under various conditions. The mechanical properties of specimens were tested and the fracture surfaces for tensile specimens were analyzed by scanning electron microscopy. The microstructures were observed by using optical microscopy and electron back scattering diffraction. The results show that the specimen after extrusion has finer microstructure and better mechanical properties compared with the casting specimens. Furthermore, the microstructure and mechanical properties differ under different extrusion speeds and temperatures. The fracture surfaces of the casting specimens show a typical brittle fracture character while the extruded specimens exhibit a mixed mode of brittle fracture and toughness fracture. Finally, the dynamic recrystallization during hot extrusion was found to be able to change the crystal orientation, and influence the mechanical properties of the alloys.

Modeling of Residual Stress Fields in Structural Materials: Computational, Mechanical, and Metallurgical Approaches: *Christopher Lammi*[1]; Diana Lados[1]; [1]Worcester Polytechnic Institute

Adjusting/controlling macro residual stress fields while preserving a desired microstructure is often a challenging proposition. A novel mechanical/geometrical technique able to generate controlled residual stress fields was developed. The method is based on a "plug-and-hole" approach and was used to produce set residual stress magnitudes and distributions in rectangular coupons and compact tension specimens. Residual stress fields created through this method were first modeled computationally using ANSYS software and then reproduced using metallurgical means by adjusting the processing conditions. Long fatigue crack growth data for low and high residual stress conditions were generated and compared. High residual stresses were introduced in the testing samples using both the mechanical/"plug-and-hole" method and metallurgical/processing techniques. Effects of residual stress on crack growth thresholds and fracture toughness are presented and discussed. The developed method is proposed to facilitate the acquisition and analysis of fatigue crack growth data in the presence of residual stress.

Schmid Factor Dependence of Deformation Twinning in Magnesium, Uranium, and Zirconium: *Rodney McCabe*[1]; Laurent Capolungo[1]; Peter Marshall[1]; Dhriti Bhattacharyya[1]; Gwenaelle Proust[2]; [1]Los Alamos National Laboratory; [2]University of Sydney

Twinning is an important deformation mode in the hexagonal metals magnesium and zirconium and in orthorhombic uranium, and these three metals represent three potentially different behaviors with regard to twin Schmid factor dependence. For compression perpendicular to a strong (0001) texture, most grains in Zr and Mg have high resolved shear stresses on at least one variant of the {10-12} twin system. This is the dominant deformation mode in Mg while it competes with prismatic slip in Zr. For stress perpendicular to a strong (001) texture in U, grains are distributed approximately evenly from highly positive to

highly negative with regard to Schmid factor for the primary twin mode {130}. We use Electron backscatter diffraction (EBSD) to quantify the area fraction distribution of Schmid factors driving twin formation in textured, polycrystalline samples of Mg, Zr, and U. The results from these experiments are compared to predictions from polycrystal plasticity models.

Study on Conductivity Change of M/SWNTs as Methane Adsorption by Density Functional Theory: *Xuehui Zhan*[1]; Zhongliang Xiao[1]; Yan Shi[1]; Fei Li[1]; Weilian Zheng[1]; [1]Chansha University of Science and Technology

Density functional theory(DFT) is used to calculate adsorption of methane molecules in transition metal dispersed in surfaces of the single walled carbon nano-tubes(M/SWNTs). Structures of methane molecules inside the tubes have been studied by DFT for nano-tubes of diameters 0.954, 2.719 and 4.077nm at ambient temperature 300K. Then the electron structure and density of sates of the different association scheme of M/SWNTs with methane molecules were investigated. The results indicate that there is a conjugative effect between M/SWNTs and methane. Which induces the change of electron distribution on the SWNTs surface. It increased the Cartier density and the electrical conductivity of SWNTs became higher.

Study on Methane Adsorption in Single Walled Carbon Nano-Tube by Density Functional Theory: *Xuehui Zhan*[1]; Zhongliang Xiao[1]; Yan Shi[1]; [1]Chansha University of Science and Technology

Density functional theory(DFT) is used to calculate adsorption of methane molecules in single walled carbon nano-tubes. Adsorption isotherms and structures of methane molecules inside the tubes have been studied by DFT for nano-tubes of diameters 0.954, 2.719 and 4.077nm at ambient temperature 300K. By using the grand potential, the effect of tube diameter on adsorption is discussed. We found that increasing the pore size of several nanometer is preferable for the methane adsorption.

The Application of Bayesian Neural Network Modeling and Critical Experimentation for the Prediction of Tensile and Fracture Toughness Properties in Alpha/Beta Titanium Alloys: *Santhosh Koduri*[1]; Vikas Dixit[1]; Peter Collins[1]; Hamish Fraser[1]; [1]Ohio State Univ

The development of computational tools that permit microstructurally-based predictions for tensile and fracture toughness properties of commercially important titanium alloys is a valuable step towards the accelerated maturation of materials. Modeling tools, such as Neural Network Models based on Bayesian statistics have been used to predict the yield strength, ultimate tensile strength and toughness of Ti-6Al-4V at room temperature. The development of such rules-based models requires the population of extensive databases containing compositional and microstructural information. These databases have been used to train and test Neural Network models. These models have been successfully used to identify the influence of individual microstructural features on the mechanical properties, consequently guiding the efforts towards development of more robust phenomenological models. The influence of the individual microstructural features on tensile and toughness have been subsequently probed using a variety of characterization techniques, including orientation microscopy and transmission electron microscopy. These results will be discussed.

The Complex Formula and Capability Evaluation of Corrosion-Scale Inhibitor for Power Plant Cooling Water System: *Daowu Yang*[1]; Linping Yu[1]; Chengfeng Wang[1]; Zhongliang Xiao[1]; [1]Changsha University of Science and Technology

Orthogonal and corrosion-linked methods were applied in this paper to find a higher efficient corrosion-scale inhibitor, the formula contained HEDP 3.0mg/L AMPS2.0mg/L PESA1.5 mg/L imidazole2.0 mg/L. Limit carbonate and electrochemical methods were used to evaluate the efficiency of scale and corrosion inhibition, which achieved 97.7% and 88.6% respectively. Additionally, the best pH rage was found during these experiments. The inhibitor in our research shows advantages of small agent, good inhibition effect, and environment protection and so on.

2009 Functional and Structural Nanomaterials: Fabrication, Properties, and Applications: Low Dimensional Nanostructures I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Nanomaterials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Gregory Thompson, University of Alabama; Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute; Jiyoung Kim, University of Texas at Dallas; Seong Jin Koh, University of Texas at Arlington; Wonbong Choi, Florida International University; Alexander Howard, Air Force Research Laboratory

Monday AM — Room: 3018
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Seung Kang, Qualcomm Inc; Wonbong Choi, Florida International University

8:30 AM Invited
Nanoprobes and Applications Based on Carbon Nanotubes: *Sungho Jin*[1]; [1]University of California at San Diego

The key component of the AFM is the probe tip, the size and properties of which determine the resolution and reliability of nanoscale imaging. We have fabricated an extremely sharp probe based on carbon nanocone structure directly grown on an AFM cantilever at a predetermined tilt angle by orientation-controlled DC plasma CVD process. The high-aspect-ratio yet thermal-vibration-resistant geometry, together with excellent mechanical strength of the carbon nanocone AFM probes offer many advantages for nanoscale imaging in nanotech and biotech applications. We have also fabricated MFM probes for analysis of magnetic nanostructures, and special AFM probes with extremely low modulus, about three orders of magnitude reduced, for imaging of soft matters, living cells or delicate components. Stresses and Si cantilever beam bending introduced during CVD growth of carbon nanotubes, and techniques to prevent such undesirable geometry changes are also discussed.

9:00 AM
Atomic Scale Models of Multiwall Carbon Nanotube Thermal Properties: *Edmund Webb*[1]; [1]Sandia National Laboratories

Results will be presented for thermal conductivity of multi-wall carbon nanotubes (MWCNTs). While significant research exists on thermal transport properties of single-wall CNTs, comparatively less is known about MWCNTs. This represents a shortcoming for CNT technology since most synthesis methods currently well suited to device manufacturing produce MWCNTs, rather than SWCNTs. Furthermore, superior thermal conductivity observed for SWCNTs is expected to be impacted by phonon modes activated due to wall/wall interactions in a MWCNT. To address this, calculations were performed of the phonon density of states for two SWCNTs: (8,0) and (17,0) zigzag nanotubes. The same calculation was performed for the same two nanotubes threaded together to form a simple MWCNT. By examining differences in the density of states, predictions are made for thermal conductance changes for a MWCNT, compared to SWCNTs comprising it. Predictions are compared to thermal conductivity results from non-equilibrium (constant heat flux) molecular dynamics simulations.

9:15 AM
Carbon Nanotubes for Spacecraft Harness Wiring: *Ryne Raffaelle*[1]; Alex Howard[2]; David Wilt[2]; John Merril[2]; Roberta DiLeo[1]; Christopher Schauerman[1]; Brian Landi[1]; Brian Moses[1]; Jack Alvarenga[1]; [1]Rochester Institute of Technology; [2]Air Force Research Laboratories

We are investigating the use of carbon nanotubes for the development of next generation of electrical harnesses for spacecraft. A prime motivation for this work is the potential for mass savings due to the outstanding current carry capability of these 1-d nanostructured materials coupled with their low mass density. There are also several other potential anciallary benefits for space power systems such as their oustanding tensile strength, flexibility and resiliency, stable thermal characteristics, and radiation tolerance. We will report on recent advances in the processing of carbon nanotube wires and their resulting electrical and mechanical properties. A comparison of these results to current state-of-the-art wiring material will be presented and the potential of these novel nanomaterials for aerospace applications will be discussed.

9:30 AM Invited
Si/Ge Nanowires: Growth, Properties, and Integration: *S. Picraux*[1]; [1]Los Alamos National Laboratory

Semiconducting nanowires are of great interest for the realization of next generation functional electronic, photonic, and sensor devices, and the vapor-liquid-solid (VLS) synthesis technique has the potential for scalable production of such devices. While much progress has been made, improved understanding and control of VLS nanowire growth, properties, and integration onto platforms is now essential for further progress and future applications. Si and Ge, as the best understood electronic materials, provide an excellent test bed to advance this understanding. In this presentation our progress in the fabrication of Si, Ge, and SiGe alloy nanowires will be reviewed. Growth processes for monoatomic, alloy, and heterostructure nanowires will be discussed. Recent electrical and optical property studies will then be reviewed. Finally, directed assembly methods for the integration of nanowires into regular arrays will be presented.

10:00 AM Break

10:15 AM Invited
Functional Nanoparticles: Synthesis and Potential Applications: *Shouheng Sun*[1]; [1]Brown University

Our progress in synthesis of monodisperse transition metal nanoparticles will be presented in this talk. The major challenge in nanoparticle research is the control of particle size, shape, and composition. High temperature solution phase decomposition or reduction of metal precursors in the presence of stabilizing agents has been employed to produce well-dispersed transition metal nanoparticles with less than 5 % variation in diameter. We present our recent syntheses in solid, hollow and dumbbell-like nanoparticles with magnetic and optical functionalities. We demonstrate that monodisperse Pt- or Au-based multifunctional nanoparticles can be used to enhance the catalysis for oxygen reduction in the fuel cell reaction conditions and for CO oxidation. Self-assembled SmCo and FePt nanoparticle arrays can be made for potential information storage applications. With controlled surface functionalization, multifunctional solid and hollow nanoparticles are also promising for medical diagnostics and drug delivery applications.

10:45 AM
Role of Al in Au-Catalyzed Growth of Si Nanowires: *Suneel Kodambaka*[1]; Jerry Tersoff[2]; Cheng-Yen Wen[3]; Eric Stach[3]; Frances Ross[2]; [1]University of California, Los Angeles; [2]IBM T.J. Watson Research Center; [3]Purdue University

Using in situ transmission electron microscopy, we investigate the growth kinetics of Si nanowires using Au-Al alloy and disilane gas. We find that the Si nanowires grow via vapor-liquid-solid (VLS) process with liquid AuAl droplets at temperatures (~520°C) than are relatively higher than that required in the Au/Si system. At lower temperatures, nanowires grow in presence of a solid catalyst. Presence of Al leads to the growth of narrower wires with smoother surfaces. We expect that our results provide new insights into nanowire growth kinetics and enable synthesis of nanowires with tunable morphologies and compositions. *work done at the IBM T.J. Watson Research Center.

11:00 AM
Morphology of Epitaxial Core-Shell Nanostructures: Nanowires and Nanoparticles: *Moneesh Upmanyu*[1]; Hailong Wang[1]; Cristian Ciobanu[1]; [1]Colorado School of Mines

We analyze the morphological stability against azimuthal, axial, and general helical perturbations for epitaxial core-shell nanowires in the growth regimes limited by either surface diffusion or evaporation-condensation surface kinetics. For both regimes, we find that geometric parameters (i.e., core radius and shell thickness) play a central role in determining whether the nanowire remains cylindrical or its shell breaks up into epitaxial islands similar to those observed during Stranski-Krastanow growth in thin epilayers. The combination of small cores and rapid growth of the shell emerge as key ingredients for stable shell growth. Our results provide an explanation for the different core-shell morphologies reported in the Si-Ge system experimentally, and also identify a growth-induced intrinsic mechanism for helical nanowire morphologies. Finally, we will also present the results of a similar analysis for the morphological stability of epitaxial nanoparticles.

11:15 AM
MoO3 Nanobelts: Lithiation and Electrochemistry: *Liqiang Mai*[1]; Bin Hu[1]; Yuan Gao[1]; Yanyuan Qi[1]; Ying Dai[1]; Wen Chen[1]; [1]Wuhan University of Technology

Nanostructured molybdenum oxides have attracted much attention as the promising cathode materials for lithium batteries. MoO3 nanobelts were fabricated through rheological self-assembling and lithiated MoO3 nanobelts were attained by the direct and non-direct autoclave reaction. The influence of lithiation methods on the structure and electrochemistry of the nanobelts were studied by XRD, Raman, FTIR, SEM, TEM, C-V and model battery. The results show that Li+ is inserted in the interlayer of the nanobelts, which takes up some space and leads to the decrease of the capacity, but lithiation can improve transport and diffusion of Li+, resulting in the enhancement of the insertion/extraction reversibility of Li+ ions and the cycling properties of MoO3 nanobelt cathode materials. Interestingly, it has been found that direct and non-direct lithiation can bring about the improvement of electroactivity of MoO3 nanobelts which shows good promise for fundamental study and application of this kind of nanomaterial.

11:30 AM
Zinc Phosphide Nanorods: Fabrication and Transport: *Liqiang Mai*[1]; Yuan Gao[1]; Wen Chen[1]; Bin Hu[1]; Ying Dai[1]; [1]Wuhan University of Technology

Zinc phosphide nanomaterials have attracted much attention as theoretical optimum for solar power conversion and photovoltaic devices. In the present work, Zn3P2 nanorods were fabricated via a mild solvent-thermal method using P, ZnCl2 and NaBH4. The results show that the attained products are tetragonal phased Zn3P2 with certain crystallinity. The nanorod with a diameter of 90 nm and length of more than 1 μm can be observed. Current-voltage (I-V) curve of single Zn3P2 nanorod exhibits a novel hysteresis due to the hole trapping in the single Zn3P2 nanorod.This work was supported by the National Nature Science Foundation of China (50702039, 50672071, 50672072), the Research Fund for the Doctoral Program of Higher Education (20070497012), Program for Changjiang Scholars and Innovative Research Team in University (IRT0547), MOE, China. The authors are pleased to thank the strong support of Professor ZL Wang and Dr RS Yang of Georgia Institute of Technology.

11:45 AM
Influence of the Temperature on the Growth of Core–Shell SiC–SiO2: *Guang Zhu*[1]; [1]Beijing Information Technology Institute

The core-shell SiC-SiO2 nanowires with different diameters have been synthesized through a simple thermal evaporation of the mixture powders of silica and active carbon and condensation on Si substrate without assistance of any metal catalyst. Their microstructures were characterized by scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray diffraction and high-resolution transmission electron microscopy. The results show that the nanowires have a crystalline SiC core and a surrounding amorphous SiO2 layer, and we also have found that the thickness of the SiO2 shell layer could be controlled using different synthesis temperature. The growth of the core–shell SiC–SiO2 nanowires is via a vapour–solid (VS) process, on which a detailed study of both the chemical and structural composition has been carried out.

Biological Materials Science: Implant Biomaterials I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Ryan Roeder, University of Notre Dame; John Nychka, University of Alberta; Paul Calvert, University of Massachusetts Dartmouth; Marc Meyers, University of California

Monday AM — Room: 3014
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Ryan Roeder, University of Notre Dame; Devesh Misra, University of Louisiana

8:30 AM Invited
Structural Aspects of Fracture in Ultra High Molecular Weight Polyethylene and the Implications for Total Joint Replacements: *Lisa Pruitt*[1]; [1]University of California, Berkeley

This work investigates the role of processing and microstructure on the fracture properties of medical grade ultra high molecular weight polyethylene and examines the implications for total joint replacement design. The effects of various process conditions including thermal treatments and crosslinking on the mechanical properties are evaluated. This study examines the role of crystallinity and lamellae size distribution on the deformation, yield and fracture processes of UHMWPE.

9:00 AM Invited
Micromechanisms of Fracture in Resin Based Dental Restorative Composites: *Jamie Kruzic*[1]; Minal Shah[1]; Jack Ferracane[2]; [1]Oregon State University; [2]Oregon Health and Science University

Understanding the micromechanisms of fracture is an important aspect to achieving an optimal balance between the aesthetic and mechanical performance of dental composites. The fracture behavior of two commercial dental composites (micro-hybrid and nanofill) are examined using double notched four point beam bending and pre-cracked compact-tension, C(T), specimens. The influences of water and post-cure heat treatments are also considered. Fracture resistance curve (*R*-curve) experiments revealed that both materials exhibit rising fracture resistance with crack extension, and that the micro-hybrid has superior fracture resistance. The latter is reasoned to give the higher observed flexural strength, as measured by un-notched four point beam bending. Optical and SEM observations reveal an interparticle crack growth mechanism independent of the environmental conditions. Crack deflection and crack bridging are both observed toughening mechanisms, and the nano-agglomerates appear to be less effective at deflecting cracks than solid particles, contributing to the lower toughness for the nanofill composite.

9:30 AM
Indentation Deformation of Crystallized-Bioglass 45S5: *Ding Li*[1]; Fuqian Yang[1]; [1]University of Kentucky

Bioactive glasses have been used as the bone replacement implants for several decades. It generally prefers that the materials remain the glass state without presence of crystallinity for the implant applications. Due to heat treatment and mechanical stresses, it is possible to have crystal phases in bioactive glasses which could alter the bioactivity and biofunctionality of the implanted materials. In this work, a bioglass 45S5 was crystallized at temperature of 650ºC. Microindentation was used to examine localized mechanical deformation of the crystallized-bioglass 45S5 and the effect of crystallization on the dissolution in phosphate buffer solution. Both the indentation hardness and the fracture toughness were determined as a function of the indentation load. The crystallization had a strong effect on the indentation hardness and a less effect on the fracture toughness. The indentation hardness of the crystallized-bioglass 45S5 is less than that of the corresponding material at the glass state.

9:50 AM Invited
Nanoporous Membranes for Medical Applications: Jeffrey Elam[1]; Michael Pellin[1]; Shashishekar Adiga[1]; Larry Curtiss[1]; *Roger Narayan*[2]; [1]Argonne National Laboratory; [2]University of North Carolina - and - North Carolina State University

Membrane biofouling and inflammation play significant roles in instability of implantable biosensors. An ideal biosensor membrane material must both exhibit

limited protein adsorption and promote tissue-biosensor integration. Furthermore, biosensor membranes must exhibit low thickness and high porosity values in order to allow the biosensor to respond to analyte fluctuations. In this study, the use of atomic layer deposition and pulsed laser deposition to deposit biologically functional thin films on anodized aluminum oxide membranes is discussed. The nanoporous membranes were examined using several techniques, including scanning electron microscopy, atomic force microscopy, X-ray photoelectron spectroscopy, Raman spectroscopy, and platelet rich plasma testing.

10:20 AM Break

10:30 AM

Patterned Bioactive Coatings of Ca-P and Bioglass on Ti-6Al-4V Substrate Using a Pulsed Nano Second Marking Laser: *Sameer Paital*[1]; Narendra Dahotre[1]; [1]University of Tennessee

A key tenet of bone tissue engineering is the development of scaffold materials with both physical and chemical cues at the surface so as to stimulate cell attachment, bone regeneration and bone integration at the defect sites. In the present study a marking laser delivering nanosecond pulses was used to synthesize three different patterned coatings, each of Ca-P and Bioglass on Ti-6Al-4V substrate. XRD and EDS analysis were used to study the various kinds of phases and elemental distribution of the coated samples. Osteoblast and fibroblast like cells were cultured on both the samples to study the influence of pattern and chemistry on cell attachment, cell orientation, and cell proliferation. Microstructure and morphological evolutions of the coated samples were studied using a SEM. Phase contrast and confocal laser scanning microscopy were used to study the cell morphology, qualitative cell proliferation and focal contact of the cells with time.

10:50 AM

Favorable Surface Adhesion Response of Electrodeposited Nano-Hydroxyapatite on Ultrafine-Grained (UFG)/Nano-Grained (NG) Austenitic Stainless Steel: *Sachin Mali*[1]; Sashank Nayak[1]; Devesh Misra[1]; Mahesh Somani[2]; Pentti Karjalainen[2]; [1]University of Louisiana; [2]University of Oulu

We describe here the significance of ultrafine-grained (UFG)/nanograined (NG) structures in the electrodeposition of nano-hydroxyapatite and compare with conventional coarse-grained structures. The study demonstrates superior adhesion of electrodeposited nano-hydroxyapatite on UFG/NG structures in relation to coarse-grained austenitic stainless steel examined using nanosratching by a nanoindenter. It is proposed that hydrophilicity (contact angle) and grain structure are the underlying reasons for the difference in nanoscratching or adherent nature of nano-hydroxyapatite coatings on coarse-grained and UFG/NG austenitic stainless steel. An accompanying aspect that emerged from the primary objective is that the amorphous calcium phosphate is a precursor to the formation of nano-hydroxyapatite.

11:10 AM

Compositionally and Structurally Graded Ti-TiO2 Coatings Using Laser Engineered Net Shaping: Vamsi Balla[1]; Paul DeVasConCellos[1]; Weichang Xue[1]; Susmita Bose[1]; *Amit Bandyopadhyay*[1]; [1]Washington State University

Novel structures with functional gradation in composition were successfully made with Ti-TiO2 combination using Laser Engineering Net Shaping (LENS). Addition of fully dense, compositionally graded TiO2 ceramic on porous Ti significantly increased the surface wettability and hardness. The graded structures with varying concentration of TiO2 on top surface found to be non-toxic and biocompatible. Besides the higher wettability, surfaces with TiO2 can enhance their ability to form chemisorbed lubricating films and thus potentially lower the friction coefficient against ultrahigh molecular weight polyethylene liner reducing its wear rate. These unitized structures with open porosity on one side and hard, low friction on the other side can eliminate the need for multiple parts with different compositions for load bearing implants such as total hip prosthesis. The presentation will primarily focus on processing and characterization of these graded Ti-TiO2 structures with special emphasis on biomedical applications.

11:30 AM

Anodization of Dental Archwires and Miniscrews: *Wu Hsin Jay*[1]; Huang Li-ling[1]; Chen Sinn-wen[1]; Liou Jein-Wein[2]; Lee Yueh-Tse[2]; [1]Department of Chemical Engineering, National Tsing Hua University; [2]Department of Orthodontics and Craniofacial Dentistry, Chang Gung Memorial Hospital

Commercial titanium and titanium-based archwires and miniscrews are anodized and examined in this study. The ß-Ti archwires with different colors are produced by anodization with different anodized voltages. The surface of the anodized wire is titanium oxide, and the oxidation states of Ti vary from TiO2 on the surface to inwardly a mixture of TiO2 and Ti2O3. For most of the anodization conditions, the oxide layers are amorphous. The thickness of the oxide layer is determined by using TEM and AES. With longer anodization time, the archwires change to milky white color. Compositional and structural analysis results of the milky white archwires indicate that it is still TiO2 on the surface, but the titanium oxide layers become crystalline. Ti-6V-4V miniscrews with mesoporous surfaces are produced by anodization using electrolyte with a small amount of fluorine.

Bulk Metallic Glasses VI: Alloy Development and Glass Forming Ability I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter Liaw, The University of Tennessee; Hahn Choo, The University of Tennessee; Yanfei Gao, The University of Tennessee; Gongyao Wang, University of Tennessee

Monday AM — Room: 3007
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: William Johnson, California Institute of Technology; Peter Liaw, University of Tennessee

8:30 AM Keynote

Metallic Glass Matrix Composites; A High Performance Structural Material: *William Johnson*[1]; Douglas Hofmann[1]; [1]California Institute of Technology

While bulk metallic glasses (BMG's) exhibit high strength and moderate fracture toughness, they lack a strain hardening mechanism and deform by highly localized shear banding. This leads to brittle failure under unconfined loading. In non-uniform stress states such as bending, shear band extension is limited by stress gradients and global plasticity becomes possible. To exploit this, one can "design" partially crystallized BMG-composites wherein inhomogeneous stress states are created by a two-phase microstructure under applied stress. Shear band initiation and propagation are then controlled by internal stress distributions created by the microstructure. To apply this strategy, one must consider the elastic/plastic properties of the glassy matrix and embedded crystals, spatial scale of the microstructure, and geometry of the crystalline inclusions. With proper design, BMG-composites can exhibit exceptional combinations of strength, toughness, and ductility. Experimental examples of successful BMG-composites will be presented.

8:55 AM

Bulk Metallic Glass Foams via Equal Channel Angular Extrusion: *Suveen Mathaudhu*[1]; Marie Cox[2]; Laszlo Kecskes[1]; K. Hartwig[3]; David Dunand[2]; [1]U.S. Army Research Laboratory; [2]Northwestern University; [3]Texas A&M University

To a first approximation, a ductile foam energy absorption scales with its strength. Thus, foams based on bulk metallic glasses (BMG), which have the highest strength of any metals, should be optimal. An obstacle to the use of BMG foams is the brittle behavior of BMG. However, recent demonstrations show that the thin, sub-millimeter, struts of BMG foams are ductile in compression, with outstanding energy absorption. The work presented here will demonstrate that equal channel angular extrusion (ECAE) can be used to create composites of BMG powders and metallic powders (Cu, Ni, W) which can subsequently be converted to BMG open-cell foams by leaching of the metallic second phase. These foams show excellent mechanical properties and particularly high energy absorption. Comparisons with similar melt cast BMG foams will be made. Acoustic emission measurements to assess damage accumulation will also be presented.

9:05 AM

Atomic Origins of High Glass Forming Ability of a Multicomponent Bulk Metallic Glass: *Mingwei Chen*[1]; [1]Tohoku University

Formation mechanism of multicomponent bulk metallic glasses (BMGs) with very low critical cooling rates has been one of the most outstanding issues in solid-state physics and materials science. Traditionally, atomic size ratios of constituent elements have been suggested to be the most important factor

governing glass forming ability (GFA), particularly in alloys only containing transition metals, and chemical effects arising from interatomic interactions have not been well assessed. In this talk we will report the atomic structure of multicomponent BMGs investigated by state-of-the-art experimental and computational techniques. The excellent GFA of the BMGs is demonstrated to be associated with chemical short- and medium-range order through the formation of extended clusters. This study uncovers the atomic origins of the excellent GFA of the multicomponent BMG and underscores the importance of chemical effect in the formation of amorphous structures in metal-metal based alloys.

9:15 AM Invited

Thermal Stability and Glass-Forming Ability of (Fe,Co)-Gd-Nb-B Glassy Alloys with Good Soft Magnetic Properties: *Wei Zhang*[1]; Fei Jia[2]; Xingguo Zhang[2]; Guoqiang Xie[1]; Hisamichi Kimura[1]; Akihisa Inoue[1]; [1]Tohoku University; [2]Dalian University of Technology

The glass-forming ability (GFA) and the stabilization of supercooled liquid of (Fe0.9Co0.1)71.5-xGd3.5B25Nbx (x=0-6) glassy alloys were greatly enhanced. The large supercooled liquid region of 105 K and the highest reduced glass transition temperature of 0.57 were obtained at x=4, leading to the formation of the glassy alloy rods with diameters up to 3 mm. The new Fe-based bulk glassy alloys exhibit good soft magnetic properties and high strength. However, a distinct two step glass transition phenomenon was observed in the x=4-6 alloys, and little is known about the two step glass transition of the Fe-based alloys. With the aim of clarifying the reason for the two-stage glass transition, we further examined the structure, thermal stability and crystalline behavior of (Fe,Co)-Gd-B, (Fe,Co)-Nb-B and (Fe,Co)-Gd-Nb-B glassy alloys by XRD, DSC and TEM. In addition, the effects of Co concentration on the thermal stability, GFA and magnetic properties of (Fe,Co)-Gd-Nb-B alloys were investigated.

9:30 AM Invited

Bulk Metallic Glasses for Biomedical Applications: *Marios Demetriou*[1]; Aaron Wiest[1]; Gongyao Wang[2]; Nikolaj Wolfson[3]; Bo Han[3]; William Johnson[1]; Peter Liaw[2]; [1]California Institute of Technology; [2]The University of Tennessee; [3]University of Southern California

Owing to a unique liquid-like atomic structure, glassy metals exhibit a combination of chemical and mechanical properties that are considered attractive for biomedical applications. These include a good corrosion resistance combined with high hardness, strength, and elasticity. There are certain features of their behavior however which from a bioengineering perspective can be regarded as inadequate, such as a near-zero ductility, a toughness that spans over a very broad range, and a fatigue performance that is at present poorly understood. More critically, most known compositions contain elements that are undesirable from a biomedical perspective (e.g. Ni and Cu), as they have been associated with adverse biological reactions. In this presentation, several new compositions designed according to a set of criteria to satisfy the requirements for chemical and mechanical biocompatibility will be introduced. Static and cyclic loading performances will be presented along with results from in vitro and in vivo cytotoxicity studies.

9:45 AM Invited

Processing of Bulk Metallic Glass: *Jan Schroers*[1]; [1]Yale University

The sluggish crystallization kinetic of bulk metallic glass results in two fundamentally different processing opportunities. BMG can be directly cast. But even for BMGs with low critical cooling rates geometries with high aspect ratio are particularly challenging since during casting cooling and filling of the mold must occur simultaneously. This limits the complexity of the geometries that can be cast even when processing parameters are carefully balanced. Alternatively, BMG can be thermo plastically formed in the supercooled liquid region. In this case the required fast cooling and forming are decoupled. The BMG is formed in a high viscous state where it behaves very similar to plastics when compared by processing temperature and forming pressure. A measure for the formability of BMGs will be introduced. Processing potentials and challenges will be discussed and various examples will be given including blow-molding, miniature fabrication, and nano-patterning.

10:00 AM Break

10:10 AM

Cooling Slope Casting Process for Synthesis of Bulk Metallic Glass Based Composites with Semisolid Structure: *Advenit Makaya*[1]; Takuya Tamura[1]; Kenji Miwa[1]; [1]National Institute of Advanced Industrial Science and Technology (AIST)

A process combining cooling slope casting and suction casting was developed to generate a semisolid structure in a Zr-based bulk metallic glass matrix composite. The melt was injected onto a cooling slope and subsequently vacuum-sucked into a cylindrical mould placed at the end of the slope. The structure obtained for 4-mm diameter cylindrical specimens of composition $Zr_{66.4}Nb_{6.4}Cu_{10.5}Ni_{8.7}Al_8$ consists of a dispersion of spheroidal and rosette-like crystals in a glassy matrix. The effects of parameters such as the cooling slope angle, the slope length and the casting temperature on the microstructure were studied. The obtained materials are expected to show improved mechanical properties and ductility inherent to the semisolid structure.

10:20 AM

New Low-Density, Low-Cost Glass-Dendrite Composites with Tensile Ductility: *Douglas Hofmann*[1]; Jin-Yoo Suh[1]; William Johnson[1]; [1]California Institute of Technology

Recently, nanostructure-dendrite composites have been demonstrated which are said to exhibit enhanced mechanical properties over metallic glasses and their composites. We notice however, that these new alloys are similar to metallic glass composites where the matrix material has crystallized, creating an apparently brittle material in unconfined loading conditions. In this work, we demonstrate that by successfully freezing the matrix material of the composites as a glass and designing the inclusions to have a low shear modulus, tensile ductility and fracture toughness similar to other high performance crystalline alloys is observed. We report a new system of low density (4.9-5.2 g/cm3) glass-dendrite composites with at least 5% tensile ductility in each alloy. The new alloys have low cost, exhibit volume fractions of glass from 20-70%, and high glass forming ability. The work demonstrate that metallic glass composites can be competitive with the best crystalline alloys for real structural applications.

10:30 AM

Fabrication of Cu-Zr-Al Bulk Metallic Glasses via Spark Plasma Sintering Process: *Zhihui Zhang*[1]; Troy Topping[1]; Ying Li[1]; Yizhang Zhou[1]; Enrique Lavernia[1]; [1]University of California

$(Cu_{0.5}Zr_{0.5})_{100-x}Al_x$ bulk metallic glasses have shown an exceptional combination of high strength and large compression ductility; however, their fabrication via conventional ingot casting methods is limited to samples of only a few millimeters (i.e., diameter ~5 mm). In this study, a series of $(Cu_{0.5}Zr_{0.5})_{100-x}Al_x$ alloys (x=0, 2.5, 5, 7.5 and 10), as amorphous precursor powders, are employed to study their consolidation behavior in the supercooled liquid region via spark plasma sintering (SPS). The SPS sintering was used to provide a fast heating rate (0-400 °C/min) and accurate control of the consolidation temperatures. X-ray diffraction indicated that fully amorphous disks were generated. In addition, partial crystallization was promoted during consolidation in order to study the in-situ formation of nanocrystalline precipitates during sintering as well as their influence on mechanical properties. The microstructure of the glassy billets was investigated using electron microscopy and differential scanning calorimetry. Mechanical properties under compression were evaluated.

10:40 AM Invited

Malleable Hypoeutectic Zr-Ni-Cu-Al Bulk Glassy Alloys with Tensile Elongation at Room Temperature: *Yoshihiko Yokoyama*[1]; Kazutaka Fujita[2]; Alain Yavari[3]; Peter Liaw[4]; Akihisa Inoue[1]; [1]Institute of Materials Research; [2]Ube National College; [3]LTPCM-CNRS UA29, Domaine Universitaire BP 75; [4]University of Tennessee

Ternary Zr-TM-Al (TM: Cu, Ni or Co) bulk glassy alloys were investigated to clarify the relationship among the volume, thermal, and mechanical properties, and we tried to accomplish the alloy design of Zr-TM-Al bulk glassy alloys. Ultimately, we found that the relationship between the Young's modulus and volume-change ratio provides the suitable guide for the alloy design of bulk glassy alloys. Based on this guide, we suggested that hypoeutectic bulk glassy alloys exhibit superior ductility due to enhancement of metallic bond nature, and never show the structural relaxation embrittlement. Furthermore, we examined about more ductile and malleable cast bulk metallic glass in hypoeutectic Zr-Ni-Cu-Al alloys with high Poisson's ratio over 0.39. This hypoeutectic Zr-Ni-Cu-Al bulk glassy alloy reveals the distinct tensile plastic elongation and unlimited compressive plastic deformation at room temperature.

10:55 AM
Synthesis and Behavior of Mg-Based Bulk Glasses via Spark Plasma Sintering (SPS): *Baolong Zheng*[1]; Zhihui Zhang[1]; Troy Topping[1]; Yizhang Zhou[1]; Chi Y.A. Tsao[2]; Enrique J. Lavernia[1]; [1]University of California, Davis; [2]National Cheng Kung University, Tainan

Mg-based bulk metallic glasses (BMGs) are of interest due to their high specific strength and excellent corrosion resistance relative to those of conventional Mg alloys. In particular, the Mg-Cu-Gd alloy has a good glass forming ability (GFA) and a relatively wide supercooled liquid region, which facilitates fabrication of bulk metallic glasses via consolidation of powder precursor. Spark plasma sintering (SPS) has recently been studied as an attractive consolidation technique for amorphous powders due to its fast heating rate and consolidation cycle. In view of these factors, the primary objectives of this research are: consolidate the Mg65Cu25Gd10 (at.%) amorphous powders into bulk component by using the SPS technique; investigate the microstructural evolution of Mg-Cu-Gd powder and their microstructural stability during thermal exposure of SPS processing using SEM, XRD, DSC, and TEM. Microstructural variations and mechanical properties were also investigated as a function of the initial powder size and SPS processing parameters.

11:05 AM
A Study on the Critical Stress for Continuous Shear Banding of a Bulk Metallic Glass: *Zheng Han*[1]; Hai Yang[1]; Yi Li[1]; [1]National University of Singapore

The serrated flow characteristic of the plastic deformation behavior of recently reported plastic bulk metallic glasses (BMGs) indicates that shear bands have been repeatedly initiated and arrested. By properly taking the instant load-bearing area into consideration, we have obtained sustained true stress of the BMG samples during plastic deformation under various distinct deformation modes. Our present work reveals that the critical stress for continuous shear banding maintains invariant on and after yielding over a large plastic strain, contrary to the previously reported assumptions of "strain-softening" and "strain-hardening". With an understanding of the mechanism of yielding and thus the origin of yield strength, we further point out that the atomic cohesive energy constantly serves to be the controlling factor of the critical stress for shear banding. On the other hand, the mechanism for arresting the propagating shear bands will be later suggested.

11:15 AM
Preparation of Spray-Formed La62Al15.7(Cu,Ni)22.3 Bulk Amorphous Alloy with Large Size: *Bin Yang*[1]; [1]University of Science and Technology Beijing

A large sized La62Al15.7(Cu,Ni)22.3 amorphous alloy was produced by melt atomization and spray deposition, and XRD proved a plate-like sample with a diameter of 340 mm and maximum thickness of 13 mm is fully amorphous. The formation mechanism of amorphous phase during the process was analyzed. The experimental results showed that there always exist some porosities in the spray-deposited La62Al15.7(Cu,Ni)22.3 amorphous alloy. The densification parameters of the La62Al15.7(Cu,Ni)22.3 amorphous alloy were determined by hot pressing in the undercooled liquid region of the amorphous alloy. The present results indicated that spray deposition is a potential technique to prepare bulk amorphous alloys with larger size, especially for bigger plate-shaped deposit.

Challenges for Sustainable Growth in the Aluminum Industry - Through the Current Crisis and on to the Future: Aluminum Plenary Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: David DeYoung, Alcoa Inc; Halvor Kvande, Hydro Aluminium AS; Geoffrey Bearne, Rio Tinto Alcan; Ray Peterson, Aleris International Inc; Wolfgang Schneider, Hydro Aluminium GmbH

Monday AM — Room: 2001/2003
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: David DeYoung, Alcoa Inc

8:30 AM
Welcome and Introduction

8:35 AM Keynote
Current Economic Situation, Comparison to Past Recessions in the Metals Industry, Future Projections: *Dave Persampieri*, CRA

9:05 AM
Dick Evans, Rio Tinto Alcan

9:30 AM
Jan Arve Haugen, Hydro

9:55 AM Break

10:10 AM
Galdino Claro, Aleris

10:35 AM
Bill O'Rourke, Alcoa

11:00 AM
To Be Announced, Rusal

11:25 AM Panel Discussion

Characterization of Minerals, Metals and Materials: Emerging Characterization Techniques

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee, TMS/ASM: Composite Materials Committee
Program Organizers: Toru Okabe, University of Tokyo; Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF

Monday AM — Room: 3009
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Ann Hagni, Geoscience Consultant; Jian Li, Natural Resources Canada

8:30 AM
Synchrotron X-Ray Computer Microtomographic and Light Optical Microscopic Investigation of Strength and Microstructure of Polymer-Metal Joints Produced by FricRiveting: *Sergio Amancio-Filho*[1]; Felix Beckmann[1]; Jorge dos Santos[1]; [1]GKSS Research Centre

The FricRiveting technique is an alternative new spot joining process developed for thermoplastic-lightweight alloy structures. In this technique a cylindrical metallic rivet is used to join one or more thermoplastic-metal components by means of plasticizing and deforming the tip of the rotating rivet through frictional heating. Advantages of this new technique are e.g. short joining cycles, minimal sample preparation, absence of environmental emissions and enhanced mechanical performance in comparison to base materials. This paper will demonstrate through qualitative and quantitative synchrotron X-Ray microtomographic and light optical microscopic analysis of a case-study joint on aircraft materials polyetherimide and aluminum 2024 - that FricRiveting joint performance under tensile loading is mainly dependent on geometrical

and microstructural features of the deformed tip of the rivet. The influence of processing on joint strength will be demonstrated in terms of rotation speed.

8:45 AM

On the Role of Low-Concentration Elements in the Formation of Misoriented Grains in Ni-Based Superalloys: Combination of Atom-Probe Tomography and Electron-Dispersive X-Ray Spectroscopy: *Yaron Amouyal*[1]; David Seidman[1]; [1]Northwestern University

Single-crystal, two-phase Ni-based superalloys are currently used for turbine blades and they represent an incredible technological achievement. Their superior properties are manifested by their high tensile strength and creep resistance up to a working temperature of 1400°C. Avoiding the formation of highly-misoriented grains (MGs) during solidification is crucial, since their boundaries are preferential sites for cracks and fast-diffusion. This goal can be achieved by characterization of the composition of such MGs. We employ both electron-dispersive X-ray spectroscopy (EDS) and atom probe tomography (APT) to determine the partitioning of elements into MGs in two-phase, multi-component (>10 elements) Ni-based superalloys. The combination of both techniques provides us with composition measurements for the large length scale of 10-100 μm, which is typical for EDS with high detectability (< ppm) typical for APT. We discuss the effect of several low-concentration (<500 ppm) elements on the formation of MGs in these alloys.

9:00 AM

3D Atom Probe Techniques for the Atomic Scale Investigation on the Quantitative Composition of Boron in Steels: *Jae Bok Seol*[1]; Ju Seok Kang[1]; Yo Sep Yang[1]; Chan Gyung Park[2]; [1]Pohang University of Science and Technology (POSTECH), Department of Materials Science and Engineering; [2]National Center for Nanomaterials and Technology (NCNT), Pohang University of Science and Technology (POSTECH)

It has been widely known to enhance the hardenability of any structural materials by the addition of a small amount of B due to the segregation of solute boron at austenite grain boundary. However, the analytical techniques to identify boron, such as Fission Track Etching (FTE) and Auger Electron Spectroscopy (AES), have limits on observing B in the concentration as low as a few ppm because of their poor sensitivity. Newly developed atom probe tomography (APT) can provide the highest available spatial resolution, 3D tomography imaging and resolve quantitative chemical information with a resolution better than 2nm. Therefore, the exact behavior and position of boron in steels can be investigated by the APT techniques in the sub-nanometer scale. The results obtained by 3D-APT revealed that the B atoms were mainly segregated to C enriched area rather than C depleted area, which were clearly a retained austenite and ferrite phase, respectively due to the strong attraction in the analytical volume of 35 x 35 x 96 nm^3. The current results demonstrate that the-state-of-art LAWATAP has made it possible to excellently identify and quantify the exact B state in steels with 3D tomography imaging and composition profile in the atomic scale.

9:15 AM

Two and Three-Dimensional Characterization of Microstructural Evolution in Ti-550 and Ti-6Al-4V: Vikas Dixit[1]; Santhosh Koduri[1]; John Sosa[1]; Dan Huber[1]; *Peter Collins*[1]; Hamish Fraser[1]; [1]Ohio State University

The microstructures of titanium alloys, which can significantly influence the mechanical properties, are often quite complex and span across length scales. The microstructural evolution of both a ß-processed Ti-alloy, Ti-550 (Ti-4Al-4Mo-1Zr-1Sn, wt%) and an a+ß-processed Ti-alloy, Ti-64, have been explored using both two-dimensional techniques, including optical microscopy, scanning electron microscopy (SEM), orientation microscopy (OM), and transmission electron microscopy (TEM), and their three-dimensional analogues (Robo. Met-3D™, DB-FIB/SEM, and (S)TEM tomography). The three dimensional datasets will be presented, and the feature size/fraction compared with the values obtained using two-dimensional stereological approaches.

9:30 AM

Three-Dimensional Materials Characterization Using HAADF-STEM Tomography: *Michael Sarahan*[1]; Daniel Masiel[1]; Bryant Gipson[1]; David Morgan[2]; Nigel Browning[1]; [1]University of California, Davis; [2]Indiana University

High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) is a powerful technique to visualize compositional differences in a material due to its sensitivity to atomic number(Z-contrast). We have used advanced image processing techniques to derive an average structure and the average chemical composition from HAADF-STEM images and to map variations from the average across the field of the original image. This allows us to resolve minute compositional details on the length scale of atoms. This study uses the analytical features of STEM imaging in combination with tomographic reconstruction of a tilt series of STEM images to create 3-dimensional models of nanomaterials with high-resolution compositional information. Additional high-resolution information in support of STEM images is obtained from diffractive imaging using iterative image reconstruction techniques, and STEM image-based models are refined using diffraction pattern-based models.

9:45 AM

High Resolution X-Ray Tomography for Polymeric Structural Composites: *Stephen Young*[1]; Dayakar Penumadu[1]; Vlastimil Kunc[2]; Eliot Specht[2]; [1]University of Tennessee; [2]Oak Ridge National Laboratory

Polymeric composites have been of significant interest in the automotive field due to their light weight and high strength. In the current research, 3-dimensional x-ray imaging techniques with high resolution are used to study the microstructure of glass and carbon fiber polymeric composites using polypropylene resin. The radiographs were reconstructed to visualize the fiber content and arrangement. In-situ tensile testing system was developed and integrated into the existing hardware for tomography equipment to study the evolution of damage and micro-structural features as a function of applied mechanical stress and example results are included. This research will pave the way to quantify damage and develop new class of scale-dependent constitutive models for composites materials for immediate use in structural and transportation applications.

10:00 AM Break

10:20 AM

In-Situ Characterization of Creep and Creep-Damage in Copper by X-Ray Microtomography: Federico Sket[1]; Krzysztof Dzieciol[1]; Augusta Isaac[1]; Marco Michiel[2]; Thomas Buslaps[2]; *Andras Borbely*[1]; Anke Pyzalla[1]; [1]Max Planck Institut für Eisenforschung; [2]European Synchrotron Radiation Facility

Geometrical shape and microstructure evolution during high temperature creep of a copper was in-situ characterized by X-ray microtomography. It is shown that geometric information of tomographic data combined with image correlation techniques is accurate enough to characterize plastic strain within material slabs with areas equal to the sample cross section but with heights of only 10-20 μm. Imposing a constant temperature gradient along the gauge length of the sample allows for evaluating the apparent activation energy of steady-state creep on a single specimen. The slice correlation technique developed for tracking local material flow during plastic deformation showed to be an excellent tool for tracking the evolution of single damage events, too. It was possible to evaluate the volumetric growth rate of more than 10,000 cavities evolving during the creep process. The obtained growth rates are in good correlation with the "power law creep" theory of Cocks and Ashby.

10:35 AM

Spatially Resolved Diffusivity Measurement with Thermography: *Christian Wögerer*[1]; Gerhard Traxler[1]; [1]Profactor Research and Solutions GmbH

For material or process development as well as for quality checking of materials, thermal diffusivity measurement may be the tool of choice in many applications. Especially if diffusivity is measured locally resolved, a lot of information can be read out of such a "diffusivity image". This paper explains the method to measure parallelpiped specimen like plates and lists some interpretations of diffusivity images, like inhomogeneity of sintered materials, structural voids or inclusions. Diffusivity is a material property, describing transportation of heat in solids in spatial and temporal terms. With thermo cameras it is possible, to measure diffusivity of larger areas simultaneously. The result is an area of diffusivity values that may be plotted or displayed like an image. Locations with lower diffusivity values could be caused by the presents of structural differences in the material, by decomposition of the material components or by inclusions of other material like air.

10:50 AM

In-Situ TEM Observation of Repeating Events of Nucleation in Epitaxial Growth of Nano CoSi2 in Nanowires of Si: *Yi-Chia Chou*[1]; King-Ning Tu[1]; Lih-Juann Chen[2]; Wen-Wei Wu[3]; [1]University of California, Los Angeles; [2]National Tsing Hua University; [3]National Chiao Tung University

CoSi2 formation in Si nanowires has been investigated in high-resolution transmission electron microscopes at 800°C by point contact reactions between Co and Si nanowires. The CoSi2 has undergone an axial epitaxial growth in

the Si nanowire and a stepwise growth mode was found. We observed that the stepwise growth occurs repeatedly in the form of an atomic step sweeping across the CoSi2/Si interface. It appears that the growth of a new step or a new silicide layer requires an independent event of nucleation. We are able to resolve the nucleation stage and the growth stage of each layer of the epitaxial growth in video images. The epitaxial growth consists of a repeating nucleation and a rapid stepwise growth across the epitaxial interface. This is a general behavior of epitaxial growth in nanowires. A discussion of the kinetics of supply-limited reaction in nanowire case by point contact reaction is proposed.

11:05 AM

The Application of Electron Backscatter Diffraction to Study Abnormal Grain Growth and Twinning in Nanocrystalline Nickel: *Hsiao-Wei Yang*[1]; Shehreen S. Dheda[1]; John R. Porter[1]; [1]University of California

Electrodeposited (ED) Nanocrystalline (nc) nickel, with a grain size of 20nm, has the superior properties typical of nc materials. Understanding the effect of thermal annealing on grain growth morphology and kinetics is important to fully determine the functional capabilities of these materials. Nc nickel has been observed to undergo both grain growth and the formation of twinning upon annealing. Electron backscatter diffraction (EBSD) in a scanning electron microscope (SEM) was used to measure the microstructural evolution due to heat treatment at temperatures between 0.27 and 0.43 of the melting temperature. Sequential EBSD mapping shows the change in grain size, twinning structure and overall texture of the material. Abnormal grain growth and the propensity of coherent twin boundaries (coincidence site lattice (CSL) sigma 3 boundaries) to develop were shown to increase with increasing annealing temperature. The implication of this observation for the application of nc nickel will be discussed.

Computational Thermodynamics and Kinetics: Energy Materials

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, ASM Materials Science Critical Technology Sector, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee

Program Organizers: Long Qing Chen, Pennsylvania State University; Yunzhi Wang, Ohio State University; Pascal Bellon, University of Illinois at Urbana-Champaign; Yongmei Jin, Texas A&M

Monday AM — Room: 3002
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Christopher Wolverton, Northwestern University

8:30 AM Introductory Comments

8:35 AM Invited

Thermodynamics and Kinetics of Phase Transformations in Hydrogen Storage Materials: Alireza Akbarzadeh[1]; Hakan Gunaydin[1]; Kyle Michel[1]; Kendall Houk[1]; Christopher Wolverton[2]; *Vidvuds Ozolins*[1]; [1]University of California, Los Angeles; [2]Northwestern University

General adoption of hydrogen as a vehicular fuel depends on the ability to store hydrogen at high volumetric and gravimetric densities, as well as on the ability to extract and recharge H2 at sufficiently rapid rates. We will show how first-principles density-functional theory (DFT) techniques can be used to gain fundamental understanding of the thermodynamic driving forces and detailed atomic-scale kinetic mechanisms of phase transformations involved in hydrogen storage reactions. The power of these techniques will be illustrated on several examples of recent work conducted in our group: (i) predicting the crystal structures and thermodynamic properties of several new solid-state borohydrides and amides/imides, (ii) calculating multicomponent phase diagrams, favored reaction pathways, and designing new thermodynamically reversible hydrogen storage reactions in borohydrides, and (iii) clarifying the atomic-scale kinetics of mass transport and hydrogen release in aluminum, sodium alanate, and lithium amide. Research supported by DOE DE-FG02-05ER46253 and DE-FG02-07ER46433, and NSF CBET-0730929.

9:05 AM Invited

***Ab-Initio* Modeling of SOFC Cathodes**: *Dane Morgan*[1]; Yueh-Lin Lee[1]; [1]University of Wisconsin - Madison

Perovskites are the major class of materials used for modern solid oxide fuel cell (SOFC) cathodes and have the ability to catalyze the oxygen reduction reaction (ORR) on their surfaces. However, difficulties in performing *in-situ* characterization of well-controlled samples means that the rate limiting steps and structure-property relationships underlying ORR on these materials are not understood. We have used *ab-initio* based thermokinetic modeling to study $(La,Sr)MnO_3$ (LSM), which is the primary cathode catalyst used in commercial SOFCs, and developed a combined bulk and surface defect model in order to better understand surface defect structure and catalytic properties. We have also studied lanthanum transition metal oxides $LaBO_3$ (B= Fe, Co, and Ni) to understand trends of surface oxygen binding, hoping, vacancy formation, and dissociation vs. transition metal types. These studies are being used to develop molecular level models of the ORR on SOFC cathodes.

9:35 AM

Hydrogen Storage on Li-Dispersed Carbon Nanotubes: *Wei Liu*[1]; Yonghao Zhao[1]; Ying Li[1]; Qing Jiang[2]; Enrique J. Lavernia[1]; [1]University of California, Davis; [2]Jilin University, China

High storage capacity and a moderate binding strength are ideal for hydrogen storage materials. Inspection of the literature indicates that the highest H2 storage capacity by simulation is 13 wt% obtained in the fullerene doped by 12 Li atoms. Using density functional theory, we achieved a similar high uptake capacity (about 13.45 wt%) by optimizing 8 Li on carbon nanotubes. The binding is found to be dramatically enhanced when the additional dopants are introduced. The electronic orbital analysis shows that dopants are essential for storage, whose bands overlap strongly with those of H2 and the nanotube simultaneously. The presence of an electric field is demonstrated to have a significant influence on the 8-Li-doped model. The calculated adsorption energy Ead decreases dramatically to -0.58 eV/H2, which is 4.5 times lower than that of pure nanotubes. This increase is attributed to the further ionization of dopants under electric fields.

9:55 AM

Role of Particle Size Distribution in Long Term Platinum Surface Area Loss in PEMFC Cathodes: *Edward Holby*[1]; Yang Shao-Horn[2]; Wenchao Sheng[2]; Dane Morgan[1]; [1]University of Wisconsin-Madison; [2]Massachusetts Institute of Technology

Long-term durability of Pt catalysts in proton exchange membrane fuel cell (PEMFC) cathodes is an important issue in achieving the commercially viability of PEMFC technology. Fuel cell efficiency is decreased as Pt electrochemically active surface area (ECASA) is lost. It is important to better understand the mechanisms of Pt surface area loss in the cathode and if they can be mitigated in order to meet lifetime requirements. Currently, ECASA loss is attributed to four mechanisms: Ostwald ripening of Pt nanoparticles; Pt nanoparticle migration and coalescence; detachment of Pt particles from the carbon support; and dissolution and reduction of Pt off of the carbon support due to crossover hydrogen from the anode. By modeling these processes, the effect of the initial particle size distribution of the Pt nanoparticles and its role in long-term ECASA loss is investigated.

10:15 AM

First Principles Modeling on Stability Mechanism of Cuboctahedral Clusters in UO_{x+2}: *Ying Chen*[1]; Hua Yun Geng[1]; Yasunori Kaneta[1]; Motoyasu Kinoshita[2]; [1]University of Tokyo; [2]Central Research Institute of Electric Power Industry

UO_2 is a widely used fuel materials in nuclear reactor, its performance is quite related to the defects behavior under irradiation environment which arises the deviation from stoichiometry of the compound. To clarify ambiguousness remaining for long in structure of nonstoichiometric uranium dioxide, the stability mechanism of oxygen clusters is investigated by first-principles LSDA+U method. A new physical model of thermodynamic competition between cuboctahadral cluster and point oxygen interstitials is proposed. Calculations reveal that the structural stability of the cuboctahadral cluster embedded into the crystal UO_2 is inherited from U_6O_{12} molecular, and the energy gain through occupying its center by one additional oxygen makes the cluster win out by competition to point oxygen interstitials at the ground state. By incorporating the temperature effect, a pseudo phase diagram of temperature and the oxygen concentration is constructed which shows that the elevation of temperature favors point interstitial over cuboctahedral clusters.

10:35 AM Break

10:50 AM Invited

First-Principles Solid-State Kinetics: *Anton Van der Ven*[1]; [1]University of Michigan

While much progress has been made in the first-principles prediction of the thermodynamics of multi-component solids, predicting the kinetics of solid-state phase transformations remains a major challenge. Both diffusion and the kinetics of first-order phase transformations in intercalation compounds (e.g. LixC6, LixCoO2, Li(Ni0.5Mn0.5)O2, LixTiS2 …) play an important role during operation of modern Li-ion batteries, as a charge-discharge cycle leads to the removal and reinsertion of Li ions from the cathode intercalation compounds. Similar kinetic processes occur in hydrogen storage materials and during corrosion processes of structural materials involving hydrogen embrittlement. In this talk, I will describe how first-principles electronic structure methods combined with statistical mechanical techniques from alloy theory have elucidated complex diffusion mechanisms in intercalation compounds and how they can shed light on the mechanisms of first-order diffusional and structural phase transformations in the solid state.

11:20 AM Invited

An Efficient Method to Study Ordering in Low-Symmetry Materials: *Tim Mueller*[1]; Gerbrand Ceder[1]; [1]Massachusetts Institute of Technology

Cluster expansions are commonly used to develop effective Hamiltonians for systems with substitutional disorder. The coefficients of the linear expansion are typically fit to training data generated using *ab-initio* methods. Low-symmetry systems, such as nanoparticles and materials with crystal defects, require the determination of a large number of coefficients. A large amount of training data must be generated for such problems, and the cost calculating the energy of each training structure is high due to the low symmetry of the system. For these reasons it has been impractical to use the cluster expansion to study low-symmetry materials with the same level of accuracy as bulk materials. We address this problem by demonstrating new methods that significantly reduce the prediction error of a cluster expansion for a given training set size. Our approach makes it possible to study atomic ordering in low-symmetry systems at a fraction of the current computational cost.

11:50 AM

Computer Simulation on Thermoelectric Energy of Rh-Ir Alloy: *Zhongliang Xiao*[1]; Weilian Zheng[1]; Yan Shi[1]; Xuehui Zhan[1]; [1]Changsha University of Science and Technology

As the environment and the energy crisis, research on thermoelectric materials become a hotspot because of their no pollution, no mobile parts and transformation between electricity and thermal energy. One of the studying hotspots is how to predict thermoelectric energy of thermoelectric materials. thermoelectric energy of Rh-Ir alloy was investigated in this paper. At first the super cell models for the alloy were built by changing the composition of alloy and its crystal structure in CASTEP. Then the density of states (DOS), Fermi energy and the electronics structure can be calculated with the plane-wave pseudo potential local density algorithm (LDA) in CASTEP software package. The thermoelectric energy can be obtained according to the Onsager's relationship and DOS of alloys with different compositions. The calculated results is consistent with that from measurement.

12:10 PM

Molecular Dynamics Simulations on the Inhibition of Methane Hydrate Formation by Polyesteramides: *Zhiju Zheng*[1]; Monica Lamm[1]; Richard LeSar[1]; [1]Iowa State University

Gas hydrates form in transmission pipelines as stable solid networks composed of hydrogen-bonded water molecules trapping a second group of encaged molecules, such as methane and hydrogen, leading to undesired blockages in the lines that result in both economic loss and safety risks. To control the formation of hydrates, additives can be added that inhibit hydrate plugging. In this study, we employ atomistic simulations to examine the effects of hyperbranched polyesteramides, Hybrane H1500 (1,3-isobenzofurandione, hexahydro-, polymer with 1,1´-imino-bis[2-propanol]) on hydrate formation. These systems have large numbers of hydroxyl functional groups that form extensive hydrogen bonding with water. All-atom molecular dynamics simulations were used to determine the binding energy and structure of the hydrate/hybrane systems to elucidate the fundamental mechanism for hydrate inhibition. We discuss how the structure, polarity, and size of the polymers influence the performance of these polyesteramides as hydrate inhibitors.

Dislocations: 75 Years of Deformation Mechanisms: Dislocation Structures and Effects of Material Microstructure

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: David Bahr, Washington State University; Erica Lilleodden, GKSS Research Center; Judy Schneider, Mississippi State University; Neville Moody, Sandia National Laboratories

Monday AM — Room: 3022
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Neville Moody, Sandia National Laboratories; Kip Findley, Colorado School of Mines

8:30 AM

Atomistic Modeling of Low Temperature Dislocation Plasticity in α-Fe: *Neeraj Thirumalai*[1]; Youhong Li[1]; Michael Luton[1]; Ju Li[2]; Liu Cao[3]; Peter Gordon[1]; [1]ExxonMobil Research and Engineering Company; [2]University of Pennsylvania; [3]The Ohio State University

It is well-known that at low temperatures the flow stress in α-Fe exhibits strong temperature dependence. This dependence arises from the motion of screw dislocations, known to be controlled by double-kink nucleation. Previous atomistic studies suggested that this temperature sensitivity is partly a consequence of the polarized core structure of the screw dislocations. However, recent ab initio calculations have shown the core structure of screw dislocations to be compact. We have investigated the kink nucleation pathways that control screw dislocation motion using a recently developed interatomic potential for α-Fe using Nudged Elastic Band (NEB) and Molecular Dynamics (MD) simulations. In addition, we have also investigated the stability of core states using ab-initio calculations. In this presentation we will discuss the results of this study and its relevance towards understanding low temperature plasticity in α-Fe.

8:50 AM

Modeling and Dislocation Dynamics Study of Precipitation Strengthening in Steels at Low Temperature: *Ghiath Monnet*[1]; Benoit Devincre[2]; [1]EDF - R&D; [2]CNRS

At low temperature in iron, dislocation mobility depends strongly on the dislocation character. Screw dislocations are known to move slowly compared to other dislocations and are found to control plastic behavior of steels. The classical picture of dislocation interaction with precipitates is no longer valid and the corresponding strengthening cannot be predicted using classical theories, based on the line tension approximation. In this work we provide a new model, supported by the results of dislocation dynamics simulations, describing the effect of temperature, the strain rate and the precipitation microstructure on the flow stress. It is shown that the difference in mobility between dislocations of different character induces a difference in the effective stress on these dislocations. This leads systematically to a decrease of the precipitation strengthening compared to that measured in the athermal regime.

9:10 AM

The Effect of Microstructure on the Low Temperature Dislocation Behavior in Plate Steels: *Kimani Partin*[1]; Kip Findley[1]; Chester Van Tyne[1]; [1]Colorado School of Mines

Plate steels are often used in low temperature applications such as chemical transport and naval armor. However, the strengthening mechanisms at low temperature in light of dislocation mobility are not fully understood. The focus of this study is to examine the effect of microstructure on plastic flow and strengthening behavior in steels. Noble and Hull and later Gupta and Li developed methods utilizing stress relaxation tests to determine the dislocation velocity exponent, m*. Combined with tensile testing, the temperature independent internal stress and temperature dependent effective stress can be calculated. These methods have been used to evaluate a single steel grade, ASTM A514. Testing of green, as-quenched, and quenched and tempered conditions was performed to compare steel with the same composition but different microstructures. The activation energies to overcome the Peierls barrier for dislocation motion are

also compared, and the results provide insight into the differences in strength between microstructures.

9:30 AM

Ternary Junctions and Hardening in BCC and FCC Crystals: *Ronan Madec*[1]; Ladislas Kubin[2]; [1]Commissariat a l'Energie Atomique; [2]CNRS/ONERA

When two dislocations cross each other, they may form a junction. When a third dislocation crosses a junction, a ternary junction may be formed. We examine the possible configurations of ternary junctions in bcc and fcc crystals and estimate their contribution to strain hardening. Two distinct families are found: axial ternary junctions are formed when three slip planes share a common axis; zigzag ternary junctions form when the binary junctions can glide to react with a third dislocation along a new direction. In bcc metals, the second type of reaction is statistically the most probable and the strongest junction is associated with a zigzag ternary junction. Ternary junctions are found to induce Taylor hardening only in bcc metals, whereas an increased strain hardening is expected to result from increased dislocation storage in both fcc and bcc metals.

9:50 AM

Dislocation Dissociation and Locking in Supersaturated Co-Ni Based Superalloys Due to Aging Treatment: *Akihiko Chiba*[1]; Satoshi Tadano[1]; Hiroaki Matsumoto[1]; Toyohiko Konno[1]; [1]Tohoku University

To establish high-temperature strengthening materials with excellent ductility, we have studied a new strengthening mechanism operative in Co-based alloys without η' precipitation. Dislocation structures in a Co-Ni-based alloy have been examined. The alloy studied is found to be a supersaturated solid solution with fcc crystal structure. With aging treatment at 973K for 10h after pre-straining the alloy at room temperature, the dislocations widely dissociate into Shockley partials bounding the stacking fault (SF), resulting from Suzuki segregation to the SF bounded by Shockley partials. It has been found that the widely dissociated dislocations is transformed into precipitations of δ phase with crystal structure of DO19, resulting in strengthener of the Co-Ni based alloy at around 800°C. As a result, the dislocations which exhibit tendency to be widely dissociated into the Shockley partials at elevated temperatures can act as the effective strengthener of the high temperature strength of the alloys.

10:10 AM Break

10:30 AM

Fine Structure of c-Component Dislocations Associated with Pyramidal Slip Activity in Ti3Al: *Jorg Wiezorek*[1]; Andreas Kulovits[1]; [1]University of Pittsburgh

The hexagonal ordered phase Ti3Al is a minor constituent in current TiAl-based alloys, which offer potential for applications in advanced transportation system. The minority phase affects the deformation behavior of lamellar grains in promising these TiAl alloys. General plastic deformation of Ti3Al requires pyramidal plane c-component dislocation slip, {2-201}<11-26> and {11-21}<11-26>, which operates only for loading close to the c-axis and exhibits anomalous yielding. We use binary Ti-48at%Al model alloys to elaborate the fine structure of c-component dislocations activated during c-axis loading of lamellar grains in TiAl-alloys at ambient and elevated temperature by conventional diffraction contrast and high-resolution transmission electron microscopy. In addition to the large amounts of debris characteristically associated with pyramidal slip in Ti3Al, we have observed non-planar dissociated configurations of the c-component dislocations with atomic resolution. The results are discussed in relation to the mechanical behavior of Ti3Al and two-phase TiAl.

10:50 AM

Monazite (Monoclinic LaPO4) Slip Systems at Room Temperature: *Randall Hay*[1]; [1]Air Force Research Laboratory

Polycrystalline monazite (monoclinic LaPO4) was deformed by spherical indentation at room temperature. Dislocation Burgers vectors were identified by Burgers circuit closure in high resolution TEM images, supplemented by diffraction contrast where possible. A total of 441 b determinations were made in 97 grains. The most common slip systems were [001]/(010), [100]/(010), and [010]/(100). Slip on (001) was less common. Many other less dislocations were also identified, including [101], [10-1], [011], [110], and [111]. [101] dislocations dissociate into ½[101] and ½[10-1] partials. b = [100] dislocations may dissociate into ¼[210] + ¼[2-10] partials. Several other partial dislocations were tentatively identified. All partial dislocations were climb dissociated. Dislocation line energies were calculated, and stacking fault structures and energies between partial dislocations were analyzed. Satisfaction of the Von Mises criteria most likely involves [101]/(11-1) and <011>/{011} slip. If deformation twinning is active <011> slip may not be necessary for full ductility.

11:10 AM

Deformation within Molecular Single Crystals: *Kyle Ramos*[1]; Daniel Hooks[2]; David Bahr[1]; [1]Washington State University; [2]Los Alamos National Laboratory

Dislocations have remained relatively unexplored within molecular crystals. Lower symmetry, intermolecular degrees of freedom, and susceptibility to electron beam degradation have prohibited studies of deformation within these materials. Using the newer technological capabilities of nanoindentation and scanning probe microscopy, older etching analysis techniques have been revitalized to overcome some complications associated with molecular crystals and enable the experimental investigation of deformation at an elementary level previously unattainable. The approximate two order of magnitude increase in characterization resolution has permitted experimental observations that are near length scales attainable for molecular dynamics simulations employing fully flexible potentials. A collaborative experimental-simulation effort promises a new insight to dislocation mediated plasticity within molecular materials. Experimental data from several molecular crystals will be presented to emphasize new capabilities and future possibilities.

11:30 AM

Slip Paths of <c+a> Dislocations and Stacking Faults in HCP Metals: *Bin Li*[1]; Evan Ma[1]; [1]Johns Hopkins University

The nature of the pyramidal slip of <c+a> dislocations in HCP metals remains unclear. We present molecular dynamics (MD) simulations in single-crystal HCP magnesium, as well as TEM observations. The <c+a> slip is found to be composed of two consecutive dislocation slips, each having a Burgers vector of only about one half of <c+a> and gliding in a corrugated manner adjacent to the twinning plane. The constituent dislocations of the <c+a> slip nucleate independently, leading to a wide stacking fault (hundreds of nanometers) on the twinning plane. We have indeed observed such stacking faults under TEM. This mode of deformation can be generalized to the four major twinning modes observed in HCP metals, regardless of the c/a ratio. The relationship between the <c+a> dislocations and the twinning dislocations will also be briefly discussed, and elaborated in a separate talk.

11:50 AM

Dislocation Dipole Formation and Breaking during Shear Deformation: *Dongsheng Xu*[1]; Hao Wang[1]; Rui Yang[1]; [1]Institute of Metal Research, Chinese Academy of Sciences

Molecular simulations were carried out to investigate the formation and breaking of dislocation dipoles in some fcc metals and intermetallic compounds. Dipoles of various heights and orientations were formed and their configuration changes during shear deformation were studied. It was found that for dipoles with small height, of several interplanar spacing, due to the strong reaction among the constituent dislocations, various reacted structures formed, such as faulted dipoles, stacking fault tetrahedra and zigzagged configurations, depending on the dipole height and deformation temperature. The breaking stress of small height dipoles increased substantially due to the formation of the above products, compared with elastic calculations. Furthermore, these reacted products are hard to move or break, and will be strong obstacles to the movement of other dislocations, so as to contribute to work hardening during deformation and may serve as the anchoring point for the dislocation wall formation during fatigue process.

12:10 PM

A Quantitative Model of Strength in Mg from First-Principles: *Joseph Yasi*[1]; Louis Hector[2]; Dallas Trinkle[1]; [1]University of Illinois at Urbana-Champaign; [2]General Motors Technical Center

Computational modeling of solute-dislocation interactions in magnesium is essential to develop new alloys with high ductility. We present a quantitative first-principles model of plastic deformation in Mg from density functional theory calculations of basal and prismatic dislocation core geometries with and without applied stress. An ultrasoft pseudopotential for Mg with PW91 GGA accurately reproduces lattice constants, elastic constants, phonon spectra and stacking fault energies from experiment. We calculate the Peierls stress, dislocation core splitting and the activation energy for cross-slip for basal and prismatic screw, edge and mixed dislocations. To include alloying effects, we calculate dislocation-solute interaction energies and changes in the dislocation

core splitting with Al and Zn solutes. The first-principles data parameterizes a model for the motion of a dislocation in a field of solutes at finite temperature to predict the strength of Mg alloys.

Emerging Applications of Neutron Scattering in Materials Science and Engineering: Neutron Diffraction and Structure Determination

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Xun-li Wang, Oak Ridge National Laboratory; Brent Fultz, California Institute of Technology; Hahn Choo, University of Tennessee

Monday AM Room: 3012
February 16, 2009 Location: Moscone West Convention Center

Session Chairs: Xun-li Wang, Oak Ridge National Laboratory; Bill David, STFC

MONDAY AM

8:30 AM Invited

Scientific Opportunities at the SNS and Upgraded HFIR: *Ian Anderson*[1]; [1]Oak Ridge National Laboratory

The Spallation Neutron Source (SNS) at Oak Ridge National Laboratory (ORNL) provides the research community with access to the most intense pulsed neutron beams in the world. ORNL is also home to the High Flux Isotope Reactor (HFIR), one of the world's most powerful research reactors. Together, the SNS and HFIR provide scientists and engineers around the world with the opportunity to carry out research at the forefront of physics, chemistry, materials science, engineering, and biology. A project to upgrade the power of the SNS accelerator from 1.4 MW to 3 MW is underway and construction of a second, long pulse target station is planned. These new facilities will extend the present capabilities of the neutron scattering techniques to high resolution studies of both the structure and dynamics of materials in a wide range of environments.

9:00 AM Invited

Neutron Powder Diffraction Studies of Hydrogen Storage Materials: *Bill David*[1]; Marco Sommariva[1]; [1]STFC

This presentation focusses on the use of neutron powder diffraction to study the detailed behaviour of hydrogen storage materials. Particular emphasis will be given to the combined in-situ gravimetric and neutron powder diffraction analysis that enables detailed structural and gravimetric information to be obtained as a function of both temperature and pressure. Neutrons are the ideal tool to investigate the structure of hydrogen storage materials and, in addition to discussing in-situ hydrogen absorption and desorption measurements, high resolution diffraction data will be presented that provide insights into the detailed mechanisms of lightweight hydrogen storage materials.

9:30 AM

Time Resolved Neutron Diffraction Studies of Defect Structure Formation in Lithium Imide during Hydrogenation and De-hydrogenation: *Ashfia Huq*[1]; Jason Hodges[1]; Luke Heroux[1]; Evan Maxey[2]; Dhanesh Chandra[3]; [1]Oak Ridge National Laboratory; [2]Argonne National Laboratory; [3]University of Nevada, Reno

Reversible hydrogen absorption and desorption properties of lithium imide to lithium amide at 250°C have generated a great interest in this system. The structure of lithium amide is fairly well established where as there is significant debate about the structure of lithium imide. While there is general agreement about the position of the Li and N atoms which form an antiflourite structure, several types of hydrogen positions have been proposed. In all these models, however, a common theme is the disordered hydrogen. In situ neutron diffraction measurement revealing a variation in stoichiometry of cubic lithium imide during hydrogenation and dehydrogenation at 250°C will be presented. ORNL/SNS is managed by UT-Battelle, LLC, for the U.S. Department of Energy under contract DE-AC05-00OR22725. Work at ANL supported by the U.S. DOE, Basic Energy Sciences—Materials Sciences, under Contract W 31-109-ENG-38.

9:50 AM

Neutron Diffraction Experiments of Polarized Protons: *Maths Karlsson*[1]; Ted Forgan[2]; Eddy Lelièvre-Berna[3]; Ken Andersen[3]; Christian Vettier[1]; Colin Carlile[1]; Trevor Forsyth[3]; Garry McIntyre[3]; Patrik Carlsson[1]; [1]European Spallation Source Scandinavia; [2]University of Birmingham; [3]Institut Laue-Langevin

Hydrogen atoms play a key role in many materials of high interest. X-ray and neutron diffraction are the tools of choice for structural studies, but X-rays are not very sensitive to hydrogen, and neutrons give a large background due to incoherent scattering from the hydrogen. We propose to develop a new method, with which we will exploit the strong spin dependence of the proton cross section and reduce the incoherent scattering from the hydrogen by aligning their nuclear spins. A very large increase in the signal-to-noise ratio will then result, which will enable new science and improve existing methods for studying hydrogenous materials. We aim to implement the method on a suitable instrument at ILL, with the longer term goal to install it at the forthcoming European Spallation Source. Initially though, we will investigate the key aspects of the technique. In this talk we present our first results from this work.

10:10 AM Break

10:30 AM Invited

High-Pressure Neutron Diffraction Studies for Materials Sciences and Energy Sciences: *Yusheng Zhao*[1]; [1]Los Alamos National Laboratory

The neutron diffraction under pressure and temperature conditions is highly valuable to condensed matter physics, crystal chemistry, materials sciences. We have incorporated a 500-ton press TAP-98 into the HiPPO diffractometer to conduct in situ high P-T neutron diffraction experiments. Recently, we have developed high-P low-T gas/fluid cells in conjunction with neutron diffraction and inelastic neutron scattering instruments. We have successfully used these techniques to study the equation of state, structural phase transition, and thermo-mechanical properties of metals, ceramics, and minerals. We have conducted researches on the formation of methane and hydrogen clathrates, and hydrogen adsorption of the inclusion compounds such as the recently discovered metal-organic frameworks. The aim of our research is to accurately map phase diagram, lattice parameters, thermal parameters, bond lengths, bond angles, neighboring atomic environments, and phase stability in P-T-X space. Studies based on high-pressure neutron diffraction are important for multidisciplinary science, particularly for the theoretical/computational modeling/simulations.

11:00 AM

Investigation of the Crystallographic Structure of the ε Phase in the Fe-Al System by High-Temperature Neutron Diffraction: *Sven Vogel*[1]; Frank Stein[2]; Martin Palm[2]; M. Eumann[2]; [1]Los Alamos National Laboratory; [2]MPI für Eisenforschung GmbH

In the central part of the Fe-Al system between about 58 and 65 at.% Al, a high-temperature phase denoted as ε occurs with a hitherto unknown crystallographic structure. The phase is stable between 1231 and 1095°C and disintegrates at the lower temperature by a spontaneous eutectoid reaction into a fine-scaled, lamellar FeAl (B2) + FeAl2 microstructure. Because this reaction can not be suppressed even by rapid quenching, the crystallographic structure of the ε phase could not be determined yet. An alloy with 60 at.% Al, i.e. approximately eutectoid composition, has been produced by crucible-free levitation melting. In order to study the crystallographic structure of the high-temperature ε phase, in-situ high-temperature neutron diffraction experiments have been performed at the HIPPO instrument at Los Alamos Neutron Science Center (LANSCE) at Los Alamos National Laboratories (LANL).

11:20 AM

Neutron Diffraction Study of Structure Parameters of Chemisorbed NaX and NaY Zeolite Catalysts: *Stanislav Vratislav*[1]; Maja Dlouhá[1]; Vladimír Bosácek[2]; [1]CTU in Prague, Faculty of Nuclear Sciences and Physical Engineering; [2]CAS, Institute of Physical Chemistry of Jaroslav Heyrovsky

The regular structure of zeolites can be easily modified, many laboratories try therefore to "tailor" zeolitic catalysts of the requested properties. Nature of acid or basic sites, their amount and distribution in the zeolitic lattice belong to the most important problem of surface chemistry. Some organic cations like methylium or ethylium, create chemisorbed, with more or less polarized, but covalently bonded alkoxy species in zeolitic structures. Careful 13C MAS NMR measurents make possible to distinquish between signals of bridging and terminal methoxy groups. Neutron powder diffraction patterns were collected from evacuated ampoules with samples at room and 7 K on the KSN-2 diffractometer

which is placed at the LVR-15 research reactor in Rež near Prague. The complete structural parameters (including chemisorbed ions at O1 and O4 in NaX and at O1 in NaY and redistribution of Na+ cations) were determined by Rietveld analysis using GSAS package.

11:40 AM

First Results from POWGEN: A New Neutron Powder Diffractometer at the SNS: *Jason Hodges*[1]; Ashfia Huq[1]; Olivier Gourdon[1]; Luke Heroux[1]; [1]Oak Ridge National Laboratory

POWGEN is a fundamental departure from previous designs for a time-of-flight powder diffractometer at a spallation neutron source and may be considered a third-generation design. The instrument is optimized for both parametric studies of materials under a wide range of conditions (T, P, H, flowing gases, etc) and *ab-initio* crystal structure determinations of complex solid-state materials with asymmetric unit-cells of the order ~1500 Å^3. The geometric design of the instrument allows for all detected scattered neutrons to be focused onto a single diffraction profile yielding high count rate while preserving good resolution ($\Delta d/d$ = 0.0015 at d = 1 Å). The new time-event mode for data acquisition will permit stroboscopic experiments with approximately 100 ms time resolution. Early results from commissioning experiments will be presented.

12:00 PM

Nested Neutron Microfocusing Optics on SNAP: *Gene Ice*[1]; Jae-Young Choi[2]; Peter Takacs[3]; Yevgeniy Puzyrev[4]; Jamie Molaison[4]; Chris Tulk[4]; Ken Andersen[5]; Terry Bigault[5]; [1]Oak Ridge National Laboratory; [2]Pohang Accelerator Laboratory; [3]Brookhaven National Laboratory; [4]Oak Ridge National Laboratory; [5]Institut Laue-Langevin

The Spallation Neutron Source (SNS) together with large detector solid angles, now makes possible neutron experiments with much smaller sample volumes. Nested Kirkpatrick-Baez supermirror optics provide a practical and efficient way to further decrease the useable neutron sample size by focusing polychromatic neutron beams. Because the optics are nondispersive, they are ideal for spallation sources and for polychromatic beam experiments on reactor sources. Theoretical calculations indicate that nested mirrors can preserve source brilliance at the sample for small beams and for modest divergences that are appropriate for diffraction experiments. Here we describe the design, calibration and performance of a nested neutron mirror pair for the Spallation Neutrons At Pressure (SNAP) beamline at the SNS. This is an example of a general class of experiments that can benefit from spatially-resolved diffraction inside environmental chambers.

Energy Conservation in Metals Extraction and Materials Processing II: Extraction Processes/ Refractories/Modeling and Analysis

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Energy Committee

Program Organizers: Edgar Vidal, Brush Wellman, Inc.; Cynthia Belt, Aleris International Inc; Marie Kistler, Air Products and Chemicals, Inc; Mark Cooksey, CSIRO; Rob Hardin, Burner Dynamics, Inc.

Monday AM — Room: 2012
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Edgar Vidal, Brush Wellman, Inc.; Mark Cooksey, CSIRO

8:30 AM Introductory Comments

8:35 AM

Energy and Sustainable Development in Hydrometallurgy - An Emerging Perspective: *Katragadda Sarveswara Rao*[1]; [1]Institute of Minerals and Materials Technology (CSIR)

Any innovative approach made in non-ferrous metallurgical industry centers upon developing new processes, saving materials, improving production quality and born-again materials. Aqueous processing is commonly used to treat lean grade or more complex ore bodies. Its sustenance largely depends on energy and environmental compliance including water and wastewater management. The present paper focuses the importance of using particle size distribution and surface area measurements during ammoniacal dissolution of a Cu-Pb-Zn sulphide bulk concentrate and with an overview of the effect of water salinity on the process chemistry and residue mineralogy. Accordingly, an effort is made here to discuss the emerging perspective and highlight the recent successes and trends in terms of energy savings in non-ferrous hydrometallurgy.

8:55 AM

Copper Electrowinning Using Noble Metal Oxide Coated Titanium - Based Bipolar Electrodes: *Krishnasamy Asokan*[1]; Kandasamy Subramanian[1]; [1]Central Electro Chemical Research Institute

Electro winning of copper by monopolar cells require common anode and cathode bus bar and cross bars to connect each electrode to the respective common bus bar. On the other hand, the bipolar configuration warrants the end electrodes only to be connected to bus bars; there is substantial reduction in copper requirement. In the present work, an electro winning cell with bipolar electrodes and end electrodes, each having an area of 1000 cm2 was designed and operated. The bipolar electrode is made of mixed metal oxide coated titanium mesh welded to plain titanium sheet. Coated titanium mesh acts as anode and the plain titanium sheet acts as cathode. Environmentally unacceptable lead anode and the recurring loss of lead are done away with. Closer spacing of the electrodes, paves way for the application of higher current density leading to mass transfer enhancement. Performance of bipolar copper electro winning cell is reported.

9:15 AM

Microbial Reduction of Lateritic Nickel Ore for Enhanced Recovery of Nickel and Cobalt Through Bio-Hydrometallurgical Route: *L. Sukla*[1]; N. Pradhan[1]; R.K. Mohapatra[1]; B.K. Mohapatra[1]; B.D. Nayak[1]; B.K. Mishra[1]; [1]Institute of Mineral and Material Technology

The chromite overburden sample of Sukinda, India is lateritic, where valuable metals like Ni (1%) and Co (0.04%) are bound within goethite (a-FeOOH) phase. To recover these metals an ecofriendly biohydrometallurgical route has been developed.An anaerobic dissimilatory iron (III) reducing bacterial consortium capable of using acetate as carbon source and lateritic ore as terminal electron acceptor was used for reducing iron hydroxide to iron oxide. The initial light brown colour of the ore changed to dark brown colour. The change in colour is due to the conversion of goethite to hematite/magnetite which was confirmed by XRD and EPMA. The treated sample when subjected to bio/acid leaching, higher recovery of Ni and Co was obtained. Thus it is possible to make use of dissimilatory iron reducing bacteria for recovery of valuable metals from low grade iron ore.

9:35 AM Break

9:55 AM

Energy Saving Strategies for the Use of Refractory Materials in Molten Material Contact: *James Hemrick*[1]; Klaus-Markus Peters[2]; John Damiano[3]; James Keiser[1]; [1]Oak Ridge National Laboratory; [2]Fireline TCON, Inc.; [3]MINTEQ International, Inc.

This paper will present work performed by Oak Ridge National Laboratory (ORNL), in collaboration with industrial refractory manufacturers and users, to employ novel refractory systems and techniques to reduce energy consumption of molten material processing vessels found in industries such as aluminum, glass and pulp and paper. Energy savings discussed will be achieved through reduction of chemical reactions, mechanical degradation by the service environment, temperature limitations of materials, and costly installation and repair needs. Key results of several case studies resulting from Department of Energy (DOE) funded research programs will be discussed with emphasis on applicability of these results to all high temperature processing industries.

10:15 AM

Energy Savings through Phosphate-Bonded Refractory Materials: *Jens Decker*[1]; [1]Stellar Materials

In consideration of energy savings the ideal refractory furnace lining should possess the following features:- Lowest thermal conductivity possible in order to avoid heat loss. - Single component lining in order to allow freeze plane changes caused by wear, infiltration and temperature changes of the furnace. - Resistance against mechanical wear from cleaning tools and stirring in order to allow maximum output without equipment downtime due to maintenance. - Resistance against thermo-chemical attack from aluminum and alloying elements. However, refractory materials with a low thermal conductivity typically possess a higher porosity and this leads to lower strengths and resistance against chemical attack and wear. Hence, as a compromise, multi layer linings are required in order to meet energy saving standards. In this paper we present

the features of chemically phosphate-bonded dense and light weight refractories and how such refractories can contribute to energy savings.

10:35 AM
Advanced Ceramic Composites for Improved Thermal Management in Molten Aluminum Applications: *Klaus-Markus Peters*[1]; Robert Cravens[2]; James Hemrick[3]; [1]Fireline TCON Inc; [2]Rex Materials Group; [3]Oak Ridge National Laboratory

Degradation of refractories in molten aluminum applications leads to energy inefficiencies, both in terms of increased energy consumption during use as well as due to frequent and premature production shutdowns. Therefore, the ability to enhance and extend the performance of refractory systems will improve the energy efficiency through out the service life. TCON® ceramic composite materials are being produced via a collaboration between Fireline TCON, Inc. and Rex Materials Group; these materials were found to be extremely resistant to erosion and corrosion by molten aluminum alloys during an evaluation funded by the U.S. Department of Energy and it was concluded that they positively impact the performance of refractory systems. These findings were subsequently verified by field tests. Data will be presented on how TCON® shapes are used to significantly improve the thermal management of molten aluminum contact applications and extend the performance of such refractory systems.

10:55 AM
Energy Efficient, Non-Wetting, Microporous Refractory Material for Molten Aluminum Contact Applications: *Kenneth McGowan*[1]; [1]Westmoreland Advanced Materials, LLC

Result of a R&D effort addressing the need for energy efficient refractories serving the aluminum industry is the development of patented microporous refractory materials for molten metal contact applications in all furnace areas including the belly band. It has been demonstrated that this material can reduce energy consumption up to 38% in reverb and holding furnaces and significantly reduce heat loss in transfer ladles, launders and trough systems. Furthermore, the material developed is non-wetting and remains un-penetrated by metal throughout its lifetime. The material does not contribute to the formation of corundum. As a result, the energy efficiency of an older lining remains intact compared to standard refractory materials which may have an initial high insulating value (such as lightweights and board) or standard dense refractory (even with penetration inhibitors) both of which show a rapid increase in thermal conductivity as the refractory is penetrated with aluminum and formed corundum.

11:15 AM
CFD Modeling for Optimization of Aluminum Melting Furnace Design Parameters: *Mohamed Hassan Ali*[1]; Zhengdong Long[1]; Shridas Ningileri[1]; Subodh Das[2]; [1]University of Kentucky; [2]Phinix LLC

In order to enhance the energy efficiency of aluminum melting furnace, several models using computational fluid dynamics (CFD) have been developed. The models can be used to answer questions as to: how can the melting cycle be shortened; how does geometry and shape affect melting efficiency; what is the best shape and location of the flue gas exit as well as burners; how does the flow circulate; and how does the temperature distribute within the furnace space. The results will show the flow and temperature contours within the furnace combustion space as well as the metal load. The models will take into account the temperature dependency of the load's thermal properties, furnace burner design configuration in addition to the load shape and lining properties. The model will be validated by using real measurements from a base model then followed by a CFD parametric study.

11:35 AM
A Study of Exergy Analysis for Combustion in Direct Fired Heater: *Ahmed Abd Elrahman*[1]; [1]Egyptalum

Heat transfer with organic media has often been able to replace or improve the classic steam-water operation. The possibility of transferring and closely controlling temperature up to > 300ºC has provided the heat transfer media technology with many new fields of application. The growing application of heat transfer plants with liquid heat transfer media other than water has made it necessary to produce complete and accurate engineering database for combustion and his devices to continuous improvement of industrial heating. Heating is an important operation in almost all industrial fields. The analysis of related combustion process and estimation of the effective coefficients is the first step toward a successful design. The process of combustion, fuel and their combustion and combustion devices are considered in this study, direct fired heater Exergy and energy analysis are performed taking into account precisely calculation of chemical Exergy for products of combustion.

11:55 AM Concluding Comments

Fatigue: Mechanisms, Theory, Experiments and Industry Practice: Characterization Methods for Elucidating Fatigue Mechanisms

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee

Program Organizers: Koenraad Janssens, Paul Scherrer Institute; Corbett Battaile, Sandia National Laboratories; Brad Boyce, Sandia National Laboratories; Luke Brewer, Sandia National Laboratories

Monday AM — Room: 3008
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Luke Brewer, Sandia National Laboratories

8:30 AM Invited
Elastic Strain and Dislocation Distributions near Fatigue Cracks Measured Using High Resolution Electron Back Scatter Diffraction: *Angus Wilkinson*[1]; [1]University of Oxford

Over the past few years there has been a significant advance in the sensitivity of EBSD measurements so that the technique now allows determination of the elastic strain tensor and small angle rotations to a level of ~10^{-4}. In this presentation the basis of the technique will be briefly described before turning attention to applications in analysis of fatigued metals. Images and linescans taken near the tips and wakes of fatigue cracks in single crystal superalloy samples will be presented. Lattice curvature shows the extent of the plastic zone and quantiative analysis using Nye's dislocation tensor allows the geometrically necessary dislocation content to be assessed. Elastic strains (ie stresses) are also measured. Stresses normal to the crack plane are observed to be compressive within an inner reversed plasticty zone, while weaker tensile stresses are seen in an outer zone.

9:00 AM
3D Characterisation of Short Cracks in Ti-6246 Using X-Ray Tomography and EBSD: *Soran Birosca*[1]; [1]The University of Manchester

In the present study, crack propagation was imaged non-destructively in 3 dimensions during in-situ fatigue loading of Ti-6246 using X-ray micro-tomography on beamline ID19 at the European Synchrotron Radiation Facility (ESRF), Grenoble, France. Phase contrast enabled the visualization of the two-phase microstructure but in order to obtain the crystallographic orientation of individual grains along the crack path a 3D EBSD volume was recorded subsequently. By combining both techniques it was possible to relate the crystallographic orientation of grains to crack arrest and accelerated crack propagation. It is shown that the lamellar grain orientation and morphology have a great influence on the crack direction and growth. Moreover, crack resistance property of the alloy is investigated by means of EBSD grain characterisation methodology, orientation and misorientation data evaluations.

9:20 AM
An Electron Microscope Study of Low-Cycle Fatigue in a High Niobium Containing and Precipitation Hardend TiAl Alloy: *Fritz Appel*[1]; Thomas Heckel[2]; Hans-Jürgen Christ[2]; [1]GKSS Research Centre Geesthacht; [2]Universität Siegen

The micromechanisms controlling low cycle fatigue of a Nb-bearing TiAl alloy (TNB-V2) have been characterized by conventional and high-resolution transmission electron microscopy. Fully reversed isothermal tests were performed under strain control at temperatures of 25, 550 and 850°C. Samples fatigued at 25 and 550°C exhibited dense structures of ordinary dislocations and debris that were accumulated in tangles. The dipole defects apparently serve as additional glide obstacles but may also contribute to dislocation multiplication if the local stress rises. In situ heating experiments have been performed in order to assess the thermal stability of the dipole defects. Another important low temperature

deformation mechanism is the stress-induced transformation of an orthorhombic phase, which is a significant constituent of the microstructure. The orthorhombic phase is apparently unstable under tetragonal distortion and transforms into γ phase. Under high-temperature fatigue the lamellar microstructure degrades by phase transformation combined with dynamic recrystallization.

9:40 AM
Acoustic Effects on Cyclic-Tension Fatigue of Al-4Cu-1Mg Alloy by Ultrasonic Shear Wave Methods: *Hideki Yamagishi*[1]; Mikio Fukuhara[2]; Akihiko Chiba[2]; [1]Toyama Industrial Technology Center; [2]Tohoku University

Cyclic-tension fatigue of aluminum alloy, Al-4Cu-1Mg, has been determined by usage of SV wave reflection and SH wave transmission methods in terms of nondestructive evaluation. Internal friction measured by SV method begins to increase rapidly from normalized fatigue ratio of about 0.5, showing dominating interaction of movable dislocations with the waves, as viscoelastic effect. Logarithmic damping ratio and propagation time in SH method decrease with increase of the fatigue degree due to acoustoelastic effect. According to SH wave flux model that SH wave energy shifts to specimen surface under crystal-lattice distortion by tensile load, the cyclic-tension induced residual-stress shift which correlates to the decreases in the damping ratio and the propagation time. These effects will provide an accurate and useful tool for nondestructive evaluation of fatigue of the alloy.

10:00 AM
Measuring Micromechanical Behavior for Polycrystalline Materials Under Cyclic Loading: Jun-Sang Park[1]; *Matt Miller*[1]; Alexander Kazimirov[1]; Ulrich Lienert[2]; [1]Cornell University; [2]Advanced Photon Source

Understanding the crack initiation and propagation mechanisms of a polycrystalline material under cyclic loading remains a challenging problem. Complicated crystal stresses arising from single crystal anisotropy and complex grain and phase morphologies make the prediction of crack initiation and propagation in the grain size scale difficult. In this work, oxygen free high conductivity copper specimens were cyclically loaded while x-ray diffraction experiments were performed to find the orientation-wise crystal stresses. It was found that the evolution of the crystal stress distribution over orientation space with respect to specimen life is small but not negligible. The peak widths associated with the dislocation density and the distribution of elastic strain in a material also showed small changes with respect to specimen life indicating changes in the grain size scale.

10:20 AM Break

10:50 AM Invited
Image-Based Modeling of Crack Growth in Particle Reinforced Composites: *Nikhilesh Chawla*[1]; [1]Arizona State University

The fatigue crack growth behavior of particle reinforced composites is determined by several factors, such as reinforcement volume fraction, size, and morphology. Because crack growth is significantly influenced by the morphology and spatial distribution of the reinforcement particles, it is important to adequately characterize the microstructure in simulations of crack growth. In this talk, the results of image-based simulations of crack growth in SiC particle reinforced Al matrix composites, both in two- and three-dimensions, will be described. In particular, the effect of SiC particle distribution and morphology on crack growth was studied. In addition, the effect of particle fracture on crack growth was also studied. Particle fracture ahead of the crack tip significantly alters the crack trajectory and the stress intensity at the crack-tip. Finally, it will be shown that these simulations, encompassing actual microstructures, provide an excellent basis for explaining experimental observations of crack growth in this system.

11:20 AM
Observations of Fatigue Crack Initiation in 7075-T651: John Papazian[1]; Robert Christ[1]; Joel Payne; Greg Welsh[2]; *Joel Payne*[3]; [1]Northrop Grumman; [2]United Technologies; [3]Toho Tenax

Detailed microstructural and crystallographic information on fatigue crack initiation and early stage propagation was required as part of a larger effort to model and predict the remaining life of aircraft. Commercial aluminum alloys were the materials of primary interest. In these materials, fatigue cracking is generally associated with constituent particles. Therefore, the purpose of the current study was to use direct observation (scanning electron microscopy) to establish the exact nature and timing of crack initiation in 7075-T651 with particular reference to the issues of cracking or debonding of the constituent particles, when cracking first occurs during the fatigue process, the influence of matrix orientation on the process, the transition from a constituent particle crack to a matrix crack, and the early stage propagation of short cracks. A double edge notch specimen was designed that was small enough to be imaged in the SEM yet large enough to replicate the fastener hole geometry in actual aircraft structure. Interrupted fatigue cycling starting with fractional initial cycles (20, 40, 60 and 80% of the eventual constant amplitude fatigue load) and continuing on with 1, 3, 10, 30, etc. cycles until failure. At each interval, the crack initiation and propagation process was documented for approximately 100 constituent particles. Orientation Imaging Microscopy was used to document the crystallography of the surrounding grains. The results provide a quantitative description of the fatigue crack initiation process and document the essential characteristics of the process.

11:40 AM
Effects of Microstructure on the Kinematics of Fatigue Crack Propagation in Ti-6Al-4V: Thomas Villarreal[1]; Rikki Teale[1]; *Pedro Peralta*[1]; [1]Arizona State University

Opening strain fields ahead of fatigue cracks in Ti-6Al-4V were studied for two different microstructures to investigate their effects on the kinematics of fatigue crack growth. The tests were performed on standard Compact Tension (CT) specimens at constant values of ΔK and load ratio (0.1), and the microstructure along the crack path was characterized via Electron backscattering diffraction (EBSD). In-situ loading and Digital Image Correlation (DIC) software were used to derive opening strain fields beyond the crack tip. The strain fields will be correlated to crack growth rates and the microstructure around the crack tip and will also be compared to lattice rotations obtained via EBSD. The lattice rotation, as an indirect measure of strain, will also be studied at the half thickness of the samples to investigate constraint effects. Results will assist in constructing a model of fatigue crack growth at the microscale that accounts for microstructural effects.

12:00 PM
In-Situ Investigation of Residual Stresses around Cracks in Hydrided Zircaloy SENT Specimen: *Axel Steuwer*[1]; John Daniels[2]; [1]ESS Scandinavia; [2]ESRF

Using high-energy synchrotron X-ray diffraction on ID15B at the ESRF, Grenoble, we investigated the residual stresses around a fatigue crack grown in 600ppm zircaloy SENT specimen in-situ. The diffraction patterns clearly reveal the matrix as well as the hydride diffraction peaks, allowing phase-specific strain information to be collected at different levels of load. The results as well as the general capabilities of the technique will be discussed.

Friction Stir Welding and Processing-V: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee
Program Organizers: Rajiv Mishra, Missouri University of Science and Technology; Thomas Lienert, Los Alamos National Laboratory; Murray Mahoney, formerly with Rockwell Scientific

Monday AM — Room: 2014
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Rajiv Mishra, Missouri University of Science and Technology

8:30 AM Introductory Comments

8:35 AM Invited
Microstructure – Processing Relationships in Friction Stir Processing (FSP) of NiAl Bronze: *Terry McNelley*[1]; Srinivasan Swaminathan[2]; Jianqing Su[1]; Sarath Menon[1]; [1]Naval Postgraduate School; [2]GE Global Research

The use of FSP for localized modification of microstructure and properties in large cast NiAl bronze components is envisioned to reduce costs and improve component service performance. As-processed stir zone (SZ) microstructures reflect transients and gradients in strain, strain rate and temperature although SZ strength and ductility values are both typically enhanced relative to as-cast properties. The evolution of SZ and thermomechanically affected zone (TMAZ) microstructures during single-pass and multi-pass FSP by rectangular and spiral raster processes will be summarized. Microstructures produced by

thermomechanical simulations will be compared to those produced during FSP. Current models for recrystallization need to be modified to include the transients and gradients in FSP in order to account for the exceptional refinement of microstructure and enhancement of mechanical properties associated with this process.

8:55 AM Invited

Advancements in FSW of Hard Metals: *Jeff Bernath*[1]; Nate Ames[1]; Brian Thompson[1]; Timothy Stotler[1]; [1]EWI

Friction Stir Welding (FSW) is a solid state joining process originally developed and applied on soft metals such as aluminum. As the technology has matured, much of the recent research has shifted to FSW of hard metals. Novel advancements have been achieved in FSW of hard metals including steels, titanium, and nickel based alloys. Developments have been made to improve process robustness, tool life, and microstructure. Improvements to tool materials have allowed welding of increased thicknesses of hard metals using conventional and bobbin methods. New tool geometries have been designed through finite element analysis of the FSW process. Developments in process control mechanisms have provided improved methods for microstructural control of the stir zone. These recent advancements have provided an overall improvement to the capabilities and process robustness of FSW of hard metals. A summary of the advancements to date and application the technology will be discussed.

9:15 AM Invited

Microstructural Evolution during Friction Stir Welding of Near-Alpha Titanium: *Richard Fonda*[1]; Keith Knipling[2]; [1]Naval Research Laboratory; [2]Naval Research Lab

The microstructural evolution, and the deformation mechanisms that give rise to that evolution, have been analyzed in friction stir welds of a near-alpha titanium alloy, Ti-5111. In particular, this presentation will describe the base plate microstructure, how that microstructure evolves as it becomes influenced by the deformation field surrounding the tool, and what further evolutions occur as this material is deposited in the wake of the tool and cooled to ambient temperature to produce the microstructure observed in the deposited weld.

9:35 AM

Electron Backscatter Diffraction Study of Cast and Friction Stir Processed Ti-6Al-4V: *Adam Pilchak*[1]; James Williams[1]; [1]Ohio State University

Electron backscatter diffraction has been used to characterize texture in the stir zone (SZ) of investment cast and friction stir processed Ti-6Al-4V. While the maximum intensities in the orientation distributions are low compared to conventional metal working processes, simple shear textures are present in both the bcc β phase and hcp α phase. The orientation of the shear plane normal and shear direction changed as a function of position in the SZ. These observations provide insight into the strain fields that accompany this complicated deformation process. In material processed above the β transus, the SZ texture was correlated to a continuous dynamic recrystallization texture observed during hot torsion of interstitial-free steel and α-Fe. Recrystallization of the coarse colony structure in sub β-transus processed material was also investigated. The mechanism appears to be based on continuous recrystallization processes where subgrain boundaries gradually evolve into high angle boundaries with increasing dislocation density.

9:55 AM

Physical Simulation of Friction Stir Processed Ti-5111: *Melissa Rubal*[1]; John Lippold[1]; Mary Juhas[1]; [1]Ohio State University

Friction stir processing (FSP) of Ti-5111 was performed above and below the beta-transus temperature, allowing for investigation of the microstructural evolution in both conditions. Each processed panel was instrumented with thermocouples to record the thermal histories in the stir zone and adjacent heat-affected zone. Single sensor differential thermal analysis (SS-DTA) was used to determine the beta transus during processing. The FSP microstructures were characterized using light and scanning electron microscopy, while the microtextures of the FSP regions were compared using electron backscatter diffraction (EBSD). FSP produced extreme grain refinement in both processing conditions – reducing the 200-500 micron base material grains to 1-20 microns. The microstructures observed in the FSP panels were simulated using a Gleeble 3800. The strain and strain rate data may be used to verify FSP modeling programs of titanium to reduce the parameter selection phases of future friction stir projects.

10:15 AM

Thermal Stir Welding High Melting Temperature Materials: *Joseph Querin*[1]; Judy Schneider[1]; Christopher Kolb[2]; Ray Walker[2]; Bryant Walker[2]; Robert Ding[3]; [1]Mississippi State University; [2]Keystone Synergistic Enterprises, Inc.; [3]National Aeronautics and Space Administration

Thermal stir welding (TSWing) developed by the National Aeronautical and Space Administration's (NASA) Marshall Space Flight Center (MSFC) is a solid state joining technique similar to friction stir welding (FSWing). However, unlike FSWing, the heating, stirring, and forging elements of the process are decoupled allowing independent, dynamic control of each process element. With the separation of heating, stirring, and forging elements during the joining process there are more degrees of freedom allowing greater process control. In this study the thermal stir welding (TSWing) process was used to join ½ in thick commercially pure titanium in a butt joint configuration. Metallographic samples have been mounted, polished, and analyzed using optical microscopy to document the microstructure.

10:35 AM Break

10:45 AM Invited

Fatigue Crack Growth in Friction Stir Welded Ti-5111: *Peter Pao*[1]; Richard Fonda[1]; Harry Jones[1]; C.R. Feng[1]; D.W. Moon[1]; [1]Naval Research Laboratory

The effects of weld microstructure and weld speed on the fatigue crack growth kinetics of friction stir welded Ti-5111 were investigated. The FSW weld consists of very fine recrystallized grains, in contrast to coarse basketweave grains in the base metal. The fatigue crack growth rates are significantly lower and fatigue crack growth thresholds are significantly higher through the weld than those in the base metal. As the weld speed increases, the fatigue crack growth rates are progressively higher and fatigue crack growth thresholds lower through the weld. However, after stress-relief annealing, such differences in fatigue crack growth kinetics among different weld speeds no longer exist. Fatigue crack growth rates through post stress-relieved welds are slightly higher than those in the base metal. The observed fatigue crack growth responses are discussed in terms of differences in crack tip microstructure, compressive residual stress distribution, and crack closure.

11:05 AM Invited

Speed and Feed Effects on the Surface Texture and Superplastic Forming Performance of Titanium 6Al-4V Friction Stir Welds: *Daniel Sanders*[1]; M. Ramulu[2]; Paul Edwards[1]; Anthony Reynolds[3]; Glenn Grant[4]; [1]Boeing; [2]University of Washington; [3]University of South Carolina; [4]Pacific Northwest National Laboratory

The purpose of this study was to investigate the speed and feed effects of the Friction Stir Welding (FSW) process on the surface texture along the top of a butt welded nugget. The test was conducted using fine grain (0.8 to 2u) titanium alloy 6Al-4V with a thickness of 2.5 mm. Through additional development of the FSW process parameters, the butt welded nugget was also made to have equivalent superplastic forming (SPF) characteristics as the parent sheet material. By using special cooling techniques, the weld zone can be kept below the beta transus temperature, which enables the formation of a grain structure conducive to superplastic behavior.

11:25 AM Invited

Faster Temperature Response and Repeatable Power Input to Aid Automatic Control of Friction Stir Welded Copper Canisters: *Lars Cederqvist*[1]; [1]SKB

The Swedish Nuclear Fuel and Waste Management Company will join at least 12,000 lids to the extruded copper tubes containing Sweden's nuclear waste. To ensure that high quality welds are produced repeatedly, the need of an automated welding procedure controlling the tool temperature instead of the current procedure depending on a skilled welding operator is evident. The reliability of the automatic procedure is however limited by the time lag in the temperature responding to changes in heat input. Currently, the tool temperature takes 15-20 seconds to respond to heat input changes. New thermocouple placements have proved that the response time can be reduced to 5-10 seconds. The paper discusses how the shorter response time aid the development of the automatic procedure and how, due to the lag, PID-control algorithms using both tool temperature and heat input are used to automatically control the tool temperature within its process window.

11:45 AM
Investigating the Effects of Pin Tool Design on Friction Stir Welded Ti-6Al-4V: *Haley Rubisoff*[1]; Joseph Querin[1]; Judy Schneider[1]; [1]Mississippi State University

Friction stir welding (FSWing), a solid state joining technique, uses a non-consumable rotating pin tool to thermomechanically join materials. Heating of the weldment caused by friction and deformation is a function of the interaction between the pin tool and the work piece. Therefore, the geometry of the pin tool is in part responsible for the resulting microstructure and mechanical properties. In this study microwave sintered tungsten carbide (WC) pin tools with tapers and flats were used to FSW Ti-6Al-4V. Transverse sections of welds were mechanically tested, and the microstructure was characterized using optical microscopy (OM) and scanning election microscopy (SEM). X-ray diffraction (XRD) and electron back-scatter diffraction (EBSD) were used to characterize the texture within the welds produced from the different pin tool designs.

12:05 PM
The Effect of Friction Stir Processing on the Microstructural Evolution and Mechanical Properties of Ti-6Al-4V Alloy: *Nilesh Kumar*[1]; Jeffrey Rodelas[1]; Rajiv Mishra[1]; [1]Missouri University of Science and Technology

Friction stir processing (FSP) was applied to Ti-6Al-4V alloy to modify the microstructure and improve the mechanical properties. Experiments were carried out at three different tool rotational rates – 1200, 1000 and 800 rpm. Other parameters (traverse speed, tilt angle, etc) were kept unchanged. The material processed at 800 rpm showed very narrow HAZ. Hardness in the nugget region was higher than the parent material in each case. In case of sample processed at 1200 rpm, an improvement of approximately 33% in YS and UTS over as-received material was observed. It was 36% and 27% in the case of samples processed at 1000 rpm and 800 rpm, respectively. The best strength after FSP was 1236 MPa as compared to 910 MPa in as received material. This improvement in strengths was observed with no compromise in the ductility of the material (25% elongation for parent and 23-27% elongation for FSPed samples).

12:25 PM
Texture and Microstructural Evolution during the Linear Friction Welding of Ti-6Al-4V: *Elvi Dalgard*[1]; John Jonas[1]; Mohammad Jahazi[1]; [1]McGill University

The linear friction welding behavior of Ti-6Al-4V was investigated using various processing conditions of frequency (30-70 Hz), pressure (30-70 MPa) and shortening (2-3 mm). The strain and flow stress during LFW for each set of welding parameters was estimated based on known properties and behavior of the material during hot deformation. LFW samples were examined using electron backscatter diffraction (EBSD) to relate the texture and variant selection behavior to the strain and flow stress. Characterization of the welds includes analysis of the microstructure of the weld and thermomechanically affected zones (TMAZ) in relation to the parent material. Prior studies have shown that in the weld region, exposure to temperatures above the beta transus (995°C), combined with deformation and rapid cooling after joining, produced a Widmanstätten alpha-beta transformation microstructure. The relationship of the transformed structure to the prior grains was examined using EBSD and electron microscopy.

12:45 PM
Thermohydrogen Processed Friction Stir Welding: *Yuri Hovanski*[1]; [1]PNNL

Thermohydrogen processing parameters were developed to temporarily modify the properties of titanium sheet into conditions uniquely applicable to joining via friction stir welding (FSW). Modifications of mechanical properties and phase kinetics of commercially pure titanium that was temporarily alloyed with hydrogen created beneficial changes in the processability of the metal during FSW. Significant reductions in both the plunge forces required to seat a tool as well as the transverse loading during the traverse were demonstrated in temporary alloyed titanium sheet. In order to exhibit the increased workability of the thermohydrogen processed sheet, tests were conducted using conventional tool materials used for FSW titanium as well as lower cost materials more typical of FSW aluminum alloys. Hydrogen was successfully removed with a post processing vacuum anneal.

Frontiers in Solidification Science III: Fundamentals of Solidification: Interfaces, Nucleation, Growth, and Nonequilibrium Considerations

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Phase Transformations Committee, TMS: Solidification Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Ralph Napolitano, Iowa State University; James Morris, Oak Ridge National Laboratory

Monday AM — Room: 2018
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Christoph Beckermann, University of Iowa

8:30 AM Invited
Modeling Wetting and Nucleation: Some Recent Surprises: *James Warren*[1]; Daniel Wheeler[1]; Laszlo Granasy[2]; Tamas Pusztai[3]; William Boettinger[1]; [1]National Institute of Standards and Technology; [2]Brunel University; [3]RISSPO

The analysis a solid nuclei wetting and/or reacting with an impurity in a melt provides the basis for classical models of nucleation. Conversely, models of a liquid melt dissolving into/reacting with a solid substrate provide insight into phenomena as diverse a VLS growth and soldering. Developing a thermodynamically consistant picture of such phenomena forces a reconsideration of a number of classical assumptions. The notions of contact angle, phaseboundaries, surface energies, as well as a number of kinetic phenomena most all be reevaluated in the context of these new models. In this talk I will explore several phase field models of wetting and spreading and explore some of the new metrics that might provide better predictive power in understanding these systems.

8:50 AM Invited
Non-Equilibrium Solidification of Undercooled Melts of Al-Based Alloys: *Dieter Herlach*[1]; Helena Hartmann[2]; Peter Galenko[1]; Dirk Holland-Moritz[1]; [1]German Aerospace Center; [2]Ruhr-University Bochum

Electromagnetic levitation is utilized to containerlessly undercool drops of metallic melts. High-speed camera technique is employed to measure dendrite growth velocities as a function of undercooling. Significant changes of the temperature dependence of the growth dynamics are observed when solute trapping and/or disorder trapping are taking place during solidification of deeply undercooled melts far from equilibrium. The experimental data are analyzed within an extended sharp interface theory of dendrite growth. The results of the measurements of growth velocity are quantitatively described over the entire undercooling range of $\Delta T \leq 350$ K accessible by levitation experiments. The primary solidification of disordered superlattice structures of intermetallics at undercoolings exceeding a critical value for the onset of disorder trapping is confirmed by in situ energy dispersive X-ray scattering on levitation processed samples using high intensity synchrotron radiation at the European Synchrotron Radiation Facility in Grenoble.

9:10 AM
Critically Comparing Molecular Dynamics Simulations of Nucleation with Theory: *James Morris*[1]; Lujian Peng[2]; Rachel Aga[3]; [1]Oak Ridge National Laboratory; [2]University of Tennessee; [3]Wright State University, Department of Chemistry

We have performed critical examinations of nucleation in molecular dynamics simulations. The Lennard-Jones system and an EAM model of Al were used; in both cases, quantities that affect nucleation, particularly the solid-liquid interfacial free energy, were calculated separately, for a parameter-free comparison with theory. At higher temperatures, a reasonable comparison with classical nucleation theory is obtained, but transient effects must be accounted for. Simulation sizes affect the distribution of nucleation times. At low temperatures, the Lennard-Jones system transforms rapidly, suggesting a low barrier; however, in contrast to recent reports, there is no evidence of a spinodal at undercoolings near T/T_m=0.67. The Al system always shows a measurable time to nucleation, with a minimum near T/T_m=0.45, demonstrating that fast nucleation is not generic. This research has been sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract DE-AC05-00OR-22725 with UT-Battelle.

9:30 AM
Morphology Evolution and Solidification Kinetics in 2D: A Phase-Field Crystal Study: *Gyorgy Tegze*[1]; Laszlo Granasy[1]; [1]Brunel University

Using the phase-field crystal model, we address the evolution of complex solidification morphologies and the solid-liquid transformation kinetics on the atomistic scale. In single component systems, we observe a diffusion controlled growth mechanism at low supersaturations, which switches to an interface controlled mechanism at high supersaturations, a behavior reminiscent to that seen in colloidal systems. We present a morphology map that contains transitions between compact and dendritic structures and polycrystalline growth forms. Next, we use a recent model of Elder et al. (2007) to investigate morphological transitions in a binary system of ~1.6 million atoms, and determine morphological aspects of dendritic solidification including the variation of tip radius and velocity as a function of time. Finally, we address transformation kinetics of polycrystalline solidification in single component and binary systems, and compare the respective behaviors of the Avrami-Kolmogorov exponent describing the time evolution of freezing.

9:50 AM
The Microstructural Evolution of Impulse Atomized Al-Fe Powder: Jian Chen[1]; *Hani Henein*[1]; [1]University of Alberta

The microstructure and metastable phases in three compositions of Al-xFe (x=0.61, 1.90 and 7.98 in wt pct) droplets prepared by impulse atomization were studied by transmission electron microscopy (TEM). For Al-0.61Fe, the droplets exhibits microstructure of dendritic/cellular alpha-Al with eutectic AlmFe/alpha-Al (m=4.0-4.4) precipitated at the dendritic/cellular wall. The non-equilibrium condition incorporated by the impulse atomization shifts hypoeutectic Al-1.90Fe to hypereutectic composition, thus it produces a similar microstructure as demonstrated in Al-0.61Fe. For Al-7.98Fe, the microstructure is more complicated comparing with those of Al-0.61Fe and Al-1.90Fe. Metastable primary AlmFe, stable Al13Fe4 with blade morphology, eutectic Al6Fe/alpha-Al and alpha-Al coexist in the microstructure in Al-7.98Fe. Based on the above results undercooling conditions are predicted and the solidification path of phases in Al-7.98Fe is proposed.

10:10 AM Break

10:30 AM Invited
A Molecular Dynamics Simulation Study of Solute Trapping during Rapid Solidification: *Jeffrey Hoyt*[1]; Y. Yang[2]; H. Humadi[1]; D. Buta[2]; M. Asta[2]; D.Y. Sun[3]; [1]McMaster University; [2]University of California, Davis; [3]East China Normal University

It is well known that the partitioning of solute in the solid phase increases above its equilibrium value at high solidification rates, yet very few experiments have successfully measured the relationship between the segregation coefficient and the growth velocity. In this work molecular dynamics simulations of solute trapping have been performed on a model Lennard-Jones binary alloy and the Ni-Cu system modeled with the embedded atom method. The velocity dependent segregation coefficient, as a function of driving force and crystallographic growth direction, is compared with the Kaplan and Aziz continuous growth model and the results provide estimates for the diffusive speed, V_D, which reflects the interplay between solid-liquid interface motion and atomic transport across the interface. In addition, we also compare the results to more recent sharp interface models.

10:50 AM Invited
Molecular Dynamics Investigations of Faceted Growth at the Nanoscale: Tomorr Haxhimali[1]; Dorel Buta[1]; *Mark Asta*[2]; Peter Voorhees[3]; Jeff Hoyt[4]; [1]University of California; [2]University of California; [3]Northwestern University; [4]McMaster University

We present results of atomistic simulations investigating mechanisms at the solid-liquid interface underlying faceted solidification in a geometry mimicking the vapor-liquid-solid nanowire growth. These simulations employ a model potential for pure Si, with the driving force for growth applied by undercooling. The simulations yield an equilibrium solid-liquid interface shape that is non-planar, with a faceted orientation bounded by curved orientations near the solid-liquid-vacuum contact line. The curved portions lead to a capillary undercooling which increases in magnitude with decreasing nanowire diameter. In growth simulations, the interface shape is preserved. Growth is observed to proceed in a layer-by-layer mode with a rate limited by the nucleation of new (111) terraces. For a given driving force, measured as the undercooling below the capillary-corrected coexistence temperature, the growth rates are observed to increase with decreasing nanowire diameter. These results are interpreted to reflect a size dependence of the barrier for terrace nucleation.

11:10 AM Invited
Phase-Field Modelling of Liquid Crystal Solidification: *Mathis Plapp*[1]; Jesper Mellenthin[1]; Hervé Henry[1]; [1]Ecole Polytechnique

Some years ago, the nematic-isotropic transition that occurs in liquid crystals has been used as an analog for solidification. Directional "solidification" of liquid crystal alloys can be used to investigate cellular patterns for parameter regimes that are difficult to attain in experiments on metals. However, liquid crystals also exhibit a non-conventional interfacial anisotropy due to the presence of the nematic director field, the effect of which has not been studied so far. We present a phase-field model that describes the nematic ordering by a tensorial order parameter, which naturally includes the bulk dynamics of the director field and the proper anchoring condition at the nematic-isotropic interface. Numerical simulations reveal that the coupling to the director field strongly influences the linear stability of a planar front, and the shape and stability of well-developed cells. The relation of our findings to the known results for anisotropic crystals are discussed.

11:30 AM
How Do Quasicrystals Grow?: *Aaron Keys*[1]; Sharon Glotzer[1]; [1]University of Michigan

Using molecular simulations, we show that the aperiodic growth of quasicrystals from the liquid state is controlled by the ability of the growing quasicrystal nucleus to incorporate kinetically trapped atoms into the solid phase with minimal rearrangement. In the system under investigation, which forms a dodecagonal quasicrystal on cooling from a high temperature liquid, we show that this process occurs through the assimilation of stable icosahedral clusters by the growing quasicrystal. Our results demonstrate how local atomic interactions give rise to the long-range aperiodicity of quasicrystals. References: A.S. Keys and S.C. Glotzer, Phys. Rev. Lett. 99, 235503 (2007). P.J. Steinhardt, Nature 452, 43 (2008).

11:50 AM
Solidification Behavior of Tin on Quasicrystalline Surfaces: *Alok Singh*[1]; Hidetoshi Somekawa[1]; An Pang Tsai[2]; [1]National Institute for Materials Science; [2]Tohoku University

Solidification behavior of tin on quasicrystalline surfaces has been studied by embedding one micron size tin particles in Al-Cu-Fe icosahedral phase by rapid solidification followed by annealing. Another annealing treatment was carried out to obtain a microcrystalline matrix. Particle-matrix interfaces were studied by TEM, while solidification studies were carried out by DSC as well as in-situ in TEM. Tin made faceted interfaces with the icosahedral phase, matching close packed planes in various orientations. Prominent solidification peaks occurred on cooling in DSC. In the microcrystalline matrix there was a single exothermic peak, but multiple peaks occurred in the quasicrystalline matrix at a similar lever of undercooling. Three major peaks were distinguished, whose relative heights were dependent on the cooling rate. Contact angle of the solid nucleus were estimated to be 11° in the microcrystalline matrix, and 9.5°, 11° and 14° in the quasicrystalline matrix. Role of interface structures is analyzed.

12:10 PM
Effect of the Shear Flow on Morphological Stability during Directional Solidification: *Zidong Wang*[1]; [1]McGill University

The effect of a shear flow on the planar solidification process has been considered for the hypercooled pure melt. In the basic steady state solution for the flow field, there is a boundary layer for the case of small Prandtl number. A linear stability analysis shows that the morphological stability of the interface is modified by the shear flow. There are two traveling waves for the flow field along the interface paralleling to the shear flow, the solidification allows the oscillatory decay mode solution for the temperature and flow fields. As the shear flow increases, the minimum wave number that makes the flow field stable increases, the range of stability becomes smaller, then the shear flow is a destabilizing factor in this problem. If the shear flow vanishes, there is no oscillatory mode for the system, which gives the growth rate of crystal as a function of the shear flow.

General Abstracts: Extraction and Processing Division: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Energy Committee, TMS: Hydro and Electrometallurgy Committee, TMS: Materials Characterization Committee, TMS: Process Technology and Modeling Committee, TMS: Pyrometallurgy Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizer: Boyd Davis, Kingston Process Metallurgy

Monday AM Room: 2005
February 16, 2009 Location: Moscone West Convention Center

Session Chair: Elli Miettinen, Outotec Oyj

8:30 AM
Comparative Study of Cyanide and Acid Leaching of Gold and Silver from Deer Trail Mine Oxide Ore and Tailings: *Edgar Blanco*[1]; Charlie Madsen[1]; Michael Moats[2]; [1]UNICO Deer Trail Mine; [2]University of Utah

At the Deer Trail Mine, interest in recovering noble metals from ore tailings with 1.0 g/t Au and 200 g/t Ag has led to metallurgic studies focused on finding an adequate treatment for this material. Using cyanide leaching, the silver/gold recoveries from oxide ore and sulfide tailings were 53%/95% and 30%/76%, respectively. Since these recoveries were less than desired for silver, it was decided to explore a non-cyanide leaching process that would extract silver and gold better. Based on previous research the possibility of using aqua regia in combination with sulfuric acid was evaluated. On a laboratory scale, variables such as particle size, concentration of reagents, dissolved oxygen, reaction time, pH and temperature were evaluated. Under optimal conditions it was possible to achieve >80% silver recovery and >90% gold recovery for both oxide ore and tailings.

8:50 AM
Comparison of Solvent Extraction Studies on Tetravalent Platinum from Acidic Chloride Solutions Using Tri-Octyl/Decyl Amine and Bis(2,4,4-Trimethylpentyl) Monothiophosphinic Acid: *Rajesh Kumar Jyothi*[1]; [1]Korea Institute of Geoscience and Mineral Resources (KIGAM)

The extraction equilibrium study of tetravalent platinum was carried out using tri-octyl/decyl amine (Alamine 336) and bis(2,4,4-trimethylpentyl) monothiophosphinic acid (Cyanex 302) in kerosene from hydrochloric acid media to investigate their extraction capacity, since they have different donor atoms, 'N' and 'S'. Their distribution equlibria were studied as a function of acid, extractant, diluents and temperature. The title metal shows the inverse behavior at higher acid concentrations. Extraction of tetravalent platinum increases with increase of extractant concentration. The plot of log D vs. log [Extractant], mol.L-1 is linear with slopes 1±0.3, indicating the association of one mole of extractant with the extracted metal species. Stripping of metal from the loaded organic (LO) with mineral acids and bases such as hydrochloric, sulphuric, nitric acids and ammonia, hydrogen peroxide, sodium hydroxide, thio- urea were studied. Regeneration and recycling capacity of Alamine 336/Cyanex 302 and extraction behavior of associated elements was also studied.

9:10 AM
Treatment of Produced Water by Electrocoagulation: *Jewel Gomes*[1]; David Cocke[1]; Kamol Das[1]; Mallikarjuna Guttula[1]; Doanh Tran[1]; Jim Beckman[2]; [1]Lamar University; [2]Kaselco

Produced water (PW) is salty water trapped in the reservoir rock and brought up along with oil or gas during production. It subsists under high pressures and temperatures, and usually contains hydrocarbons and metals. Therefore, it must be treated before being discharged to surface water. Different techniques are being used to treat PW through phase separations, system control and design, and chemical treatments. In this paper, we discuss our experimental results on treating PW through electrocoagulation (EC). The performance of EC was investigated for the reduction of chemical oxygen demand (COD) and metal ions. Effects of different electrodes, residence time, current density, and pH were also studied to optimize the treatment conditions. Different kinds of cleansing agents, such as lime and borax were used to break the buffering effect encountered during treatment. FTIR, SEM/EDS, and XRD were used to characterize the EC-floc and thus to elucidate removal mechanisms.

9:30 AM
Mathemathical Modeling of Particle Suspension in Pachuca Tanks: Esperanza Rodriguez M.[1]; *Alfonso Castillejos*[1]; Francisco Acosta G.[1]; [1]CINVESTAV - Unidad Saltillo

The efficient behavior of Pachuca tanks as hydrometallurgical reactors is strongly linked to the suspension of the mineral particles, which results from the motion of the liquid caused by the injected gas rising, in general, through a central draft tube. This study reports a computational investigation carried out to determine the effect of operating and design parameters on the suspension of particles. By extending the classical drift-flux model to compute the gas hold-up, the three-phase (water-air-particles) system was simulated as a two-phase system formed by a variable density-liquid plus the solid particles. The two phases were treated as interpenetrated continua using an Eulerian approach to set a turbulent recirculating flow model in 2-D and transient state. The model predicted adequately the measured critical gas superficial velocity needed for complete particle suspension in pulps with 10-50 wt% solids. Additionally, the model was used to investigate the performance of industrial size reactors.

9:50 AM
The Theoretical Calculation and Validation of Burden Trajectory in Blast Furnace of Bell-less Top: *Yu Yaowei*[1]; Bai Chenguang[1]; Zhang Zhengrong[1]; Wang Feng[1]; Lv Daguang[1]; [1]Chongqing University

Blast furnace ironmaking is a main method by which iron is efficiently reduced from iron-bearing materials with CO. Charging is one of primary systems for blast furnace control. The trajectory of materials is an important parameter in determining impact point where falling materials intersect with the stockline profile in the charging. In order to clarify the trajectory of materials and validate theoretical calculated trajectory, a 1/15 scale cold model of an actual 2500m3 shaft and bell-less top charging system has been built. The results indicate that the measured trajectory is consistent with the theoretical one.

10:10 AM Break

10:30 AM
Principle and Practice of Producing Qualified Antimony White from Lead-Antimony Alloy by Blowing Directly: *Liu Weifeng*[1]; Yang Tianzu[1]; Xia Wentang[2]; Liu Wei[1]; Huang Chao[1]; [1]Central South University; [2]Chongqing University of Science and Technology

On the basis of the thermodynamic calculation of lead, antimony and arsenic about oxidating volatilization in high temperature, the possibility of separating lead,antimony and arsenic in different temperature was analyzed, and the practice of qualified antimony white through direct blowing from lead-antimony alloy was introduced in detail in this paper. The blast furnance reduction smelting was brief introduced firstly, and then alkaline refining was applied to removing arsenic from lead-antimony alloy that contains As1%,Sb39%,Pb58%, the arsenic in the treated lead-antimony alloy could be decreased to 0.008% by that method.The antimony white containing Sb2O3 99.8% could be produced by air blowing in the special furnace at 650° from the treated lead-antimony alloy. And the electrolytic lead could be achieved by electrolysis from the lead bullion which contained 83% lead.

10:50 AM
Reduction Roasting Study of Greek Nickeliferous Laterites: Emmanuel Zevgolis[1]; *Charalabos Zografidis*[1]; Iliana Halikia[1]; Eamonn Devlin[2]; [1]NTUA; [2]NCSR Demokritos

The reduction roasting experimental study of Greek nickeliferous laterite samples with a gaseous reducing mixture – CO:N2- is presented in the present work. The effect of parameters such as temperature, ore grain size and composition of the reductive mixture on the result of reduction, were examined. It is deduced that the reducibility of intermediate type laterite sample, where iron appears mainly in form of goethite, is considerably higher than that of limonitic type laterite samples, where hematite is the predominant iron mineral phase. Increase of temperature within the range of 750-900°C, unlike decrease of the ore grain size and more intensive reductive conditions, does not favor considerably reduction. Metallic iron phase co-exists with magnetite and hematite in all the reduced samples. Considerable increase of the specific surface area of the intermediate laterite ore type after calcination due to goethite dehydroxylation, can be regarded as critical parameter for its higher reducibility.

11:10 AM

Thallium Extraction from Liquid Pb-Tl Solution: *Piotr Kapias*[1]; [1]The Silesian University of Technology

The theoretical part of the paper presents the analysis of the conditions of Tl extraction from liquid Pb using different extractants: PbCl2, ZnCl2 or Cl2(g). The analysis and evaluation were made of the influence of the methods of extractants introduction into a Pb-Tl solution upon the efficiency of the Tl extraction process under thermodynamic equilibrium conditions. The analysis made it possible to formulate relevant equations describing the process of Tl extraction from Pb under static as well as under the extractant stirring and injection into the liquid Pb-Tl solution conditions. In the part concerning experimental work, results are given for tests on the Tl extraction process from Pb-Tl liquid solutions of initial Tl content of approximately 0.05% mass or 0.02% mass using PbCl2, ZnCl2 or Cl2(g) conducted in laboratorial scale. The idea of a reactor for thallium extraction from lead has been presented.

11:30 AM

Thermal Conductivity and Characterisation of Copper Flash Smelting Flue Dust: *Elli Miettinen*[1]; [1]Outotec Oyj

In Copper Flash Smelting operation disturbances may lead into dust build-up in the off-gas handling system causing reduced heat recovery efficiency and process availability. Flue dust of a Copper Flash Smelting process heat recovery boiler has been characterized and its thermal conductivity and diffusivity have been determined. Dust accretions consist of several layers possessing varying particle sizes, densities, and thermal properties, with the binding phase mostly being sulphate. All studied samples can be regarded as effective thermal insulators with thermal conductivity values of less than 2 W/mK and thermal diffusivity values of less than 0.005 cm2/s. Porosity can be regarded as a fairly good indicator of the thermal transport efficiency of these types of materials, but the material microstructure must also be considered. The results can be used in the scaling of metallurgical heat recovery boilers and they also provide accurate input data for process models.

11:50 AM

Study on Health Risk Assessment of POPs from a Copper Smelt Enterprise: *He Dewen*[1]; Du Lu[1]; Wang Wei-lian[1]; Liang Ding-ming[1]; [1]Central South University

The distribution of persistent organic pollutants in the environment is studied using the multimedia environmental fugacity model, and environmental health risk assessment of POPs nearby a copper smelt enterprise is carried out. The result indicates that all are in an agreeable risk level without exception. The annual individual chronic risk index of HCB is 7.50×10-11. The annual individual risk index of 3,3',4,4'-TCB is 1.57×10-9, and the annual individual risk index of 2,3,7,8-TCDD is 6.57×10-9.

12:10 PM

Performance on Leaching of Antimony Trioxide with Polyhydric Organics in Alkaline Solutions: *Wei Liu*[1]; Tianzu Yang[1]; Duchao Zhang[1]; Xing Xia[1]; Weifeng Liu[1]; [1]Central South University

Antimony trioxide was leaching in alkaline solutions containing polyhydric organics (glycerol, xylitol and tartaric acid). Experimental based on orthogonal array table (L9) was utilized to determine the optimum leaching conditions. The influences of concentrations of polyhydric organics and sodium hydroxide on the leaching efficiency were studied in detail. The reaction mechanism was also discussed. The experimental results show that the antimony trioxide could be dissolved easily in alkaline solutions containing polyhydric organics. The concentrations of polyhydric organics and sodium hydroxide have significant effect upon the leaching efficiency, while the leaching temperature and time have few effects. The leaching rate arises with increasing concentrations of polyhydric organics and sodium hydroxide. The resultant may be Sb-Na double alkoxides. The formation of the resultant could be restrained by the reactivity of hydroxyl. Under the same conditions, the react ability of antimony trioxide with glycerol is weaker than that with xylitol and tartaric acid.

General Abstracts: Structural Materials Division: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Composite Materials Committee, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee, TMS: Refractory Metals Committee, TMS: Titanium Committee

Program Organizers: Robert Hanrahan, National Nuclear Security Administration; Eric Ott, GE Aviation

Monday AM — Room: 2010
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

8:30 AM

A Methodology for Non Destructive Evaluation of Dwell Fatigue Susceptibility of a Near Alpha Titanium Alloy: *Amit Bhattacharjee*[1]; S.I. Rokhlin[1]; Andy Woodfield[2]; J. C. Williams[1]; [1]Ohio State University; [2]General Electric

Acoustic wave attenuation has been measured in ultrasonic range for specimens cut from a series of Ti6242 forgings. Large variation has been observed. Orientation imaging microscopy scans also was carried out using electron backscatter diffraction technique to determine the degree of microtexture in the forgings. The correlation between the microtexture and the attenuation will be presented. These data suggest that acoustic attenuation can be used to non-destructively assess the types and degree of microtexture in Ti alloy forgings. This technique has the potential to assess the degree of microtexture in existing forgings that have been processed by various routes prior to the recognition of the importance of microtexture. It has been recognized for some time that microtexture in titanium alloys leads to a debit in dwell fatigue life. Therefore, the outlined procedure may be useful to non-destructively assess the dwell sensitivity of existing titanium alloy hardware of varying processing history.

8:50 AM

Brittle Compressive Failure: Transition from Splitting to Faulting in Ice: *Erland Schulson*[1]; Carl Renshaw[1]; Luke Wachter[1]; [1]Dartmouth College

When loaded under compression, ice and other Coulombic materials fail by axial splitting when unconfined and by shear faulting when moderately confined. The question is: how moderate is moderate? We show from systematic experiments on columnar-grained fresh-water ice biaxially loaded across the columns that the transition from one mode to the other occurs continuously, but rapidly: once the ratio of the minor to major stress reaches R=0.01, it is complete. Moderate is thus very small indeed—so small that from a practical perspective splitting is of little importance to either ice-structure interactions or other geophysical/engineering situations. Ceramics and rock are expected to exhibit the same behavior. The transition can be understood in terms of the growth of wing cracks and the stress field ahead of them. A quantitative model will be presented.

9:10 AM

Effects of Tantalum on the Phase Decomposition of a Model Ni-Al-Cr Superalloy on a Nanoscale: *Christopher Booth-Morrison*[1]; Ronald Noebe[2]; David Seidman[1]; [1]Northwestern University; [2]NASA Glenn Research Center

The effects of a 2.0 at.% addition of Ta to a model Ni-10.0 Al-8.5 Cr at.% superalloy are assessed by atom-probe tomography. The $\gamma(L1_2)$-precipitate morphology that develops as a result of γ(f.c.c.)-matrix phase decomposition at 1073 K is found to evolve from a bimodal distribution of spheroidal precipitates, to {001}-faceted cudoids and parallelepipeds aligned along the elastically soft <001>-type directions. The phase compositions and the widths of the γ-precipitate/γ-matrix interfaces evolve temporally as the Ni-Al-Cr-Ta alloy undergoes quasi-stationary state coarsening after 1 h of aging. Tantalum is observed to suppress the mobility of Ni in the γ-matrix sufficiently to cause an accumulation of Ni on the γ-matrix side of the γ/γ interface. Computational modeling employing Thermo-Calc, Dictra and PrecipiCalc, elucidates the kinetic pathways that lead to phase decomposition in this concentrated Ni-Al-Cr-Ta alloy.

9:30 AM
Method for Determining Dislocation Viscous Drag Coefficients: *John Gilman*[1]; [1]University of California

In imperfect crystals, dislocation motion is of the stick/slip type so fundamental viscosity coefficients are difficult to measure. However, the maximum velocities are determined by a balance between the driving stress, and the drag. The driving stress is limited by cohesion so the drag can be determined at the terminal velocity where the motion is of the slip type alone. Fortunately, there is a reliable equation for extrapolating from intermediate velocities to the terminal velocity. Given the terminal velocity, an expression without disposable parameters yields the viscosity coefficient. The latter can be compared with values measured directly at low stresses, or derived from internal friction measurements. This method will allow values for a wide range of materials to be made and a library to be constructed that will be useful for fundamental studies.

9:50 AM
Life Prediction and Reconstruction of Creep Curves Based on an Evaluation of Strain Rate Change in Secondary Creep: *Hiroyuki Sato*[1]; Takaya Miyano[2]; [1]Hirosaki Universiity; [2]Ritsumeikan University

Shape of creep curves and change of strain rate in secondary creep are quantitatively evaluated; furthermore, reconstructions of creep curves are performed by means of extrapolation of strain rate change in secondary stage. We have reported that the behavior of strain rate change in secondary creep depends on the classes of magnesium-based solution strengthened alloys; and have proposed a characteristic parameter that reflects the strain rate change quantitatively. In this report, we show that a reconstructed creep curve based on the proposed parameter fairly agree with experimental creep curves in the alloys; moreover, we propose one method of creep life prediction. It is shown that a combination of the minimum creep rate and the proposed parameter that reflects strain rate change reasonably describe creep curves. The changes in strain rate acceleration reasonably agree with the transition of creep characteristics evaluated by minimum creep rate.

10:10 AM
Mechanical Properties and Phase Stability of Ti-Cr System Alloys: *Yonosuke Murayama*[1]; Shuichi Sasaki[1]; Rajagopalan Srinivasan[2]; Daniel Huber[2]; Hisamichi Kimura[3]; Akihiko Chiba[3]; Hamish Fraser[2]; [1]Mechanical and Control Engineering, Niigata Institute of Technology; [2]Materials Science and Engineering, The Ohio State University; [3]Institute of Material Research, Tohoku University

Low modulus beta titanium alloys are attractive alloys for biomedical application. This work examines the mechanical properties of Ti-Cr-Sn-Zr system alloys. The elastic modulus of the alloy varies with the composition, which variation is caused from the competition between the meta-stable beta phase and the omega phase. The elastic modulus of the alloy decreases very much owing to the addition of alloy element that depresses the omega phase, which is similar to Ti-Nb system alloys. This work focuses on the effect of the varying alloy composition on the microstructure, the elastic modulus, the deformation mechanism and the deformability. The deformation modes of the Ti-Cr-Sn-Zr alloy, which are the mechanical twinning, the deformation by slip and the deformation-induced transformation, change by the composition of the alloy. We discuss the effect of the transition of the deformation modes on the mechanical properties.

10:30 AM
Mode I Penny Shape Crack in Sandwich Multilayered Composites: *H. Y. (Sean) Yu*[1]; [1]Naval Research Laboratory

The solution of a mode I penny shape crack at the center of a sandwich layered composite is obtained. The sandwich composite consists of any number of layers of homogeneous, isotropic materials. The upper half of the sandwich is the mirror image of the lower half. For example, if the crack is at the center of layer A, then the composite consisting of layers A, B, C, D is with the stacking sequence D/C/B/A/B/C/D. The point force Green's functions for this composites is derived first. The solution of the crack problem is formulated by integrating the Green's function over the crack surface with a given point force distribution. The dual integral equations of the unknown crack surface displacement are established by considering the boundary conditions on the crack surface of the multilayered solid, which can be converted into a Fredholm integral equation of the second kind and solved numerically.

10:50 AM
Physical and Microstuctural Characterization of TiC Reinforced Al-Cu Matrix Alloy Composites: *Hülya Kaftelen*[1]; Necip Ünlü[1]; Lütfi Öveçoglu[1]; Hani Henein[2]; [1]Istanbul Technical University; [2]University of Alberta

Aluminum - 4 wt. % copper composites containing 5-20 vol. % of TiC powders in 2 µm size were fabricated using K-Al-F type flux-assisted conventional casting method. The resulting morphologies and compositions of the composites were characterized as a function of TiC volume fraction by optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction analysis (XRD) and microhardness tests. The microstructural investigations exhibited the reasonably homogeneous distribution of the carbides in Al4Cu matrix alloys. The XRD results of the composite samples revealed that the phases of the Al, Al2Cu and TiC were detected. In addition, the intensity of the diffraction peaks belonging to the TiC phase gradually increased with increasing of the volume fraction of the reinforcement. Increasing of the volume percent of TiC particles from 5 through 20% contributed to increase in hardness values of Al-Cu based composites.

11:10 AM
The Dynamic Strength of a Representative Double Layer Prismatic Core: A Combined Experimental, Numerical and Analytical Assessment: *Enrico Ferri*[1]; Tony Evans[1]; Vikram Deshpande[1]; [1]University of California, Santa Barbara

Dynamic out-of-plane compressive testing is used to characterize the dynamic strength of metal prismatic cores with double layer topology. The dynamic strength was evaluated by measuring the stresses transmitted to a Hopkinson pressure bar impacted at constant velocities up to 140 m/s. 2D plane strain, FE calculations successfully predicted the experimental results with appropriately calibrated imperfections. To infer the response of this core when included in a sandwich plate subject to blast loading, the finite element model was modified to unsupported (free-standing) back face boundary conditions. The transmitted stress is found to be modulated by the momentum acquired by the back face mass and, as the mass becomes larger, the core strength approaches that measured and simulated for stationary conditions. An analytical model that accounts for the shock effects in a homogenized core is presented and shown to capture the observations and simulated results with acceptable fidelity.

Global Innovations in Photovoltaics and Thermoelectrics: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Energy Committee, TMS: Global Innovations Committee
Program Organizers: Sivaraman Guruswamy, University of Utah; Joy Forsmark, Ford Motor Co; John Smugeresky, Sandia National Laboratories

Monday AM — Room: 3005
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Sivaraman Guruswamy, University of Utah; Narsingh Singh, Northrop Grumman Corp ES; Joy Forsmark, Ford Motor Co

8:30 AM Keynote
Recent Advances in Thermoelectric Power Generation Materials, Technology and Terrestrial Application Opportunities: *Jean-Pierre Fleurial*[1]; [1]Jet Propulsion Laboratory/California Institute of Technology

Thermoelectric power sources have consistently demonstrated their extraordinary reliability and longevity for deep space missions (67 missions to date, more than 30 years of life) as well as terrestrial applications where unattended operation in remote locations is required. The development of new, more efficient materials and devices is the key to improving existing space power technology and expanding the range of terrestrial applications. The Jet Propulsion Laboratory is leading collaborative research and development on novel advanced bulk materials capable of long term operation at temperatures up to 1300 K at more than 20% conversion efficiency. The research areas include refractory rare earth compounds and bulk 3-D nanostructures that emulate results obtained on low dimensional superlattices through "force engineering" and "self-assembling" techniques. Recent experimental results will be highlighted, and progress in transitioning thermoelectric technology to a more flexible, lower cost modular array configuration suitable for various application opportunities will be discussed.

9:15 AM

Melt Spinning and Spark Plasma Sintering for Manufacturing of Highly Textured Thermoelectric Materials: *Juergen Schmidt*[1]; Dirk Ebling[2]; Alexandre Jacquot[2]; Harald Boettner[2]; Thomas Weissgaerber[1]; Bernd Kieback[1]; [1]Fraunhofer Institute for Manufacturing and Applied Materials Sciences IFAM; [2]Fraunhofer Institute for Physical Measurement Techniques IPM

V-VI thermoelectric compounds are well known for room temperature applications like Peltier coolers. The anisotropic physical properties andthe mechanical weakness of the crystals are a problem for the manufacturing. Polycrystalline bismuth telluride based n- and p-type thermoelectric materials were fabricated through Spark Plasma Sintering (SPS) technique. The combination of a rapidly solidified alloy and temporary liquid phase sintering by SPS allows controlling the texture of microstructure. With this technique sintered, polycrystalline (Bi,Sb)2(Te,Se)3 with ZT values > 1 were produced. This paper will report on the preparation by melt spinning technique and the influence of the SPS process on the texture, the thermoelectric and mechanical properties of the polycrystalline materials.

9:35 AM Invited

Morphology of High Temperature Boron-Based Thermoelectric Materials: *Takao Mori*[1]; [1]National Institute for Materials Science (NIMS)

The useful energy conversion of waste heat is a huge incentive to find viable thermoelectric materials. Obviously a particular need exists to develop materials which can function at high temperatures. Boron-rich cluster compounds are attractive materials for their stability under high temperature typically exhibiting melting points above 2200 K. As a synthesis method it has been found that addition of small amounts of third elements like carbon, nitrogen, and silicon can result in the formation of novel and varied rare-earth boron cluster structures. $REB_{44}Si_2$ compounds exhibit Seebeck coefficients greater than 200 μV/K at high temperatures and unlike most compounds, the figure of merit shows a steep increase at T>1000 K. Homologous RE-B-C(N) compounds were recently discovered to be the long awaited n-type counterparts to p-type boron carbide. The focus of the talk will be on the control of morphology of these materials in relation to their high temperature thermoelectric properties.

10:00 AM

The Formation of Aligned Ag_2Te Precipitates in $AgSbTe_2$: *Joshua Sugar*[1]; Douglas Medlin[1]; [1]Materials Physics Department, Sandia National Laboratories, Livermore

The thermoelectric alloy $AgSbTe_2$ is a relatively simple alloy with a ZT as high as 1.3 at 720 K. It is also a primary constituent in the more complicated, high-performance $(AgSbTe_2)_{1-x}(GeTe)_{1-x}$ and $(AgSbTe_2)_{1-x}(PbTe)_x$ systems. The high ZT of these more complicated alloys is generally attributed to compositional heterogeneities in their microstructure, which have been inferred to contribute different mechanisms to the interfacial scattering of electrons and phonons. $AgSbTe_2$ provides a good system for understanding the types of compositional inhomogeneities that one could expect in similar thermoelectric alloys. This study investigates the decomposition of $AgSbTe_2$ into crystallographically aligned precipitates of monoclinic Ag_2Te in a matrix of cubic $Ag_{22}Sb_{28}Te_{50}$. The precipitate formation is energetically easy because of the topotactic alignment of the high-temperature Ag_2Te phase. Below 145°C, cubic Ag_2Te undergoes a displacive transformation to a monoclinic structure, which creates several symmetric orientation relationship variants and complicates the diffraction analysis.

10:20 AM Break

10:30 AM Invited

Effect of Growth Parameters on the Quality of PbSe Nanocubes and Nanodots: *Narsingh Singh*[1]; Eric Jones[1]; E. Jelen[1]; B. Wagner[1]; S. Mc Laughlin[1]; A. Berghmans[1]; D. Kahler[1]; D. Knuteson[1]; [1]Northrop Grumman Corporation

The concept is based on utilizing peaks in the solar spectrum irradiance to design materials which have bandgap very closed to those wavelengths. These can be grown as quantum Dot (QD) materials to increase quantum yield which could increase efficiency due to a quantum effect called multiple exciton generation. We developed a series of lead selenide (PbSe) detector materials with different characteristics. We observed that substrate temperature and purity has pronounced effect on the morphology, resistivity and crystallinity. As the substrate temperature changed, the crystal orientation changed from (111) to (001) orientation. We grew very good quality film with full width of maxima of 0.3 degree. The virgin material showed 60.7 K Ohm-cm and annealed sample showed a resistivity value of 5.0 M Ohm.cm.

10:55 AM

Towards Predicting Reaction Pathways in the Cu-In-Se-Ga System: *Carelyn Campbell*[1]; [1]National Institute of Standards and Technology

To reduce the production costs of CIGS (α-$CuIn_xGa_{1-x}Se_2$) photovoltaic cells, the processing time must be reduced from approximately 30 minutes to less than 2 minutes. This challenge requires finding new reaction pathways in the Cu-In-Se-Ga system to increase the synthesis rate of the CIGS absorber material. The complex chemistry of the CIGS system has limited efficient exploration of potential processing sequences. Combining CALPHAD-based thermodynamics and diffusion mobilities descriptions enables prediction of reaction pathways for prospective processing sequences. Preliminary diffusion mobility descriptions for the Cu-In-Ga-Se system, based on previously developed thermodynamic descriptions, will be presented. These mobility descriptions are derived from both measured unary, binary and ternary tracer, intrinsic and chemical diffusion data and experimentally derived activation energies. The diffusion mobility descriptions are then used to simulate a wide range of model reactions. The fundamentals of the approach and the simulations will be discussed.

11:15 AM

Using Patterned Si Thin Foils to Build 3-D Photovoltaic Devices: Xiaoying Guo[1]; Huan Li[1]; *Jimmy Hsia*[1]; Ralph Nuzzo[1]; [1]University of Illinois at Urbana-Champaign

Unlike flat panel photovoltaic systems, three dimensional photovoltaic devices present a promising way to harvest solar energy efficiently without requiring additional apparatus to adjust the orientation of the device. In this research, we developed a technique to fabricate 3-D photovoltaic devices using patterned Si thin foils, which make use of the self-assembly process driven by capillary forces. One key requirement for this technique, however, is the condition under which folding of the patterned thin foil occurs. A mechanics model based on the theory of thin plate has been developed to identify this critical condition. The model is capable of predicting the critical condition for thin foil folding for complicated foil shapes. Our experimental measurements of the foil folding condition agree with the model predictions beautifully. Furthermore, an intrinsic, non-dimensional material parameter has been identified in the model to be the single parameter controlling the foil folding process.

11:35 AM

Preparation of Metallic Precursor of Cu(In, Ga)Se2 Thin Film for Solar Cell Applications by Sputtering Alloyed Cu-In-Ga Target: *Yaojun Lin*[1]; Paul Gilman[1]; [1]Praxair Electronics

As solar energy materials, Cu(In, Ga)Se2 (CIGS) thin film is attracting considerable interest due to long-term stability and highest conversion efficiency. Among various techniques to prepare metallic precursors of CIGS thin film, sputter techniques are the most promising since they can be easily scaled up and facilitate roll to roll production on flexible substrate. This presentation reports preparation of metallic precursor of CIGS by sputtering alloyed Cu-In-Ga target, which is advantageous over co-sputtering of elemental targets in substantial compositional uniformity, and successive sputtering of elemental targets due to reduction in production time. Compositional uniformity, microstructure and phases in metallic precursor, which have dominant effects on CIGS properties, are studied in detail using EDS, XRD and SEM.The approaches to optimize compositional uniformity and microstructure of metallic precursor are discussed, including an improvement in alloyed target's metallurgical quality and modification of sputter parameters.

11:55 AM

Effect of Electric Current Assisted Thermal Treatment on Thermoelectric Properties of Bi-Sb-Te and Bi-Se-Te Based Thin Films Prepared by Sputtering: *Kuen-Ming Liou*[1]; Chien-Neng Liao[1]; Hsu-Shen Chu[1]; [1]National Tsing-Hua University/Department of Materials Science and Engineering

In this study a novel approach of electric current assisted thermal treatment for improving thermoelectric properties of sputtered Bi-Sb-Te and Bi-Se-Te films is presented. Both electrical conductivity and Seebeck coefficient of the sputtered Bi-Sb-Te and Bi-Se-Te films were enhanced by introducing a high density of electric current through the films during thermal annealing. The electrically stressed films were found to have lower carrier concentration but much higher mobility than the films that were only thermally annealed at the same temperatures. An electromigration induced defect elimination model is proposed to explain the observed electrical transport properties and microstructure evolution of the electrically stressed thin films. The study shall lead to an effective strategy of improving thermoelectric properties of the thermoelectric films by electric current stressing.

Magnesium Technology 2009: Magnesium Town Hall Meeting - A Decade of Modern Magnesium in China

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Monday AM Room: 2007
February 16, 2009 Location: Moscone West Convention Center

Session Chair: Eric Nyberg, Pacific Northwest National Laboratory

8:30 AM Introductory Comments by Eric Nyberg

8:35 AM
Overview of Advanced Magnesium Alloy Development, Forming, Welding and Corrosion Protection in IMR: *En-Hou Han*[1]; [1]Chinese Academy of Sciences

Due to the remarkable increase of energy price and the decrease of the source of the raw structural materials such as steel, aluminum, magnesium and its alloys become more and more popular in last decade. The primary magnesium production in China now becomes dominant in the world. In the meanwhile, China promotes the development and application of magnesium alloys. Institute of Metal Research (IMR) developed various advanced magnesium alloys, such as magnesium-lithium alloys, high strength high ductility cast-alloys, and high strength high toughness wrought alloys. IMR also developed various processing techniques, such as ultrasonic grain refinement, ECAP, friction stirring welding, etc. Especially various corrosion protection techniques, such as chemical conversion coatings, micro-arc oxidation, anodizing (MAO), electroless plating, electroplating, were developed. At last, future need for magnesium alloys development and application was proposed.

8:55 AM
Global Magnesium Market Fundamentals: *Susan Slade*[1]; [1]US Magnesium LLC

The supply and demand balance in the global magnesium market continues to be dynamic based on the changing business environment in China. Magnesium production in China supplies over 80% of global demand, creating a situation in which even minor changes in the Chinesemagnesium industry can have a significant impact on all markets. Factors affecting ingot supply and magnesium demand growth will be reviewed. Analyses of global supply and demand forecasts will provide an outlook for the future.

9:15 AM
Recent Developments in the Chinese Magnesium Industry: *Liming Peng*[1]; [1]Shanghai Jiaotong University, China

Abstract not available.

9:35 AM Question and Answer Period

10:00 AM Break

Magnesium Technology 2009: Alloys I: Rare Earth (Gadolinium, Neodymium)

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Monday AM Room: 2006
February 16, 2009 Location: Moscone West Convention Center

Session Chairs: Liming Peng, Shanghai Jiaotong University; Karl Kainer, GKSS Research Center

10:15 AM Introductory Comments

10:20 AM
Characterization of Dynamic Strain Ageing in Mg-3.11wt.%Gd Alloy: Lei Gao[1]; *Rongshi Chen*[2]; Enhou Han[1]; [1]Institute of Metal Research Chinese Academy of Sciences; [2]Institute of Metal Research, Chinese Academy of Sciences

To elucidate the dynamic strain ageing behavior of Mg-Gd based alloys which were developed as high specific strength and good creep resistant magnesium alloys at elevated temperature, tensile tests were carried out for Mg-3.11wt.%Gd in the temperature range of 25-300°C and in the strain rate range from 1×10^{-4} to 1×10^{-2} s^{-1}. At given strains, stress relaxation (SR) experiments were performed. Serrated flow, negative strain rate sensitivity, and post-relaxation effect were observed in some cases. The post-relaxation effect was sensitive to testing temperature and the strain at which the stress relaxation was performed. The critical strain for the onset of serrated flow was observed to increase with increasing strain rate but decrease with increasing temperature. In addition, activation energy for serrated flow was calculated. The results were analysed in relation to dynamic strain ageing effect (DSA) due to interactions between dislocations and solute Gd atoms.

10:40 AM
Effect of Cold Roll on Microstructure and Mechanical Properties of Mg-8Gd-3Y-0.5Zr Alloy: *Li Dejiang*[1]; Zeng Xiaoqin[1]; Dong Jie[1]; Zhai Chuanquan[1]; [1]Shanghai Jiao Tong University

The simplest TMT process including cold roll with strain of 8%,15%,22% and subsequently aged at different temperatures to peak hardness were carried out to investigate the influence on microstructure and mechanical properties of heat resistant Mg-8Gd-3Y-0.5Zr alloy. The microstructure observation showed that basal plane dislocation sliding and twinnings (including double twinning) were the main deformation mechanisms during cold rolling, the amount of twins were increased with increasing deformation strain. The initial hardness of the alloy specimen was increased with the increasing of strain and the aging time to peak hardness was greatly shortened for the reason of work hardening and acceleration of precipitation from the supersaturated solid solution, respectively. However, the peak hardness value of the deformed and non-deformed alloy specimens remained almost the same. TEM investigation confirmed that the precipitation in the deformed microstructure preference for the equilibrium phase was attributed to lower age hardening response.

11:00 AM
Effects of Heat Treatments on Tensile Properties and Creep Behavior of Mg-Y-Gd-Zr Alloys: Yan Gao[1]; *Qudong Wang*[1]; Jinhai Gu[2]; Yang Zhao[1]; [1]Shanghai Jiaotong University; [2]Hitachi (China) Research & Development Corp., Shanghai Research Institute

We have investigated the microstructure, mechanical properties at room and elevated temperatures (250°C, 300°C), strengthening mechanisms, creep behavior and creep deformation mechanisms of Mg-10Y-5Gd-0.5Zr alloys of in the cast and T6 conditions. The results showed that the tensile properties of the cast-T6 specimen are much higher than that of the as-cast specimen and the creep resistance of the cast-T6 specimen is markedly better than that of the as-cast specimen at both conditions. The creep resistance of the Mg-10Y-5Gd-0.5Zr at T=250, σ=80MPa is markedly better than that at T=300, σ=50MPa. This means the temperature makes more effects on the creep resistance than the stress. Finally, the creep mechanism of the alloy at different condition is further analyzed.

11:20 AM
Microstructure and Mechanical Properties of Hot Extruded Mg-3Nd-0.2Zn-0.4Zr (wt. %) Alloy: Penghuai Fu[1]; *Liming Peng*[1]; [1]National Engineering Research Center of Light Alloy Net Forming, School of Materials Science and Engineering, Shanghai Jiaotong University

The microstructure and mechanical properties of 350, 450 and 525°C hot extruded Mg-3Nd-0.2Zn-0.4Zr (NZ30K) (wt. %) alloys are investigated. The grains are significantly refined by hot extrusion and the extruded alloys show a bimodal grain distribution, in which the finer grains are less than 1μm and the coarser grains are several to ten micrometers in size. The lower extrusion temperature, the finer grains are got. The extruded NZ30K alloys have aging hardening ability. The higher extrusion temperature, the higher aging hardening effect ΔHV (HV peak-aged – HV as-extruded) is obtained. After aging treatment, both yield strength (YS) and ultimate tensile strength (UTS) are significantly improved. The 350°C extruded NZ30K alloy shows the best strength after 200°C peak-age treatment. The YS, UTS and elongation are 290MPa, 317MPa

and 22%, respectively. The extruded alloys show dimple fracture pattern characterized by dimples covered all of fracture surfaces.

11:40 AM
Mechanical Properties and Microstructure of Mg-Zn-Gd Aalloys with Long Period Stacking Ordered Structure: *Michiaki Yamasaki*[1]; Minami Sasaki[1]; Yoshihito Kawamura[1]; [1]Kumamoto University

Rare earth-containing Mg alloys are of interest because of the precipitation reaction that results in age hardening. In general, isothermal aging of supersaturated alpha-Mg solid solutions in these alloys has been performed at about 473 K. The hardness of alloys increases with increasing aging time, and reaches a peak value with beta' phase precipitation and then decreases with beta phase precipitation. Recently, it was found that the addition of Zn to the Mg-Gd alloys brings about the precipitation of the 14H long period stacking ordered (LPSO) structure at more than 623 K. Therefore, we have investigated the aging behavior of the Mg-Zn-Gd (at.%) alloy at temperatures ranging from 473 K to 773 K and propose a TTT diagram for beta', beta-1, beta and 14H-LPSO phase precipitation of the alloys. The relationship between the mechanical properties and the microstructure of the cast and extruded alloy will be discussed.

Magnesium Technology 2009: Fatigue and Tension/ Compression Asymmetry

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Monday AM Room: 2007
February 16, 2009 Location: Moscone West Convention Center

Session Chair: Sean Agnew, University of Virginia

10:15 AM Introductory Comments

10:20 AM
Enhancing In-Plane Fatigue Resistance of Rolled AZ31 Magnesium Alloy by Pre-Straining: Chong Soo Lee[1]; Seong-Gu Hong[1]; *Sung Hyuk Park*[1]; [1]Pohang University of Science and Technology

Rolled AZ31 magnesium alloy has a strong basal texture so that it favors extensive twinning under compressive loading, resulting in a low compressive flow stress compared to a tensile flow stress; this induced a tensile mean stress, which reduced the fatigue resistance during fatigue deformation. The improvement of fatigue resistance was attempted by reducing the developed tensile mean stress, which was achievable by the fact that the lattice reorientation caused by twinning during compressive loading favors detwinning in the twined regions during the subsequent tensile reloading, leading to a significant drop in tensile flow stress. The variation of the twinning-detwinning characteristics was made by pre-compressions of 2, 5, and 8 % and their effect was evaluated at the fully reversed strain amplitude of 1% at room temperature. The enhancement of fatigue resistance was explained in the relation with the amount of pre-compression.

10:40 AM
Influence of Stress Ratio on Fatigue Crack Propagation Behavior of AZ31 Alloy: *KyoSoo Song*[1]; Hwa Chul Jung[1]; Kwang Seon Shin[1]; [1]Seoul National University

Although the resistance to fatigue crack propagation (FCP) is one of the most important design criteria for structural materials, there have been limited studies on FCP behavior of magnesium alloys, particularly in the near-threshold region. In the present study, the influence of stress ratio on FCP behavior of an AZ31 alloy was investigated in conjunction with crack closure phenomena. FCP experiments were carried out under the constant load amplitude at ambient temperature. The ΔK_{th} value of the AZ31 alloy was affected by the change in stress ratio. The ΔK_{th} value decreased with increasing the stress ratio, while the $\Delta K_{th,eff}$ showed almost constant value regardless of the stress ratio. The crack closure levels were high in the near-threshold region and decreased with increasing FCP rates. It was found that the crack closure effect diminished in the high stress ratio region.

11:00 AM
Comparison of Quasistatic and Cyclic Plastic Behaviour of Wrought Magnesium Alloys: *Lenka Fuskova*[1]; Jan Bohlen[1]; Dietmar Letzig[1]; Karl Ulrich Kainer[1]; [1]GKSS Research Centre Geesthacht GmbH

The extrusion of magnesium alloys causes the occurrence of characteristic crystallographic textures and leads to a significant orientation dependence of the mechanical properties, as well as a distinctive tension-compression yield asymmetry. This will also affect the cyclic deformation behaviour of such profiles. The objective of this study is to investigate and compare the deformation behaviour of textured profiles from the magnesium AZ-series during static and cyclic testing. The influence of the loading condition on the microstructure and crystallographic texture evolution during testing will be shown. Fatigue tests are performed under tensile and compressive loading (stress ratio R = 0.05 and R = 8) at low frequency. A relation between the maximum stress during cyclic loading in comparison to the yield strength in tension and compression will be given. Microstructure and texture analysis before and after testing will enable a discussion on fatigue behaviour of textured samples in tension and compression.

11:20 AM
Tension / Compression Test of Mg AZ31B at Elevated Temperature: *Kun Piao*[1]; June Lee[2]; Heon Lim[3]; Robert Wagoner[1]; [1]Department of Materials Science and Engineering, The Ohio State University; [2]Department of Mechanical Engineering, The Ohio State University; [3]Division of Mechanical Engineering & Mechatronics, Kangwon National University

A large-strain tension/compression test for elevated temperature for in-plane continuous testing of sheet metal in the sheet plane has been designed, simulated, optimized, and constructed. Thermal and mechanical analysis was carried out using finite-element method to optimize heating system, particularly material selection and placement of heating cartridges. The result is a device that can attain a temperature of 350ºC within 15 minutes, and can maintain a constant temperature throughout the gage length of specimen within 10ºC. To demonstrate the capabilities of the device, testing of AZ31B Mg sheet was carried out up to 250ºC. The room-temperature asymmetry between tensile and compressive deformation (inflected hardening curve in compression accompanying twinnig) vanished between 125ºC and 150ºC. The mechanical results indicate that dislocation slip instead of twinning deformation dominates the hardening behavior of Mg AZ31B alloy sheet above 150ºC. This conclusion was confirmed by metallography to reveal the presence of twins after testing.

11:40 AM
Comparison of Flow Stress Anisotropy and Tension-Compression Asymmetry in Ultrafine Grained AZ31B and ZK60 Magnesium Alloys: *Majid Al-Maharbi*[1]; David Foley[1]; Ibrahim Karaman[1]; Suveen Mathaudhu[2]; Laszlo Kecskes[2]; [1]Texas A&M University; [2]Army Research Laboratory

Two magnesium alloys, AZ31B and ZK60A, have been processed using Equal Channel Angular Extrusion (ECAE) to enhance their mechanical properties by introducing ultra-fine grained (UFG) structures with grain sizes less than 1 µm. The mechanical flow anisotropy of ECAE processed samples is investigated taking into account the competition between the crystallographic texture and microstructural morphology. The flow stress anisotropy, tension-compression asymmetry, and Bauschinger effect are monitored as a function of number of ECAE passes and processing routes. The anisotropy as well as texture evolution during ECAE is predicted using a visco-plastic self-consistent crystal plasticity model. In this talk, similarities and differences between these two magnesium alloys in UFG form will be presented in terms of aforementioned properties.

Manufacturing Issues in Fuel Cells: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS: Shaping and Forming Committee

Program Organizers: Tsung-Yu Pan, Consultant, Ann Arbor Michigan; John Bradley, General Motors Corp; Michael Miles, Brigham Young University

Monday AM — Room: 3006
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: John Bradley, General Motors Corp

8:30 AM

Failure Mechanism of Nb-Cladded Stainless Steel Sheets under Bending: *Kamran Asim*[1]; Sung-Tae Hong[2]; Scott Weil[3]; William Hosford[1]; Jwo Pan[1]; [1]University of Michigan; [2]University of Ulsan; [3]Pacific Northwest National Laboratory

Niobium (Nb)-cladded 304L stainless steel sheets can potentially be used as bipolar plates in polymer electrolyte membrane (PEM) fuel cells. Mechanical behavior and failure mechanism of Niobium (Nb)-cladded 304L stainless steel sheets were examined. Uniaxial tensile, bend and flattening tests of as-rolled and annealed specimens were conducted. The effects of different annealing temperatures and times on the mechanical behavior and failure mechanism were investigated. A micrographic analysis of bent and flattened specimens showed that the as-rolled specimens have limited ductility. The results also show that the specimens annealed above 900ºC developed a micron thick intermetallic layer. The annealed specimens failed due to the breakage of intermetallic layer and subsequent localized necking failure of Nb layer. The springback angles of these specimens can be correlated to their elastic moduli and the yield strengths of these as-rolled and annealed specimens.

8:55 AM

Failure Mechanism of Polymer-Coated Stainless Steel Sheets under Bending: *Kamran Asim*[1]; Jwo Pan[1]; Daniel Wilkosz[2]; Tsung-Yu Pan[3]; [1]University of Michigan; [2]Ford Motor Company; [3]Consultant, Ann Arbor Michigan

Polymer-coated stainless steel sheets can potentially be used as bipolar plates in polymer electrolyte membrane (PEM) fuel cells. A polymer coating EB-815 was selected in this investigation. EB815-coated 316L stainless steel sheets were examined for their ductility and formability. Uniaxial tensile tests, bend tests and flattening tests were carried out to determine the mechanical behavior and failure mechanism of these sheets under large plastic deformation. EB815 coating failure was observed at about 15% tensile strain in uniaxial tensile tests. A micrographic analysis of bent and flattened specimens showed that these specimens have good ductility under bending. Failure of the polymer coating in bend and flattening tests depends on the bend radius and the amount of bending. The data obtained form these tests will be helpful in future modeling of these sheets for forming fuel channels in bipolar plates.

9:20 AM

Low-Cost High-Volume Production of Fuel Cell Bipolar Plates by Electromagnetic Impact Forming: Steve Hatkevich[1]; *Glenn Daehn*[2]; Shekhar Srinivasan[2]; Jason Johnson[2]; John Bradley[3]; [1]American Trim; [2]Ohio State University; [3]General Motors Corp

Investments from American Trim, GM, The Ohio State University and the State of Ohio Third Frontier Fuel Cell Program are supporting the development of a pilot plant for the manufacture of fuel cell metallic bipolar plates using a process where a magnetic field generated from a Uniform Pressure Actuator upon capacitor discharge accelerates a conductive workpiece sheet uniformly to high velocity. It then impacts a die with the desired shape, and takes the shape of that surface. Several challenges related to the development of a robust manufacturing method from this fundamentally new method are addressed. These will include discussion of design methods and validating experiments for modeling launch efficiency of materials of varied conductivity and the development of methods to qualify, test and assure long-life actuators. The overall economics of this emerging manufacturing process will be quantitatively discussed.

9:45 AM

Formability of Thin Sheet Metals in Impact-Forming of Fuel Cell Bipolar Plates: *Shekhar Srinivasan*[1]; Glenn Daehn[1]; Geoff Taber[1]; John Bradley[2]; Steve Hatkevich[3]; [1]Ohio State University; [2]General Motors Corp; [3]American Trim

Conventional sheet metal stamping is an attractive manufacturing process for metallic fuel cell bipolar plates. However, successful stamping of the plates can be limited by aggressive flow field channel geometries, as well as by plate material properties. High velocity impact forming using a Uniform Pressure Actuator (UPA) with capacitor bank discharge represents an alternative method to manufacture these shapes. Here, plates are formed via high velocity (100-200 m/s) impact against a properly shaped die. This work compares the formability of several candidate high strength materials formed with this process against that obtainable from quasi-static forming. Impact velocities are measured using photon Doppler velocimetry and numerical modeling of the impact forming using LS-DYNA is used to explain the improved forming limits using local failure criteria.

10:10 AM Break

10:25 AM

Manufacturing, Assembling and Testing of Micro- PEM Fuel Cells: *Yuhao Lu*[1]; Alton Highsmith[1]; Ramana Reddy[2]; [1]University of Alabama; [2]University of Alabama

Micro-proton exchange membrane fuel cells (μ-PEMFCs) can provide more than 10 times the energy density of a rechargeable lithium-ion battery. Thus, it has been developed as a promising electrochemical power source. In this study, the microelectromechanical system (MEMS) techology was employed to manufacture the end plates of μ-PEMFCs with micro-channel flow field on the silicon wafers. The μ-PEMFCs were assembled using the end plates and two kinds of membrane electrode assembly (MEA) fabricated at different pressure and temperature. The technologies of polarization and electrochemical impedance spectroscopy (EIS) were used to test the μ-PEMFCs at different conditions. The results in this study demonstrated that the process of fabriction and integration drastically affect the perfermance of the μ-PEMFCs, which provided a clear idea for further designing and optimizing the μ-PEMFCs.

10:50 AM

Processing and Properties of Porous Metallic Sandwiches for Solid-Oxide Fuel Cell Interconnects: *Justin Scott*[1]; John DeFouw[1]; David Dunand[1]; [1]Northwestern University

Weight and cost remain an issue in mobile applications of solid-oxide fuel cells. One approach to mitigate these problems is incorporating porosity in the interconnects. Accordingly, two types of sandwiches with porous faces and a dense core were created. First, E-Brite (Fe-Cr-Mo) sandwiches were fabricated by cold-pressing three layers of elemental powders and subsequent co-sintering. Porosity was formed in the outer layers through a NaCl placeholder, which was mixed with the metallic powders before pressing and removed upon sintering. A similar placeholder technique was used for J5 (Ni-Mo-Cr-Ti-Mn-Al-Y) sandwiches, which were prepared by casting the alloy around a sandwich scaffold consisting of permanent alumina spheres in the core and temporary sodium aluminate in the faces. Following infiltration, the sodium aluminate placeholder was leached out, resulting in porous faces around a syntactic core. Mechanical properties of both types of sandwiches were measured in three-point bending tests and compared to finite-element models.

11:15 AM

Fabrication of Ni/YSZ Anode for SOFC Application by Plasma Spraying: *Yung-Chin Yang*[1]; Yu-Chuan Wu[1]; Yung-Fu Hsu[1]; Yuh-Ruey Wang[1]; Sea-Fue Wang[1]; [1]National Taipei University of Technology

By introducing the pore former into the composite powder, the porous structure of SOFC anode will be obtained by plasma spraying. In this study, bi-feedstock of the pure nickel powder and composite (Na2CO3/YSZ) powder were simultaneously deposited on a stainless substrate. At high temperature of plasma torch, the solid state of Na2CO3 would decompose to release CO2 and then eject the molten powder to induce the interconnected pores in the coatings. After cleaning and soaking in deionized water, the residual Na2CO3 in the coating would dissolve to form the open pores, and the porous YSZ would exist at the inner coating. By varying the size of the composite powder, the porosity of porous coating could be varied from 20 to 40%. These results suggest that the method exhibits the potential to manufacture the porous ceramic/metal composite anode of SOFC to achieve the large three phase boundary for fuel oxidation.

11:40 AM

Fracture of Perfluorosulfonate Polymers for Fuel Cell Proton Exchange Membranes: *Ruiliang Jia*[1]; Ekaterina Kolozhvari[1]; Takuya Hasegawa[2]; Jiping Ye[3]; Reinhold Dauskardt[1]; [1]Stanford University; [2]Nissan Research Center, Nissan Motor Company, Ltd; [3]Research Department, Nissan Arc Ltd.

Perfluorinated sulfonic polymers are widely used as proton exchange membranes in fuel cells as the proton conductor. Fracture of the membrane is a common failure mode that limits the operational life of the cell. Surprisingly, there are no well established test methods to assess fracture properties, particularly under constrained conditions, and little is understood regarding the fracture properties of such polymer membranes in simulated operational environments. In the present work, we examine a number of novel fracture methods to assess the fracture properties of Nafion films under mixed mode loading conditions, and with varying degrees of mechanical constraint. Techniques are adapted from methods used in thin film cohesion and adhesion testing, and are used to reveal the significant effect of simulated operational environments on fracture resistance.

12:05 PM

Novel Technology for Producing Bipolar Plates in Metal Material at High Rate: Katarina Franzén Byttner[1]; *Bill Walczak*[1]; [1]Cell Impact AB

Cell Impact has a patented new developed technology for producing customized, highly detailed bipolar plates in metal material for fuel cells, as well as flow field plates for heat exchangers, using adiabatic forming. An impact with a piston is controlled so that the energy is transmitted into the metal during a few milliseconds. Adiabatic softening occurs at the point where the impact energy is concentrated, making the material receptive to processing. The plate is shaped from both sides at the same time. Flow fields can be oriented in any direction and any shape single or double sided can be produced. Forming takes place in a single rapid operation. The manufacturing rate of one complete plate per second reduces production costs. Production is performed in a production line with an energy level of 12.5kJ. A second production line is designed where the energy level will be increased to 146.2kJ.

Materials for High Temperature Applications: Next Generation Superalloys and Beyond: Future Application Requirements and Next Generation Superalloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS: Refractory Metals Committee
Program Organizers: Joseph Rigney, GE Aviation; Omer Dogan, National Energy Technology Laboratory; Donna Ballard, Air Force Research Laboratory; Shiela Woodard, Pratt & Whitney

Monday AM Room: 3010
February 16, 2009 Location: Moscone West Convention Center

Session Chair: Joseph Rigney, GE Aviation

8:30 AM Invited

Beyond Nickel Based Superalloys: Materials for Advanced Military Engines: *Dallis Hardwick*[1]; David Shifler[2]; [1]US Air Force; [2]Office of Naval Research

Advanced military engines of the future will undoubtedly operate at higher temperatures to meet performance requirements and/or environmental goals. We continue to push the limits of superalloys, both wrought and cast alloys, but other materials systems are also important for future systems. Ceramics, ceramic matrix composites and refractory alloys are all systems that are being investigated. Each of these alternatives has strengths and weaknesses that we continue to explore. We'll discuss the major issues and potential future directions for research.

8:55 AM Invited

Fossil Energy Extreme Conditions Materials Research Program: *Robert Romanosky*[1]; [1]NETL

One of the most difficult challenges facing the Advanced Materials Research Program of the Department of Energy, Office of Fossil Energy, is the development of materials for the extreme environments encountered in advanced power generation systems. Advanced materials are vital to higher performance and more economic operation of fossil energy systems. The scope of the program addresses material requirements for all fossil energy systems, including materials for fossil fueled advanced power generation technologies such as gasification, turbines, combustion systems, advanced sensors, and fuel cells. Research is focused on developing high-temperature, corrosion-resistant alloys and protective coatings that are compatible with advanced power system high temperature environments, as well as materials that perform specific functions in advanced fossil energy systems. A detailed overview of the Materials Program effort will be presented with an emphasis on research efforts in development of materials for the extreme environment of advanced power generation technologies.

9:20 AM

Materials Evolutions in Hot Parts of Aero Turboengines: *Jean-Yves Guedou*[1]; Claude Quillien[1]; [1]SNECMA

The requirements in aero-turbo-engines regarding endless improved performances on higher overall pressure ratios, compressor discharges and turbines entry temperatures lead to more and more severe thermo-mechanical loadings in critical parts such as turbine discs and blades. The Ni base superalloys have been developed by the 80's – 90's to fulfil those challenges and they are presently at an industrial level. So the Research activities on those materials have been carried on for the last decade more for reliability improvement and cost savings purposes than properties upgrading. New grades tailored for higher performances are still being developed both for single crystals and PM alloys but the improvement capabilities appear to be more and more limited. For Future, light highly resistant and refractory materials are sought beyond Ni base alloys. Ceramics matrix composites, eutectic solidified ceramics and high temperature intermetallics such as silicides are investigated as potential breakthroughs in the 2020 aero- turbo-engines.

9:40 AM

Materials and Component Development for Advanced Turbine Systems: *Mary Anne Alvin*[1]; [1]US DOE NETL

Hydrogen-fired and oxy-fueled land-based gas turbines currently target inlet operating temperatures of ~1425-1760C (~2600-3200F). In view of natural gas or syngas-fired engines, advancements in both materials, as well as aerothermal cooling configurations are anticipated prior to commercial operation in 2015. This paper reviews recent technical accomplishments resulting from NETL's collaborative research efforts with the University of Pittsburgh and West Virginia University for future land-based gas turbine applications.

10:00 AM Break

10:10 AM Invited

The Properties of New High Temperature Cobalt-Based Superalloys: *Tresa Pollock*[1]; Akane Suzuki[1]; [1]University of Michigan

The recent discovery of Ishida and co-workers of the existence of a stable L12 phase field in the ternary Co-Al-W system suggests a path for development of a new class of high temperature alloys. The properties of quaternary and higher-order Co-Al-W base alloys with additions of Ta, Ti, Cr, Re, Mo and Ni have been investigated. Two phase microstructures with high volume fractions of the gamma prime phase have been observed over a range of composition. Single crystals of these materials have been grown using a conventional Bridgman process. In the [001] orientation, a temperature-dependent flow stress anomaly is observed. The rise in flow stress above 873K is much higher in comparison to two-phase nickel superalloys and the deformation mechanisms responsible for this behavior will be discussed along with the influence of alloy composition.

10:35 AM

Enhanced Creep Rupture Strength in Re and Ru Containing Nickel-Base-Superalloys by Addition of Minor Elements: *Astrid Heckl*[1]; Robert Singer[1]; [1]University of Erlangen-Nürnberg

The performance of gas turbines in power plants is governed by nickel-base-superalloys, which can sustain severe thermal and mechanical stresses under extreme conditions. Alloy development to increase the gas inlet temperature is fundamental for a continuous efficiency improvement, which simultaneously leads to a cost decrease of energy production, as well as lower CO2-emissions. Important improvements in the creep rupture strength of modern Nickel-Base-Superalloys have been achieved by adding elements like Rhenium (Re) and Ruthenium (Ru). In the present work we investigate the creep rupture strength of the alloy CMSX-4 and in-house designed new alloys with different Re and Ru contents. Special emphasis is placed on the effect of minor elements like carbon

(C) and magnesium (Mg). These elements are found to affect the mechanical properties substantially. Single crystals as well as columnar grained samples are studied in order to clarify the reasons for the positive effect of minor element additions.

10:55 AM

Effects of the Al Content on the Mechanical Behavior of NiAl Strengthened Ferritic Fe-Based Superalloy: *Zhenke Teng*; Shenyan Huang[1]; Peter Liaw[1]; Chain Liu[2]; Gautam Ghosh[3]; Morris Fine[3]; Gongyao Wang[1]; [1]University of Tennessee; [2]Oak Ridge National Laboratory; [3]Northwestern University

For body-centered-cubic (BCC) Fe matrix, ordered B2 NiAl-type ß' precipitates form in a coherent-coplanar orientation, providing the possibility of achieving a Fe-based analogue to the Face-centered-cubic (FCC) nickel-based γ/γ' superalloys. However, the applications of this type of alloy are restricted by the limited creep resistance at temperatures higher than 973 K and the poor ductility at room temperature. In this research, the effects of Al content on the mechanical behavior of NiAl strengthened ferritic Fe-based alloy were studied. Our results show that the optimal creep properties can be reached when the Al content is about 6.5 wt.%. The ductility at room temperature will improve with decreasing the Al content. To reach an optimal balance of creep and ductility properties, the addition of Al is estimated in the range of 5 - 7 wt.%.

11:15 AM

On the Different γ-γ' Behaviors over the Dendritic Structure of the New Generation Ni-Base Single Crystal Superalloy MCNG: *Michaël Arnoux*[1]; Xavier Milhet[1]; José Mendez[1]; François Vogel[2]; [1]LMPM - UMR CNRS 6617; [2]Turboméca - Safran Group

The capability of reaching higher operating temperature is essential in prospect of the development of a new supersonic aircraft engine or for the integrity of helicopter turbines when abrupt overheating occurs during specific operating regimes. MCNG is a new generation Nickel-base single crystal superalloy, containing both Rhenium and Ruthenium. γ-γ' microstructure evolutions were studied either after very high temperature exposures at 1200°C and 1250°C or during creep at 1050°C / 140 MPa. Drastic behavior differences are observed between dendritic and interdendritic regions. The influence of Rhenium on diffusion rates and on internal stress level (through γ-γ' lattice mismatch), coupled with its preferential partitioning in dendritic cores, are discussed to explain the differences in term of directional coarsening rates, phase ripening, raft stability and γ' phase dissolution. The particular creep behavior of MCNG at 1050°C / 140 MPa is also discussed regarding the continuous microstructure evolution.

11:35 AM

Crack Observations during Sustained-Peak Low Cycle Fatigue in Single-Crystal René N5: *Akane Suzuki*[1]; Michael Gigliotti; Michael Gigliotti[1]; Brian Hazel[2]; Tresa Pollock[3]; [1]GE Global Research; [2]General Electric Aviation; [3]University of Michigan

Crack-development during compressive sustained-peak low cycle fatigue (SPLCF) was examined in vapor phase aluminide coated single-crystal René N5. Tests were conducted at 1093°C with 0.35% total strain range. Tests were ended at selected fractions of predicted life. Crack lengths on the surfaces and crack depth in longitudinal sections were examined for each specimen. Cracks were observed on the coating surface in a sample removed at 10% of predicted life. Crack lengths into the coating increased with cyclic exposure. Cracks did not penetrate into the substrate through the interdiffusion zone until about 80% of predicted life. These results suggest that understanding crack growth behavior within the coating and inter-diffusion zone would provide important insight into SPLCF behavior. The roles of mechanical properties, environmental resistance of the coating and inter-diffusion zone, and fracture mechanics will be discussed.

11:55 AM

Thermodynamical Considerations for Applying the Halogen Effect to Ni-Base Superalloys: *Hans-Eberhard Zschau*[1]; Patrick J. Masset[1]; Daniel Renusch[1]; Michael Schütze[1]; [1]DECHEMA e. V.

Future power generation and propulsion concepts require an increased efficiency for saving fuel and reducing environmental pollution. Due to their wide application in high temperature technologies the Ni-base superalloys have to withstand high temperatures and extreme corrosion environments. In the present paper a concept for next generation oxidation protection is presented. Oxidation of these alloys in most cases does not form a pure continuous protective alumina scale on the surface, but rather a complex layer structure. This structure is characterized by internal oxidation. By using the halogen effect which had successfully been applied for the TiAl-alloys the internal oxidation can be transformed into external scale formation. The thermodynamical conditions of the halogen effect in the field of Ni-base alloys are investigated and discussed.

Materials Issues in Additive Powder-Based Manufacturing Processes: Additive Manufacturing Applications

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Powder Materials Committee
Program Organizers: David Bourell, University of Texas; James Sears, South Dakota School of Mines and Technology; Pavan Suri, Mississippi State University

Monday AM
February 16, 2009
Room: 3004
Location: Moscone West Convention Center

Session Chair: David Bourell, University of Texas

8:30 AM

Overview: Laser Additive Manufacturing and Repair: Issues and Opportunities: *James Sears*[1]; [1]South Dakota School of Mines and Technology

Laser Additive Manufacturing (LAM) is serving a growing number of applications. The LAM technology employs metal and composite powders as the additive material. The LAM technology serves the market segment where traditional thermal spray and welding techniques have failed to provide adequate solutions. Laser cladding is one of the most widely used LAM techniques. LAM for directly building complete structures has been limited but also seems to be growing. Lasers have been the choice when automation and low heat input are required. So how does laser technology compare to other methods (e.g., electron beam, plasma transferred arc, and ultrasonics) being used in additive manufacturing? Also, how are the other technologies being refined for additive manufacturing to fill similar requirements that previously only lasers could perform? This paper discusses the relative attributes of each of these technologies and how they compared to each other.

8:55 AM

Material Issues in the Qualification of LENS® For Structural Applications Including Repair: *David Gill*[1]; John Smugeresky[2]; [1]Sandia National Laboratories, Albuquerque, NM; [2]Sandia National Laboratories, Livermore, CA

Laser Engineered Net Shaping ™ (LENS®) offers opportunities to repair and modify components by adding features to or replacing damaged one on existing parts. A simple bracket was used to qualify the LENS Process with minimal time and cost for testing. LENS deposited material was evaluated for interface strength, machinability, weldability, corrosion resistance, geometric effects, heat treatment, and repair strategy. Parts were subjected to mass analysis and structural dynamic testing including free-free and assembly-level modal tests, and Haversine shock tests. The brackets performed as well as conventionally processed brackets. The brackets were subjected to testing in actual subsystem level tests, which qualified the LENS process. This presentation will include an overview of the qualifying tests and evaluation completed, with special focus on the materials analysis comparing layer deposited material with wrought material. Work by Sandia is supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

9:20 AM

Biofabrication of 3D Tissue Scaffolds and Cell-Integrated Constructs: Material and Process Issues as Well as the Effects on Biological Behavior: *Wei Sun*[1]; [1]Drexel University

Recent cell biology and scaffold-guided tissue engineering research has increasingly explored using 3D in vitro cell culture models to study gene expression and other complicated biological phenomena that more closely simulating in vivo microenvironment. Solid Freeform Fabrication (SFF) has been proven as a promising technique to meet this demand. Although widely used for fabricating tissue scaffolds, there are many challenges in materials and in processes for SFF in biological application, including material biocompatibility, process feasibility, structural formability, and the limitation of fabricating cell-integrated biological structure. Furthermore, the material and process may also

affect the biophysical and biological function of the SFF-ed structure. This presentation will review some recent SFF enabled applications in biological and tissue engineering, with a discussion of issues and challenges, along with an introduction of our research on 3D tissue scaffold fabrication and construction of tissue analog for drug metabolism study.

9:45 AM
Design and Production of Bone Scaffolds with Selective Laser Melting: *Simon Van Bael*[1]; Ben Vandenbroucke[1]; Greet Kerckhofs[1]; Jan Schrooten[1]; Jean-Pierre Kruth[1]; [1]KUL

The use of bone scaffolds for treatment of large bone defects promises a solution for all disadvantages which are present with traditional care methods. The success of these scaffolds depends on its internal structure and mechanical properties. To be able to conduct a reliable investigation on the effect of these parameters, an efficient production method is required which can produce controlled internal structures. The presented work examines the ability to produce Ti6Al4V bone scaffolds with selective laser melting. The bone scaffolds were produced with a pore size range of 400-900µm and strut size 200µm. To check the repeatability mechanical en geometrical tests were performed.

10:10 AM
Computational Materials Design and Layered Fabrication of Solid Oxide Fuel Cells: *Suman Das*[1]; Chan Yoon[1]; [1]Georgia Institute of Technology

Electrodes in a solid oxide fuel cell (SOFC) must possess both electronic conductivity and porosity to perform their functions in the cell. They must be porous to permit rapid mass transport of reactant and product gases and be electronically conductive to transport electrons easily. However, it is nearly impossible to control electronic conductivity and porosity simultaneously using conventional fabrication techniques. Our aims are to investigate computational materials design of SOFCs and to develop a dry powder direct-write system for controlling the distribution of SOFC materials consistent with these designs. We then aim to apply the dry powder direct-write system to the fabrication of SOFCs with higher power density and thus higher efficiency than currently attainable in state-of-the-art SOFCs. This talk will present results of our efforts on computational materials design, experimental fabrication, and performance testing of SOFCs built through a layered fabrication approach.

10:35 AM Break

10:50 AM
Rapid Prototyping of Direct Methanol Fuel Cell (DMFC) Graphite Bipolar Plates by Indirect Selective Laser Sintering (SLS): *Kaushik Alayavalli*[1]; David Bourell[1]; [1]University of Texas

Graphite bipolar plates are highly desirable due to their high electrical conductivity, low weight and resistance to corrosion. However, the poor mechanical properties of graphite lead to prohibitive machining cost. Indirect Selective Laser Sintering (SLS), involving laser sintering of graphite powders mixed with a phenolic resin binder, offers the advantage of rapid, complex part production and testing of prototype bipolar plates. Carbonizing the poorly conducting, highly porous plates at high temperature and infiltrating with a low viscosity (~5 – 10 cps) cyanoacrylate polymer improves the electrical conductivity significantly and renders the plate fluid impermeable. A CAD model for various plate configurations was optimized for methanol flow and current distribution using a Computational Fluid Dynamics (CFD) simulation tool. Optimized models were fabricated by indirect SLS and evaluated in a fuel cell test bed. This work was supported by the Office of Naval Research MURI Grant No. N00014-07-1-0758.

11:15 AM
Layered Manufacturing of Metallic Cellular Materials via Three Dimensional Printing of Spray-Dried Metal Oxide Ceramic Powder: *Christopher Williams*[1]; Joe Cochran[2]; David Rosen[2]; [1]Virginia Tech; [2]Georgia Institute of Technology

In this paper, the authors augment the three-dimensional printing process in an effort to address geometric, build time, and cost limitations typically found in the realization of cellular materials with direct-metal layered manufacturing technologies. Specifically, metallic cellular materials are made by selectively printing solvent into a bed of spray-dried metal oxide ceramic powder. The resulting green part is then sintered in a reducing atmosphere to chemically convert it to metal. As a result of their investigation of this process, the authors are able to create cellular materials made of maraging steel that feature wall sizes as small as 400 µm, angled trusses and channels that are 1 mm in diameter, and have an estimated average cost of ~$3.00 per cubic inch.

11:40 AM
Investigation of New Materials for Selective Laser Sintering: *Ruth Goodridge*[1]; Richard Hague[1]; Chris Tuck[1]; [1]Loughborough University

Attempting to exploit the dramatic advantages in design that are achieved by taking an additive approach to manufacturing, researchers at Loughborough University are investigating the potential to produce custom-fitting sports Personal Protective Equipment (PPE) using the powder-based technique, selective laser sintering (SLS). However the limited range of materials that can be processed by SLS, and their high cost compared to materials used in existing manufacturing processes, is particularly problematic for sports PPE as current SLS polymers cannot withstand the cyclic and high impact loading conditions experienced in contact sport.Attempts have therefore been made at Loughborough to process new materials, such as UHMWPE, PLA, PCNs, with varying degrees of success. This talk will address the problems that have been experienced and the knowledge that has been gained through the search for new materials that are more suitable for this and other such demanding applications.

12:05 PM
Effect of Liquid Phase Migration on Extrusion Pressure in Freeze-Form Extrusion Fabrication: Hongjun Liu[1]; *Ming Leu*[1]; Robert Landers[1]; Gregory Hilmas[1]; [1]Missouri University of Science and Technology

Freeze-form extrusion fabrication (FEF) process extrudes an aqueous ceramic paste of high solids loading to fabricate 3D ceramic green parts. Liquid phase migration (LPM) may exist in this process and influence the paste composition and extrusion pressure. This paper describes a study of the existence of LPM and its effect on extrusion pressure in the extrusion of alumina paste by the FEF process. Based on the extrusion pressure profile, the extrusion process can be divided into three stages: the compaction stage, the steady stage, and the dead zone stage. The extrusion pressure increases gradually with ram displacement in the steady stage for the all the ram velocities tested. Also, the extrusion pressure increases when the ram velocity increases or nozzle diameter decreases. These observations can be explained using the Benbow-Bridgwater model. It is also found that the steady stage enlarges with increasing ram velocity or decreasing nozzle diameter.

Materials Processing Fundamentals: Solidification and Casting

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Technology and Modeling Committee
Program Organizer: Prince Anyalebechi, Grand Valley State Univ

Monday AM — Room: 2016
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Prince Anyalebechi, Grand Valley State University

8:30 AM
Influence of Strong Convection Patterns on Remelting and Species Transport in a Composite Casting Process: *Autumn Fjeld*[1]; Andreas Ludwig[1]; [1]University of Leoben

In a composite casting process remelting and mixing is critical to the formation of a well-bonded, durable casting and are dependent upon the flow patterns that arise during mold filling. In the casting under investigation, a thin outer shell material is first cast inside a large cylindrical mold, which is then assembled to a lower and upper neck mold, and finally the core material is poured into the shell-mold assembly. The filling process has been simulated to investigate the effect of strong convection patterns in the liquid metal on the remelting, mixing, and species transport of the outer-shell material during filling of the inner-core material. The present numerical model captures the global convection patterns and species transport and mixing between the shell and core materials. Simulations have shown that the convection patterns that develop during filling dictate the degree of shell remelting and material transport.

8:45 AM

Control of Microstructure in Electrical Steel with Directional Solidification by LASER: *Jung-Ryoul Yim*[1]; Eun-ho Choi[1]; Yo-Han Yoon[1]; Jung-Han Kim[1]; Kyu-Hwan Oh[1]; Young-Chang Joo[1]; [1]Department of Materials Science and Engineering, Seoul National University

Electrical steel needs the control of texture for its excellent soft magnetic properties. Especially, electrical steel needs the control of <100> orientation, because <100> orientation in steel is the direction of high magnetic induction and low core loss. As the melted steel continues to solidify, the grains - which have a <100> axis parallel to the steepest temperature gradient in the liquid - quickly outgrow those grains with less favorable orientation. Using Nd:YAG pulse laser (power: 4kW), 3.04%Si steel sheet with the thickness of 0.35mm can be melted through its entire thickness. Furthermore, the direction of solidification can be controlled through the change of thermal contact with the melted region. With this laser process, <001> orientation can be effectively controlled. Using EBSD, the microstructures of the lased regions are discussed.

9:00 AM

New Physical Phenomena: Temperature-Induced Liquid-Liquid Transition in Alloys and Its Effects upon Solidification: *Fang-Qiu Zu*[1]; Xian-Fen Li[1]; Lan-Jun Liu[1]; Jin Yu[1]; Yun Xi[1]; Zhi-Hao Chen[1]; Jie Chen[1]; Guo-Hua Ding[1]; Zhong-Yue Huang[1]; [1]Hefei Univesity of Technology

The knowledge on nature of liquid structures and properties remains an open problem for many fundamental and applied fields such as materials sciences & processing, condensed state physics, metallurgy etc. And as well known, there is no other defined phase line above liquidus in phase diagrams of ordinary binary systems. However, via different experimental resorts, our research results of recent years show a novel physical image: temperature induced liquid-liquid structure transition(TI-LLST)can occur hundreds of degrees above TL in over 30 metallic melts including alloys and elements. On the other hand, the solidification behaviors and structures from melts experienced TI-LLST are distinct from those from melts before TI-LLST. In this presentation, some characteristic aspects of the TI-LLST and the effects on solidification behaviors and structures are summarized, and the pertinent rules and mechanism are also theoretically analyzed.

9:15 AM

Effects of Application of Electric Current during Solidification on the Cast Microstructure of Aluminum Alloy 7050: *Prince Anyalebechi*[1]; Kathy Tomaswick[2]; [1]Grand Valley State University; [2]Alcoa, Inc.

The effects of application of steady and pulsed electric current on the cast microstructures of ingots of a 7050 type aluminum alloy have been experimentally investigated over a solidification rate range of 0.1-10 K/s. This involved the application of an electric current of 465-930 mA/cm2 of melt surface area to laboratory-size ingots solidified in a unidirectional manner. Within the electric current density and range of solidification rate investigated, the applied electric current reduced the average dendrite, grain, and second-phase particle size. It also made the size distribution of the second-phase particles more uniform. The mechanism for the observed refinement of the cast microstructure by the applied electric current is not well understood. It is provisionally attributed to the combined effects of heat-induced local convections, shear stress-induced fragmentation of dendrites, increased temperature gradient due to Joule heating and the thermal and constitutional supercooling engendered by Peltier, Thompson, and Joule heating.

9:30 AM

A Comparative Examination of the Tensile and Fatigue Properties of Aluminum Alloy A356 Automotive Suspension Components Produced by Different Shape Casting-Related Processes: *Prince Anyalebechi*[1]; [1]Grand Valley State Univ

A comparative study of the mechanical properties of automotive steering knuckles produced by three different shape-casting related processes has been conducted. It involved the characterization of the tensile and fatigue properties of aluminum alloy A356 knuckles produced by the vacuum/pressure riserless casting (VRC/PRC), pressure counter pressure casting (PCPC), and the hybrid Cobapress (a casting-forging) processes, in accordance with the appropriate ASTM standards. Surprisingly, the knuckles produced by the hybrid Cobapress process exhibited the lowest strength. For example, the average 0.2% yield strength of the Cobapress knuckles was 31 MPa and 40 MPa less than that of the VRC/PRC and PCPC knuckles, respectively. However, between stresses of 150-200 MPa, the fatigue lives of the VRC/PRC, PCPC, and Cobapress knuckles were comparable. But above 200 MPa, the Cobapress knuckles exhibited the lowest fatigue life. The observed differences in the mechanical properties are attributed to the inherently different microstructures of the different knuckles.

9:45 AM

Optimization of Submerged Entry Nozzle of Slab Continuous Casting: *Zhigang Liang*[1]; [1]Northeastern University

Effects of the structure of submerged entry nozzle on the flow filed in mould were investigated through a water modeling experiment. The results of physical simulation showed that there was an appreciable fluctuation of the free liquid surface in the mould with obvious exposure of liquid surface and entrapment of mould powder when the original submerged entry nozzle was applied. A stable liquid surface was obtained by increasing the immersion depth of submerged entry nozzle and enlarging the downward inclination angle, but the penetration depth of jet stream increased remarkably and the position of higher temperature zone descended, which is unfavorable for increasing the casting speed. By increasing the outlet area of the submerged entry nozzle, an even fluid field with good covering of free surface by mould powder was available without any change of other geometry parameters or immersion depth of submerged entry nozzle.

10:00 AM Break

10:15 AM

A Study of Non-Metallic Inclusion Evolution inside Fe-Al-Ti-O Melts: *Cong Wang*[1]; Sridhar Seetharaman[1]; [1]Carnegie Mellon University

In this study, non-metallic inclusions, such as alumina and titanium oxides, are systematically investigated by means of morphology examination, structure analysis as well as chemistry revelation. The investigation is carried out through sampling in a vacuum-induction furnace inside iron melts involving aluminum, titanium and oxygen. The purpose of this study is to simulate de-oxidation process of interstitial free steels through transient ladle reaction products. Based on structure analysis imparted via transmission electron microscopy, it is revealed that desired non-metallic inclusions are produced within accordingly predicted stable regions. In addition, it is found that inclusion morphology, under any thermodynamic stable circumstances, undergoes dramatic changes, which are always accompanied with statistically permanent evolution from initially spherical-dominated percentages to finally irregular-prevailed situation. It is also shown that chemistry may vary continuously within one individual inclusion, suggesting that local thermodynamic stable conditions may not be reached and that the inclusion was once viscous.

10:30 AM

Heat-Resistance Property of Cu-3.5Ti-0.1Zr Alloy: *Cao Xingmin*[1]; [1]Suzhou Institute of Non-Ferrous Metal Processing Research

Abstract: The effect of Zr on the heat-resistance of a Cu-3.5Ti (wt.%) alloy was investigated by mechanical tests and TEM observation. The results show that the softening temperature of the Cu-3.36Ti alloy was substantially increased by the addition of 0.11wt% Zr. The softening of the Cu-3.36Ti alloy is mainly controlled by the phase transition from TiCu4 to TiCu3 in the temperature range from 450° to 550°, and the addition of 0.11 wt.%Zr can retard this phase transition. The mechanism was discussed in relation to the microstructural evolution.

10:45 AM

Friction Stir Welding Characteristics of Different Heat-Treated-State 7075 Aluminum Alloy Plates: *Meysam Mirazizi*[1]; [1]Sharif University of Technology

Friction stir welding of 7075 al-alloys was performed to investigate the effects of the base material conditions on the FSW characteristics. The results indicated that the base material condition has a significant effect on weld morphologies, weld defects, and mechanical properties of joints.microscopy investigation showed that In the 7075-O welds, no visible interface exists between the stir zone and the ThermoMechanically Affected Zone and a weld nugget with an onion ring-like morphology not clearly exists.weld defects are formed in the lower part of the weld. In the 7075-T6 welds,there is visible interface between the SZ and the TMAZ,and a weld nugget with an onion ring-like morphology clearly exists. The defects are liable to form in the middle and upper part of the weld. The results of Shear Punch Test and tensile test showed that strength efficiency of 7075-O is greater than 7075-T6 joints (95% against 82%). The two types of joints have different fracture location characteristics.

11:00 AM
Hybrid Laser/GTAW Welding of Galvanized High-strength Steels in Gap-Free Lap Joint Configuration: *Shanglu Yang*[1]; [1]Research Center for Advanced Manufacturing

In this study, laser-GTAW hybrid welding was used to the lap welding of galvanized high-strength steels in a gap-free lap joint configuration. The effects of welding parameters such as the laser power, the distance between the laser beam and the electrode torch, the arc current on the quality of hybrid welds were studied. The reason for the formation of different weld defects arises from the development of the highly-pressurized zinc vapor at the interface of two metal sheets. Experimental results demonstrated that the elongated molten pool introduced by the GTAW can suppress the formation of spatters to some extent. However, the hybrid laser-arc welding process still suffers from a large amount of spatters that degrade the weld quality. Additionally, it was found that the weld quality was enhanced with the increase in the laser beam-arc distance and welding current.

11:15 AM
Optimization of Ductile Iron Treatment by Computing the Refining Reactions in Liquid Irons: *Simon Lekakh*[1]; David Robertson[1]; Sergey Rimoshevsky[2]; Vladimir Tribyshevsky[2]; Nikolay Bestyzev[2]; [1]Missouri University of Science & Technology; [2]Belarusian State Polytechnic University

Experimental work and thermodynamic simulations of cast iron refining were carried out using various additions of alkali and rare earth metals. The sequences of the refining reactions for sulfur and oxygen removal were found for melts treated with Mg, Ca, and Ce. The measured data and the results of the computer simulations were in agreement. Experimental kinetic data, together with the thermodynamic calculations, were used for optimization of the amount and sequence of the addition of nodulizers for ductile iron production.

11:30 AM
Influence of the Submerged Entry Nozzle Geometry on the Heat Transfer inside the Continuous Casting Mold: Jaqueline Alexander[1]; *Cesar Real-Ramirez*[1]; Manuel Palomar-Pardave[1]; Raul Miranda-Tello[1]; Jesus Gonzalez-Trejo[1]; [1]Universidad Autonoma Metropolitana - Azcapotzalco

Some of the most important phenomena which govern the continuous casting process and determine the quality of the product are the fluid flow and the heat transfer. Steel flows into the mold through the ports of a bifurcated submerged entry nozzle (SEN), which directs the jet to the mold narrow faces where the superheat contained is dissipated on the solidifying shell. Many important aspects of the fluid flow in the mold are transient and difficult to control. However, the time-averaged flow pattern in the mold is greatly influenced by the nozzle geometry, the submergence depth and the mold dimensions. The aim of this work is to analyze the relationship between the heat transfer and the fluid flow pattern inside the mold using the CFD technique at several operation conditions.

Mechanical Behavior of Nanostructured Materials: Stability of Nanostructures

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Xinghang Zhang, Texas A & M University; Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Monday AM — Room: 3024
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Xinghang Zhang, Texas A & M University; Alan Jankowski, Texas Tech University

8:30 AM Introductory Comments

8:35 AM Keynote
Thermal and Mechanical Stability of Nanocrystalline Grain Structures: *Carl Koch*[1]; [1]North Carolina State University

This talk will review the thermal, and to a lesser extent, the mechanical stability of nanocrystalline grain structures. Since grain boundaries are not equilibrium defects in crystals, polycrystalline materials are metastable and with sufficient thermal activation the grain size microstructure will coarsen. This is particularly true for nanocrystalline microstructures where the enormous grain boundary area provides a large driving force for grain growth. However, there are kinetic and thermodynamic processes that can provide significant stabilization of nanocrystalline grain sizes. This talk will briefly review examples of grain boundary stabilization in nanocrystalline materials by kinetic approaches. The major part of the presentation will focus upon thermodynamic stabilization by solute segregation to grain boundaries from reports in the literature and from the author's laboratory. The possibility of recrystallization of nanocrystalline microstructures will be discussed. The stress-induced coarsening of nanocrystalline grains will also be considered. Author's research supported by NSF, DMR-0504286.

9:05 AM
Evolution of Mechanical Properties during Room Temperature Grain Growth of Nanocrystalline Pd: *Rainer Birringer*[1]; Markus Ames[1]; Manuel Grewer[1]; Jürgen Markmann[1]; [1]Saarland University

Nanocrystalline Pd prepared by IGC exhibits room temperature grain growth in the limit of high purity thereby bridging a size range from 10nm to 10 µm. The growth kinetics significantly deviate from parabolic behavior. Possible scenarios that may explain this deviation are shortly addressed. The main focus concentrates on discussing results obtained from in situ measurements of mechanical properties during RT grain growth. Setting up appropriate scaling laws enables to extract interface stress and interface elastic moduli. Grain-size-dependent hardness and strain rate sensitivity will be discussed in the light of available data from non-in-situ measurements.

9:20 AM
Thermal Stability of LIGA Nickel Composites for High-Temperature MEMS Applications: *S.J. Suresha*[1]; Manel Haj-Taieb[2]; Jarir Aktaa[2]; Kevin Hemker[1]; [1]Johns Hopkins University, Department of Mechanical Engineering; [2]Forschungszentrum Karlsruhe, Institut für Materialforschung II

LIGA Ni-W MEMS structures with 5 and 15 at% W were prepared by electrodeposition. The thermal stability of the Ni-W specimens were investigated by annealing at 400 and 700°C for different durations (1h and 4h). The microstructure and mechanical properties were analyzed with TEM, XRD, micro-tensile testing and indentation. No precipitates were observed, but in comparison to the LIGA Ni the microstructure of the LIGA Ni-W was found to be stable up to 700°C. The tensile strengths of the LIGA Ni-W were higher than for pure LIGA nickel in both as received and annealed states. The Ni-W structure exhibited brittle failure at room temperature, but the annealed Ni-W samples exhibited considerable ductility before fracture and showed higher tensile strength. Thermal stability in this system appears to come from the segregation

of W in the grain boundary and the increased ductility and strength from grain boundary strengthening in the nanocrystalline range.

9:35 AM

Thermal Stability of Nanostructured Materials Created by Severe Plastic Deformation: Christopher Saldana[1]; Jiazhao Cai[2]; Sergei Suslov[1]; *Ravi Shankar*[2]; Srinivasan Chandrasekar[1]; Eric Stach[1]; [1]Purdue University; [2]University of Pittsburgh

Severe plastic deformation (SPD) while offering a convenient and scalable framework for creating fully-dense nanostructured materials from a range of alloy systems significantly limits the options available for achieving high levels of thermal stability in the resulting fine-grained materials. Following traditional approaches, the stability of severely deformed materials can be improved by utilizing multi-phase alloys composed of a dense dispersion of second phases. In such multi-phase systems, we show that the overall stability of the fine-grained material is in turn determined by the stability of the second phases. Examples of precipitate stabilized nanostructured materials from aluminum and nickel alloys are utilized to illustrate stabilization criteria. Rate-dependent strengthening and related mechanical behavior of these materials are presented. Finally, an emerging interface engineering approach is discussed wherein a high-density twin of nano-lamella when introduced amongst nano-scale grain boundaries through SPD at cryogenic temperatures is shown to improve the overall thermal stability.

9:50 AM

Thermal Stability of Ultra-Fine Grained Ti-6Al-4V Alloys Processed via Multi-Axis Forging: *Radhakrishna Bhat*[1]; Richard Didomizio[1]; Andrew Deal[1]; Judson Marte[1]; P. Subramanian[1]; [1]GE Global Research

A near-isothermal multi-axis forging (MAF) process was used to produce ultra-fine grained (UFG) Ti-6Al-4V alloys. The thermal stability of the resulting ultra-fine grained structure was evaluated at temperatures below the beta transus under both static and dynamic conditions in order to investigate the coarsening kinetics of the primary alpha particles in the alpha+beta phase field. Specimens were heat-treated for varying durations at different temperatures within the alpha+beta phase field for the static studies, while hot compression tests were conducted to evaluate the thermal stability under dynamic conditions. The results of the characterization with scanning electron microscopy and quantitative metallography will be presented and compared with data for the conventionally processed Ti-6Al-4V material. The coarsening mechanisms and methods to stabilize the ultra-fine grain size will be discussed, especially in the context of using UFG Ti-6Al-4V for producing near-net shape Ti components via superplastic deformation.

10:05 AM

Deformation Behaviour of Nanocrystalline Pd Studied in Conditions of High Pressure Torsion Loading: *Julia Ivanisenko*[1]; Jörg Weissmüller[1]; Hans-Jörg Fecht[2]; [1]Forschungszentrum Karlsruhe in der Helmholtz Gemeinschaft; [2]Universität Ulm

We have investigated the hardening behaviour of nanocrystalline nc igc Pd with a mean grain size of 12 nm in compression-torsion mode in a wide range of shear strains (0-400). We show that in the studied shear strain range the notable changes in the microstructure, namely a strain induced grain growth of igc Pd occurs, that controls the relevant deformation mechanisms. For lower strains when the grain size is still small enough, the plastic flow is governed by twinning and probably grain boundary sliding. For this range of shear strains a rapid strain hardening observed in the shear strain vs. torque curves is conditioned by the increase of the twins density. When the grain size becomes larger and deformation is controled exceptionally by dislocation glide a steady stage is achieved, when the grain size, dislocation density and flow stress (torque) are saturated.

10:20 AM Break

10:30 AM Invited

Plastic Flow and Irradiation Stability of Nanolayered Composites: *Amit Misra*[1]; [1]Los Alamos National Laboratory

Magnetron sputtering is used to synthesize nanolayered composites with controlled length scales in the nanometer range. These materials exhibit ultra-high flow strengths, typically within a factor of two to three of the theoretical strength limit of perfect crystals. The morphological and chemical stability of incoherent interfaces such as copper-niobium in these nanolayered composites was explored at large plastic strains via tensile tests, pillar compression and rolling. Furthermore, ion irradiation, over a range of temperatures and ion doses, was used to examine the irradiation stability. These materials exhibit remarkable thermo-mechanical and irradiation stability due to the ability of the interfaces to attract, absorb and annihilate defects. The design of the nanostructural dimensions and interface structures to achieve both ultra-high strength and high radiation damage tolerance will be discussed. This work is supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences.

10:50 AM

The Effect of Layer Thickness and Volume Fraction on Structure and Mechanical Properties of Al/TiN Multilayers: *Dhriti Bhattacharyya*[1]; Nathan Mara[1]; Patricia Dickerson[1]; Richard Hoagland[1]; Amit Misra[1]; [1]Los Alamos National Laboratory

Nano-scale multilayers of Al and TiN with Al layer thickness varying from 5nm to 500nm and TiN layer thickness varying from 1nm to 50nm were deposited in two different thickness ratios – Al:TiN :: 9:1 and Al:TiN :: 1:1. The hardness values measured by nanoindentation increased with decreasing layer thickness and the hardness of the multilayers with Al:TiN :: 1:1 thickness ratio was, in all cases, more than the hardness for the multilayers with the same bilayer thickness having a layer thickness ratio of Al:TiN :: 1:1. The hardness values of the two kinds of multilayers were found to be close for any given bilayer thickness, when normalized by their modulus of elasticity. These results are discussed in terms of possible dislocation mechanisms and the structure of the interfaces between the Al and TiN layers, which were characterized by Transmission Electron Microscopy (TEM).

11:05 AM Invited

Is There a Future for Nanograined Steel?: *John Morris*[1]; [1]University of California, Berkeley

While it is sometimes assumed that nanograined materials will come to dominate the structural, as well as the electronic applications for advanced materials, researchers who specialize in structural steels are less optimistic. While ultrafine grain size leads to exceptionally high strength, tensile ductility is lost, and it is difficult to achieve useful ductility in conventional steels with grain sizes much below 1 μm. However, strength is only one of the important properties of steel. Ductile fracture and hydrogen resistance are also important. They are also influenced by grain size, but by different mechanisms, with the consequence "grain size" has a somewhat different meaning. Given the crystallography of coherent transformations in steel, it is possible to create steels that have submicron grain size with respect to fracture or embrittlement, while retaining excellent strength and good ductility. This is the promising path to nanostructured steels with exceptional properties.

11:25 AM

Atom Probe Tomography, Small Angle Neutron Scattering and Transmission Electron Microscopy Characterization of Nano-Scale Features in MA957: *G. Robert Odette*[1]; Emmanuelle Marquis[2]; Peter Hosemann[3]; Pifeng Miao[1]; Nicholas Cunningham[1]; Sergio Lazono-Perez[2]; Matthew Alinger[4]; Erich Stergar[5]; [1]University of California; [2]University of Oxford; [3]Los Almas Nat Laboratory; [4]GE Global Research; [5]University of Leoben

Nano-dispersion strengthened ferritic alloys contain a high density of thermally stable Y-Ti-O nano-scale features (NFs) which provide both high creep strength and irradiation damage resistance. The NFs have been studied by small angle neutron scattering (SANS), three-dimensional atom probe tomography (APT) and transmission electron microscopy (TEM). However, the compositions and structures of various NFs are not well understood, and they appear to range from coherent solute enriched GP-type zones (in APT studies) to stoichometric complex oxides (Y2TiO5 and Y2Ti2O7, in some SANS and TEM studies). We cross compare the APT (four groups), SANS and TEM (two groups) characterization of the NFs in MA957. The various techniques are generally in good agreement on the sizes and number densities of the NFs. However, APT studies show high Ti+Y/O and Ti/Y ratios that are inconsistent with complex oxides. The APT indicates a complex shell structures of Y-T-O enriched cores surrounded by TiO shells.

11:40 AM

Deformation Behavior of High Strength Nano-Structured Ferritic Alloys: *David Hoelzer*[1]; Jim Bentley[1]; Meimei Li[2]; Mikhail Sokolov[1]; David McClintock[1]; [1]Oak Ridge National Laboratory; [2]Argonne National Laboratory

Reducing the grain size and dispersing precipitates are classical ways for increasing the strength of bulk metallic alloys. For an advanced ferritic alloy

produced by mechanically alloying, the dispersion of nano-size oxygen-rich clusters, i.e. nanoclusters, caused nano-size grains to form. This nano-structured ferritic alloy (NFA) possesses high tensile strengths from low (196ºC) to elevated (800ºC) temperatures with some measure of ductility at low temperatures. Furthermore, the NFA has a low ductile-to-brittle transition temperature and shows ductile failure characteristics in tension down to -196ºC. The deformation behavior of the NFA including the stability of the nanoclusters and nano-size grains during deformation at low and elevated temperatures will be presented. Research supported by the Office of Nuclear Energy, Science and Technology, by the Office of Fusion Energy Sciences, and at the SHaRE User Facility by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

11:55 AM Invited
Deformation Process of Nanocrystalline Materials with In-Situ TEM and Synchrotron: *Scott Mao*[1]; [1]University of Pittsburgh

The discovery of mechanical grain growth at liquid nitrogen temperatures show such unique stress induced microstructure evolution. It is therefore necessary to study the properties of non-equilibrium boundaries effect on the dislocation storage or grain agglomeration/ growth induced by deformation. This talk focuses on stress-induced microstructure evolution of grain agglomeration in nc materials through in-situ TEM and in-situ synchrotron tests. We used in situ TEM and observe nc Ni with an average grain size of about 10 nanometers, which shows deformation-induced grain agglomeration. It has been found that grain boundary mediated processes have become a prominent deformation mode. In collaboration with Dr.Yang in Argonne National Laboratory, in situ synchrotron on nc and micron Ni under hydrostatic stress up to 57Gpa show that peak broadening increases during loading up to 45 Gpa in nc-Ni, which indicates high dislocation density storage, and no clear grain growth or texturing.

12:15 PM
In Situ TEM Nanocompression Testing of Gum Metal: *Elizabeth Withey*[1]; Jia Ye[2]; Velimir Radmilovic[2]; Shigeru Kuramoto[3]; Andrew Minor[1]; Daryl Chrzan[1]; John Morris[1]; [1]University of California; [2]National Center for Electron Microscopy, Lawrence Berkeley National Laboratory; [3]Toyota Central Research and Development Laboratory Inc

Gum Metal is a newly developed set of ß-Ti alloys that, in the cold-worked condition, have exceptional elastic elongation and high strength. The available evidence suggests that Gum Metal does not yield until the applied stress approaches the ideal strength, and then deforms by mechanisms that do not involve conventional dislocation plasticity. In order to study the deformation behavior in more detail, in situ compression of submicron-sized pillars has been performed on solution-treated and cold-worked samples of one composition of Gum Metal. Explanation of the mechanical behavior observed was assisted through the correlation of quantitative load vs. displacement data and real-time images, along with high resolution microscopy of undeformed samples of solution-treated and cold-worked Gum Metal of the same composition used in the compression tests.

12:30 PM
Role of Nanoscale Interface Diffusion in Creep Deformation and Microstructural Stability of Si-C-N Nanocomposites in High Temperature Environments: Ming Gan[1]; *Vikas Tomar*[1]; [1]University of Notre Dame

Next generation ceramic nanocomposite coatings need to have excellent creep strength and microstructural stability at extreme operating temperatures beyond 1750 K. In the presented research atomistic analyses of microstructural stability and creep deformation in Silicon (Si) carbide (C) and nitride (N) coatings developed for this purpose are presented. The focuses is on understanding the nanoscale diffusion phenomenon in such materials and correlate the developed understanding with observed creep strength and microstructural stability characteristics. Atomistic analyses are performed using non equilibrium molecular dynamics. Such analyses are then correlated with nanoindentation creep studies on some representative samples. Analyses show that the nanoscale diffusion dominates the creep behavior of the nanocomposites. In addition by varying the nanoscale structural configuration a significant improvement in creep strength and microstructural stability could be obtained. Alternate phase arrangements based on biomimetic structures are also analyzed.

12:45 PM
Change of Deformation Mechanism in Nanocrystalline Nickel at Very Low Temperatures: *Lutz Hollang*[1]; Klemens Reuther[1]; Suhash Dey[1]; Werner Skrotzki[1]; [1]Dresden University of Technology

Pure nanocrystalline nickel was produced by pulsed electro-deposition without additives for grain refinement. The average grain size of the material is d_{EBSD} = 150 nm and d_{XRD} = 30 nm if determined by electron backscatter diffraction (EBSD) and X-ray diffraction (XRD), respectively. Tensile tests with constant deformation rate were performed at temperatures between 4 K and 320 K. The stress–strain curves are parabolic with the ultimate stress strongly decreasing with increasing temperature. Stress relaxation experiments reveal that dislocation interaction governs the plastic behaviour of the material at low temperatures. However, if the stress attains the threshold of 2400 MPa, as it is the case between 4 K and 9 K, the deformation mode suddenly changes towards "catastrophic" shear. The shear events are characterized by substantial stress drops accompanied by acoustic emission. The nature of the shear events will be discussed on the basis of microstructural investigations performed by electron microscopy.

Microstructural Processes in Irradiated Materials: Radiation Effects I: Segregation and Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Christophe Domain, Electricite De France; Gary Was, University of Michigan; Brian Wirth, University of California, Berkeley

Monday AM — Room: 2008
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Charlotte Becquart, University of Lille; Christophe Domain, Electricite De France

8:30 AM Invited
Phase Field Modeling for Irradiation-Induced Segregation and Precipitation in Undersaturated Solid Solutions: Arnoldo Badillo[1]; Daniel Schwen[1]; Robert Averback[1]; *Pascal Bellon*[1]; [1]University of Illinois

Current phase field models for diffusion-controlled evolutions in the solid state are based on phenomenological kinetic equations. The lack of absolute time and space scale raises problems when applying these models to alloys subjected to irradiation by energetic particles since this external forcing introduces new length scales and time scales. We propose here an approach that relies on a mixed continuous-discrete treatment of the evolution of chemical species and point defect concentrations. This approach makes it possible to take into account important irradiation effects, namely the production and elimination of point defects and point defect clusters and the forced chemical mixing. Examples of application of the model are given, in particular for heterogeneous segregation and precipitation reactions induced by irradiation in undersaturated solid solutions.

9:00 AM
Modeling Nanocluster Formation during Ion Beam Synthesis: *C. Yuan*[1]; Diana Yi[1]; Ian Sharp[2]; Swanee Shin[1]; Christopher Liao[1]; Julian Guzman[1]; Joel Ager III[3]; Eugene Haller[1]; Daryl Chrzan[1]; [1]Lawrence Berkeley National Laboratory; Department of Materials Science, University of California, Berkeley; [2]Walter Schottky Institut, Technische Universitat Munchen; [3]Lawrence Berkeley National Laboratory

Ion beam synthesis (IBS) is a technologically important method to produce semiconductor nanocrystals within a solid. The process involves implanting ions into a matrix at concentrations beyond their solubility limit. During IBS, a competition between cluster growth and cluster damage evolves. A model describing the nucleation, growth and fragmentation of clusters during IBS is studied via kinetic Monte Carlo simulations and the self-consistent solution to a set of coupled, mean-field rate equations. It is found that the nanocluster size distribution approaches a steady-state profile, the shape of which depends only on the ratio of the transient enhanced diffusion coefficient to the ion volumetric flux. Fitting observed distributions to theoretical predictions allows one to determine the transient enhanced diffusion coefficient. Estimates of transient enhanced diffusion coefficients so obtained for Ag, Co and Ge in silica are

presented. This research is supported by the Directorate, Office of Science, Office of Basic Energy Sciences of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

9:20 AM

Atomistic Simulation of Diffusion on Grain Boundaries and in Irradiated Metals: *Je-Wook Jang*[1]; Byeong-Joo Lee[1]; [1]POSTECH

The grain boundary (GB) diffusion in bcc-Fe has been investigated using molecular dynamics simulations. Attention was focused on the effect of type and misorientation of GBs on the diffusivity. A new method to avoid the difficulty in assuming the width of GB was developed, and applied to the calculation of GB diffusivity and activation energy for a wide range of GBs with different types and misorientations, including a special twin boundary. The calculated diffusivity was generally in a good agreement with experimental information. The calculated activation energy of GB diffusion was also comparable with that of bulk diffusion. No strong correlation between misorientation angle and diffusivity is observed in a range of 10~40°, but certain difference in diffusivity between planar or normal direction to the GB plane is observed. By performing the simulation using irradiated samples, the effect of irradiation could also be estimated and will be discussed.

9:40 AM

Phase Field Formalism for Modeling Microstructure in Irradiated Materials: Simulation of Void Growth: *Srujan Rokkam*[1]; Anter El-Azab[1]; Paul Millett[2]; Dieter Wolf[2]; [1]Florida State University; [2]Idaho National Laboratory

Void formation in irradiated materials is a subject of great technological importance for the design of high performance structures in nuclear reactors. Here we present a diffuse interface phase field model for nucleation and growth of voids in irradiated materials. The formalism developed herein treats both the nucleation and growth processes simultaneously in a spatially resolved fashion. The defect fluxes and the defect density modulations are formulated using Cahn-Hilliard type description for the vacancy and interstitial concentration fields. The dynamics of void growth are obtained in terms of the evolution of a non-conserved order parameter field, whose evolution is prescribed by a phenomenological Allen-Cahn type equation. The model also accounts for the effect of applied stress, cascade-induced and thermally-induced fluctuations, vacancy-interstitial recombination, and interaction of vacancies and interstitials with lattice sinks. Using the case of pure metals as an example, illustrative results of model capabilities are presented.

10:00 AM

Dislocation Evolution in V-4Cr-4Ti: *David Gelles*[1]; [1]Battelle Pacific Northwest National Laboratory

V-4Cr-4Ti is being considered for application in the first wall of a fusion reactor. V-4Cr-4Ti is a refractory solute strengthened body centered cubic alloy chosen in part for its low swelling characteristics. It has been subjected to a wide range of tests to determine suitability, including irradiation creep, irradiation induced swelling, post-irradiation deformation and thermal creep. A part of that effort has been to perform post-test microstructural examinations in order to better understand dislocation behavior. This report is intended to describe dislocation evolution under irradiation and/or stress along with a number of unusual observations arising from that work, including indications for the operation of the Harper-Dorn thermal creep mechanism at temperatures as high as 800°C and a novel Burgers vector found following irradiation. Recent results will be reviewed in order to provide a better understanding of dislocation evolution in this material.

10:20 AM Break

10:40 AM Invited

Monte Carlo Simulations of Irradiated Materials on the Reactor Timescales: *Vasily Bulatov*[1]; Aleksandar Donev[1]; [1]Lawrence Livermore National Laboratory

Irradiation produces copious quantities of atomic defects giving rise to complex diffusion-controlled processes defining the evolution of material microstructure. The time scales on which this evolution takes place range from nanosecond time intervals of fast atomic diffusion to tens of years of material work life in nuclear reactors. Here we present a new method for kinetic Monte Carlo simulations that encompasses all relevant time scales and connects the fundamental atomistic mechanisms directly to the long-term damage accumulation. The new simulation method is tested on a simple model of a-iron for a range of temperatures and irradiation dose rates. The results suggest an approximate scaling relationship by which material damage observed in accelerated irradiation tests can be extrapolated to predict how the same material would resist irradiation over the much longer time scales of nuclear reactors.

11:10 AM

Atomistic Simulations of Radiation Damage in Polycrystalline Metals: *Hanchen Huang*[1]; [1]Rensselaer Polytechnic Institute

Much has been learned about atomistic mechanisms of radiation damage in single crystalline solids. Meanwhile, grain boundaries have usually been assumed as sinks and sources of infinite strength and constant position and character, such as in typical rate theories. Such assumption is certainly unwarranted particularly when solids are nanostructured. Combining classical molecular dynamics and kinetic Monte Carlo based ADEPT simulations, this work presents atomic view of grain boundary effects to radiation damage and effects on grain boundaries from radiation-produced defects.

11:30 AM

Point Defect Clusters in Zirconium and Their Influence on Radiation Damage: *Alexandre Legris*[1]; Petrica Gasca[1]; Christophe Domain[2]; [1]University of Sciences and Technologies of Lille; [2]Électricité de France, Research and Development

The cladding material in Pressure Water Reactors is made with zirconium alloys that are submitted to intense neutron radiation damage in service conditions. As a consequence, the formation of dislocation loops evenly distributed in the basal and prismatic planes induce an elongation of the textured material. To understand the growth and more generally the microstructure evolution under irradiation it is crucial to know the relative stability of the point defect clusters formed which are sinks for point defects and nucleation sites for dislocation loops. The present work presents an ab initio study of small point defect clusters in zirconium including self-interstitials and vacancies. The structure (number of defects and shape) and formation energy of the clusters were determined. The results are discussed and used as input for mesoscopic simulations of radiation damage.

11:50 AM

Modeling of Diffusion in Fe-Ni-Cr Alloys Using Ab-Initio Based Approach: *Samrat Choudhury*[1]; Julie Tucker[1]; Benjamin Swoboda[1]; Dane Morgan[1]; [1]University of Wisconsin

For more than three decades materials used in nuclear reactors have been known to degrade in radiation environments. Radiation changes the materials composition through the formation and migration of large concentration of point defects to sinks. Central to the understanding of such radiation induced segregation (RIS) is explaining the complex solute-defect interaction in multi-component alloys. Prior theoretical models to study diffusion in multi-component alloys often lack adequate energetic parameters of the diffusing species. In this work, we use ab initio energitics to calculate solute-defect interaction in Fe-Ni-Cr alloys. Both ferritic and austenitic structures are considered. We observe in fcc: 1) Strong Cr-interstitial binding 2) Weak binding of Cr and Fe to vacancies and 3) Enhanced Cr diffusion compared to Ni and Fe. Diffusion coefficients for both vacancy and interstitial migration were determined from the ab initio energetic using statistical mechanics and kinetic Monte Carlo approaches.

12:10 PM

Radiation-Induced Segregation in Ferritic-Martensitic Alloys HT9, T91, and HCM12A: *Janelle Penisten*[1]; Zhijie Jiao[1]; Gary Was[1]; Kwan Wong[2]; Brian Wirth[2]; [1]University of Michigan; [2]University of California, Berkeley

Ferritic-Martensitic (F-M) alloys are candidates for cladding and structural material in the Advanced Burner Reactor. However, there is little understanding of radiation-induced segregation (RIS) in these alloys, although Cr segregation is of particular concern. Samples of T91 (9wt% Cr), HT9 (12wt% Cr), and HCM12A (11wt% Cr) were irradiated with 2.0 MeV protons at 400°C and 500°C to doses of 3, 7, and 10 dpa. Prior austenite grain boundary (PAGB) compositions were measured with scanning transmission electron microscopy with energy dispersive X-ray spectroscopy (STEM/EDS). Preliminary results from the 400°C irradiation series show that Cr depletes at PAGBs in both HT9 and HCM12A, and enriches in T91. These results suggest that RIS behavior in F-M alloys may depend on the Cr composition. Results of RIS analysis on PAGB and packet boundaries at multiple doses and at both irradiation temperatures will be discussed in the context of the atomistic-based models of Cr segregation.

Nanocomposite Materials: Nanoparticle Synthesis

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee, TMS: Nanomaterials Committee

Program Organizers: Jonathan Spowart, US Air Force; Judy Schneider, Mississippi State University; Bhaskar Majumdar, New Mexico Tech; Benji Maruyama, Air Force Research Laboratory

Monday AM Room: 3020
February 16, 2009 Location: Moscone West Convention Center

Session Chairs: Benji Maruyama, US Air Force; Jonathan Spowart, US Air Force

8:30 AM Introductory Comments

8:40 AM Invited

Synthesis and Properties of DWCNT Composites: *David Lashmore*[1]; [1]Nanocomp

The CVD growth of dual wall carbon nanotube sheets or textiles will be described. This growth process has been shown to produce very strong stand alone sheets or non-woven textiles that can be aligned after their growth. This post alignment process has a profound effect on the electronic and mechanical properties of the sheets. For example breaking strength for the randomly aligned as grown material is about 200 to 300 MPa, but following alignment breaking strength can increase to over 1 GPa. Similar changes in electronic properties will also be presented. The fabrication of composite materials from these sheets involves a high pressure infusion of the matrix. Data on elastomeric composites that exhibit a fracture toughness of over 60 J/gram will be described along with a number of practical applications both in the electronic area and for structural components.

9:05 AM

A Comparative Study on the Morphology of Strontium Hexaferrite Nano Particles Synthesis by Co-Precipitation Method and Modified Flux Method: *Sachin Tyagi*[1]; [1]Indian Institute of Technology Roorkee

In the present study single phase M-type Strontium Hexaferrite nano crystals that is SrFe12O19 were synthesized by Co-Precipitation and Modified Flux Method. Heat treatment conditions played an important role in the formation of pure SrFe12O19 hexaferrite phase. Conventional heat treatment produced α-Fe2O3 and M phase for the particles synthesized by Co-Precipitation Method whereas Modified Flux Method produced single pure M- phase as confirmed by the X-ray diffraction (XRD). Surface morphology of non porous ultra fine particles has been examined by SEM. The material was annealed at 800°, 900° and 1000°C for 6 hours and its effect on the particles size is also studied. Room temperature magnetic properties were investigated using a Vibrating Sample Magnetometer (VSM) and decomposition behavior therein was investigated by means of thermal analysis (DTA/DTG/TG).

9:25 AM

Nano-Aluminum Based Polymeric Composites: *Christopher Crouse*[1]; Stephanie Johnson[2]; Jared Boock[2]; C. Michael Lindsay[2]; Jennifer Jordan[2]; Jonathan Spowart[1]; [1]Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson Air Force Base; [2]Air Force Research Laboratory, Munitions Directorate, Eglin Air Force Base

Mass transport between reacting species typically governs the reaction kinetics in most energetic systems. The kinetics can be increased by manipulating specific features of the reactant materials such as overall surface area thereby reducing the diffusion distance between reactants. Ensuing from their large surface areas, nanoparticles have become promising candidates for energetic applications. Towards this end we have explored the development of nanoparticulate based composite materials with energy stored in the form of reactive metal nanoparticles (e.g. aluminum and nickel) capable of liberating energy through either a thermitic process or intermetallic formation. Specifically we have focused on developing a chemical route towards the preparation of these materials through manipulation of the nanoparticle surface to allow for physical and chemical intimacy within a polymer matrix. Our initial efforts towards the incorporation of aluminum nanoparticles within a poly(methyl methacrylate) matrix at varying volume percentages will be presented.

9:45 AM

Microstructure and Photoluminescence of NiO/Ni Core-Shell Nanorods on a Silicon Substrate: *Chien-Ming Liu*[1]; Chih Chen[1]; [1]National Chiao Tung University

One-dimensional Ni/NiO core-shell nanorods with an average diameter around 70 nm were grown using anodic alumina oxide (AAO) as a template on a silicon substrate. First, area arrays of Ni nanorods were grown by electroless plating in the AAO pores. Then the Ni nanorods were oxidized in air to grow the Ni oxide on the surface of the Ni nanorods. After the oxidation process, the Ni nanorods were capped with a thin layer of NiO shell. The oxide layer was about 2-10 nm thick and the NiO shell consists of poly-crystals. Photoluminescence spectrum shows emission peak at 375 nm, which suggests that the NiO/Ni core-shell nanorods have potential application as UV a sensor.

10:05 AM

Synthesis Routes for the Production of Nanoscale Tungsten Powder: *K. Scott Weil*[1]; Curt Lavender[1]; Lee Magness[2]; [1]Pacific Northwest National Laboratory; [2]Army Research Laboratory

Bench-scale testing has shown that tungsten nanocomposites may display the type of mechanical behavior required for use in a range of applications, including use in materials processing tools such as friction stir welding tips and long-life welding electrodes, as well as in munitions. However for these materials to be seriously considered for deployment, precursor fabrication (i.e. nanoscale powder synthesis) must be validated using approaches that are scaleable to high-volume production. We will present results from our recent efforts to produce nanoscale tungsten powder by three different approaches and discuss the viability for each in terms of powder purity, size, and morphology; process scalability; and high density powder consolidation practice.

10:25 AM Break

10:40 AM Invited

High-throughput Nanomaterial Fabrication, Characterization, and Consolidation: *Christopher Haines*[1]; Deepak Kapoor[1]; Darold Martin[1]; [1]US Army ARDEC

ARDEC has established a pilot-scale facility for the fabrication, characterization, and consolidation of a wide range of nanomaterials. We employ inductively-coupled plasma and inert gas condensation to synthesize nanoscale powders in the 20 – 200 nm size range and a "top-down" milling approach to fabricate nanostructured powders. The versatility and high-production rate of our systems allow us to produce kilogram quantities of nanoscale and nanostructured powders of various metals and alloys, ceramics, cermets, and energetic materials. Fully-integrated, computer control of processing parameters provides the ability to precisely control the particle size and distribution of nanoscale powders. Beyond traditional characterization techniques such as x-ray diffraction and electron microscopy, we employ both small angle x-ray scattering (SAXS) and ultra-small angle x-ray scattering (USAXS). Processing of nanoscale and nanostructured powders to fully dense bulk nanostructured materials poses a much bigger challenge; therefore, novel powder consolidation techniques are being developed to overcome this issue.

11:05 AM

Industrial Production of Nanoparticle Masterbatches: Steffen Pilotek[1]; *Kerstin Grosse*[1]; [1]Buhler Inc.

Inorganic oxide nanoparticles may be introduced into liquid product formulations using dispersions of high concentration. Due to the large specific surface area of colloidal systems, the compatibility of particle dispersion and product formulation needs to be specifically addressed. We use the chemomechanical process to produce nanoparticle masterbatches in industrial scale. In the process, agglomerated nanostructured powders are surface modified under well defined mechanical stress conditions. It enables the production of dispersions up to 60 wt.-% with a particle size of well below 100 nm. The chemical surface modification is a key component in providing a valuable masterbatch. The approach allows for chemical functionalization of the particles. The technical challenge lies in finding the right overall process parameters to manufacture a dispersion that is compatible and thus functional with respect to the product formulation. As reaction compartment, agitator bead mills are used which ensures that the masterbatches are available in ton-scale.

11:25 AM
Preparation of Porous Ultra-Fine Fiber Fe-Ni Alloy Powder Precursor by Coordinated Co-Precipitation-Direct Reduction Process: *Zhang Liang*[1]; [1]Central South University

The precursor, prepared by coordinated co-precipitation with FeSO4 and NiSO4 as the raw materials, oxalate as the precipitator, and ammonia as the coordinator, was direct reduced by hydrogen to obtain porous ultra-fine fiber Fe-Ni alloy powder. The effects of parameters such as the concentration of reactants, pH value, additives, and reaction temperatures for precursor preparation and the reductive temperature, the composition of reductive atmosphere amid the reduction process were systematically investigated. The structure, thermo-decomposition process and surface morphologies of the alloy powder derived from thermal reduction of the precursors were characterized by FTIR, XRD, TG/DSC and SEM. The experimental study shows that using 1(wt)%PVP as additive, well-dispersed precursors with a uniform morphology can be obtained in a solution with Fe2+ and Ni2+ concentration of 0.8 mol/L , pH value of 6.1 at 60°.

11:45 AM
Microscopic and Spectroscopic Characterization of Cryomilled Nanostructure of Aluminum Alloy and B4C Powder: *Clara Hofmeister*[1]; Bo Yao[1]; Helge Heinrich[1]; Yongho Sohn[1]; Cory Smith[2]; Mark van den Bergh[2]; Kyu Cho[3]; [1]University of Central Florida; [2]DWA Aluminum Composites; [3]US Army

Extensive attention has been paid to the production of tri-modal aluminum alloy composites that possess excellent strength and impact resistance. We have examined the microscopic and spectroscopic characteristics of nanostructured Al-5083 and B4C powder blends produced via large commercial scale cryomilling in liquid nitrogen. A blend of a commercial grade prealloyed Al-5083 and a commercial grade B4C powders were used as a precusor for the cryomilling. X-ray diffraction, scanning electron microscopy, transmission electron microscopy, X-ray photoelectron spectroscopy, Auger electron spectroscopy, laser dynamic scattering, and BET measurement were employed. Results from microstructural and spectroscopic characterization are presented and discussed with respect to production of tri-modal aluminum alloy composites.

12:05 PM
Preparation of Ultra-Fine MgO•Al2O3 Spinel Powder and Its Metallurgy Behavior in Low Carbon Steel: *Yang Li*[1]; Wei-Jian Li[1]; Liang-You Wang[1]; Zhou-Hua Jiang[1]; [1]Northeastern University

Micron, sub-micron and nanometer sized MgO•Al2O3 ultra-fine powder were prepared by gel precipitation, solid-phase synthesis, sol-gel and flame throwing pyrogenation methods. XRD analysis shows that the all of the ultra-fine powder is pure with a single MgO•Al2O3 spinel phase. The size is measured by laser granularity analyzer and the average size is 60, 505 and 1780 nm with quite uniform distribution. MgO•Al2O3 spinel powder with different granularity were sprayed into molten low carbon steel in MgO crucible and MoSi2 furnace at 1873 K. Quantitative microscopic examination shows that big particle inclusions are reduced and small particle inclusions increased, and the average size is reduced. Data comparison from spraying different size powders shows that spraying MgO•Al2O3 of nanometer tends to cause more small inclusions. The sprayed steel samples were rolled and heat treated for the mechanical properties tests, which shows spraying nanometer MgO•Al2O3 is the best way to improve mechanical property.

Neutron and X-Ray Studies of Advanced Materials: Resolving Local Structure

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Titanium Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter Liaw, The University of Tennessee; Jaimie Tiley, US Air Force

Monday AM — Room: 3016
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Andrea Gerson, University of South Australia; Wolfgang Pantleon, Risoe DTU

8:30 AM Keynote
At the Limit of Polychromatic Microdiffraction: *Gene Ice*[1]; Bennett Larson[1]; Jonathan Tischler[1]; Jae-Young Choi[2]; Wenjun Liu[3]; Deming Shu[3]; Ali Khounsary[3]; [1]Oak Ridge National Laboratory; [2]Pohang Accelerator Laboratory; [3]Argonne National Laboratory

With a high-energy 3rd generation source like the Advanced Photon Source (APS) it is possible to push the performance of polychromatic microdiffraction far beyond current levels and to approach the intrinsic limit of the technique based on sample damage and the diffraction limit of x-rays. We describe ongoing efforts to improve the spatial, temporal and momentum transfer resolution of polychromatic microdiffraction on beamline 34-ID-E at the APS. The goal of this effort is to provide high-resolution images of 3D crystal structures over sufficient volumes and with sufficient detail to understand the underlying physics of inhomogeneous mesoscale structural evolution. The performance of a new detector system and the development of more advanced focusing optics will be described and discussed in light of the ultimate limits set by the physics of x-rays and materials and in light of opportunities to field specialized insertion devices and optics for polychromatic microdiffraction.

9:00 AM Invited
Evolution of Deformation Structures under Varying Loading Conditions Followed In-Situ by High Angular Resolution 3DXRD: *Wolfgang Pantleon*[1]; Christian Weidemann[1]; Ulrich Lienert[2]; Bo Jakobsen[3]; Henning Poulsen[1]; [1]Risoe DTU; [2]Argonne National Laboratory; [3]Roskilde University

With the high angular resolution three-dimensional x-ray diffraction method (established at APS), individual subgrains are detected in the bulk of polycrystalline specimen and their dynamics is followed in-situ during varying loading conditions. Analysing the intensity distribution of a single Bragg reflection from an individual grain in reciprocal space, subgrains can be distinguished by their unique combination of orientation and elastic strain. Responses to different loading conditions are presented: During uninterrupted tensile deformation the subgrain structure develops intermittently. When the traction is terminated, stress relaxation occurs and number, size and orientation of subgrains are found to be constant. The structure freezes and only a minor clean-up of the microstructure is observed. Upon unloading the subgrain structure remains unchanged, but the compressive stresses of the subgrains increase in average. When changing the strain path, a systematic correlation between changes in the dislocation structure and the degree of strain path changes is established.

9:20 AM Invited
Friedel-Pair Based Indexing Method of Individual Grains in Polycrystals Investigated with Hard X-Rays: Marcin Moscicki[1]; Haroldo Pinto[1]; *Andras Borbely*[1]; Anke Pyzalla[1]; [1]Max-Planck Institut für Eisenforschung

A new procedure for characterizing the crystallographic orientation, spatial position and average strain tensor of single grains in the bulk of a polycrystalline sample is presented. It is complementary to existing indexing methods developed within the frame of three-dimensional X-ray diffraction (3DXRD). The algorithm uses detector coordinates corresponding to Friedel reflection-pairs (hkl and -h-k-l) and requires the measurement of diffraction spots in a relatively large interval of about 180°. The advantage of using Friedel pairs resides in their symmetry properties enabling a clear separation of the contributions from grain orientation, grain position and average strain to the position of the diffraction spots measured on a 2D detector. This leads to reduced number of unknown fitting parameters that have to be simultaneously considered and consequently to their higher

accuracy. The method is exemplified on experimental data obtained during in-situ straining of steel wires with 300 μm in diameter.

9:40 AM Invited
In Situ Single Grain Peak Profile Measurements on Ti-7Al during Tensile Deformation: *Ulrich Lienert*[1]; Matthew Miller[2]; Joel Bernier[3]; Matthew Brandes[4]; Michael Mills[4]; [1]Argonne National Laboratory; [2]Cornell University; [3]Lawrence Livermore National Laboratory; [4]Ohio State University

It has recently been demonstrated that the combination of focused high energy synchrotron radiation and area detectors provides a powerful tool for the structural in situ characterization of bulk polycrystalline materials on the single grain length scale. At the APS 1-ID beamline the 3DXRD technique has been extended to high reciprocal space resolution. Thus, not only can average strain tensors of individual grains be measured, but also intra-granular strains and misorientations. Here, measurements on Ti-7Al specimens are reported. Two very different dislocation structures are produced by annealing treatments. As evidenced by TEM, ice water quenching results in a random dislocation structure, while well ordered domains form by slow air cooling. Single grain strain tensors were measured up to 2% tensile deformation, and selected peaks were mapped with high resolution at selected loads and during relaxation. The sensitivity of the technique to the different dislocation structures will be discussed.

10:00 AM Invited
Measuring Local Strains and Composition in Nickel Alloys Using Synchrotron Radiation: *Stewart McIntyre*[1]; Marina Suominen Fuller[1]; [1]University of Western Ontario

To predict the onset of Stress Corrosion Cracking in Alloy 600 requires knowledge of both mechanical and chemical changes. For the strain studies we are using Laue diffraction and micron-scale xray beams from a synchrotron. Difraction patterns for each micron-sized area are indexed to yield maps that show grain orientation, composite elastic strain magnitudes as well as their directional components. Further, streaking of the diffraction spots when present, can be used to estimate the local dislocation density and the direction(s) of the slip systems. Our applications have focussed on local strain effects in polycrystalline Alloy 600 introduced by tensile extension or by placing calibrated scratches in the surface. While our current work uses APS, we are developing the VESPERS beamline at the Canadian Light Source for simultaneous micro-nd XRF measurements.

10:25 AM Invited
X-Ray Micro/Nano-Diffraction for Studies of Individual Nano-Objects: *Wenjun Liu*[1]; Paul Zschack[1]; Matthew Bierman[2]; Song Jin[2]; John Budai[3]; Gene Ice[3]; [1]Argonne National Laboratory; [2]University of Wisconsin-Madison; [3]Oak Ridge National Lab

The rapidly evolving field of x-ray micro/nano-diffraction on 3rd generation synchrotron sources opens up new frontiers in x-ray studies for nano science. Taking advantage of high brightness of the source and state-of-the-art x-ray mirror focusing optics, 3D scanning polychromatic and monochromatic diffraction microscopy developed at 34-ID beamline at the Advanced Photon source (APS) could provide detailed local structural information, such as crystallographic orientation, grain morphology, strain tensor, and lattice structure in nano-materials, with high spatial resolution of 300 nm and angular resolution of 0.2 mrad. Recent applications in nano-materials science include studies of dislocation-driven crystal growth and twist mechanism in PbS pine-tree-like and PbSe helical nanowires, and crystal structural study of ZnO and EuAlO nanorods.

10:45 AM Break

10:55 AM
A Grain-Subdivision Study of a Cyclically-Deformed Nickel-Based Superalloy Using Synchrotron X-Ray Micro-Beam Diffraction: *E-Wen Huang*[1]; Rozaliya Barabash[2]; Gene Ice[2]; Wenjun Liu[3]; Peter Liaw[1]; Chung-Hao Chen[4]; [1]Department of Materials Science and Engineering, University of Tennessee; [2]Oak Ridge National Laboratory; [3]Argonne National Laboratory ; [4]Department of Electrical Engineering and Computer Science, University of Tennessee

A newly-developed nickel-based superalloy is selected to study its fatigue behavior. The current study focuses on the local microstructure changes, which are responsible for the fatigue deformation, using the micro-beam technique at Argonne National Laboratory. The atomic structures of the cyclically-deformed superalloy can be studied as a function of the distance within one grain and from adjacent grains. The polychromatic X-ray microbeam provides the local structural information from the Laue pattern, which reflects the number of geometrically necessary dislocations (GNDs). The Laue patterns demonstrate that the plastic deformation results in the formation of the alternating regions with high and low GND densities. The inhomogeneous plastic deformation of the cyclically-deformed specimen was observed using the synchrotron X-ray. The evolution of the dislocation substructure and local texture orientations within one grain and in the adjacent grains identifies the sequence of structural changes during the cyclic loadings.

11:05 AM Invited
Synchrotron-Based White/Monochromatic Beam Micro X-Ray Diffraction at the Advanced Light Source: *Nobumichi Tamura*[1]; Martin Kunz[1]; Kai Chen[1]; [1]Lawrence Berkeley National Lab

BL 12.3.2 at the Advanced Light Source is a recently commissioned superconducting magnet beamline entirely dedicated to white/monochromatic beam micro X-ray diffraction for the measurement and mapping of strain/stress in engineered materials. The current status of the hardware and software of the beamline will be described and a few chosen applications will be presented.

11:25 AM Invited
The Application of Synchrotron Microdiffraction to Identify 3D Strains in High Performance Steel: *Andrea Gerson*[1]; Ning Xu[1]; Joe Cavallaro[1]; [1]University of South Australia

The excellent mechanical properties achieved by modern steels are, in most part, attributable to advances in the thermal processing. Recently, with the advent of highly focused, high flux density X-ray beams from third generation synchrotron sources the study of the phase changes which take place during the processing of steels can be studied with a high degree of spatial resolution. We report on monochromatic synchrotron microdiffraction mapping of cryogenically treated tool steel (Advanced Photon Source synchrotron, end-station 34ID-E). Significant spatial inhomogeneity is apparent. The mean of each of the martensitic diffraction peak's FWHM for the most rapidly cooled cryogenically treated sample were the smallest of the samples examined. This treatment also displayed the smallest and most narrow d-spacing distribution. These results suggest that rapid cryogenic cooling results in better formed, more ordered and denser martensitic crystallites as compared to slow cryogenic cooling or no cryogenic treatment at all.

11:45 AM
The Effect of Residual Stress on Texture and Growth of Oxide Scale on Zirconium Alloys: *Philipp Frankel*[1]; Richard Moat[1]; Efthymios Polatidis[1]; Michael Preuss[1]; [1]University of Manchester

Zr alloys are extensively used in PWRs as nuclear fuel cladding and structural fuel assembly components. The performance of the cladding material is strongly affected by its corrosion properties. Therefore, better understanding of the corrosion mechanisms is key to improving the degree of "burn-up" that can be sustained. This work investigates the effect of residual stresses on the crystallographic relationship between zirconium alloys and zirconium oxide formed in steam at elevated temperatures. It is believed that this relationship is very important to the growth kinetics of the oxide. High-Energy synchrotron X-ray measurements have been carried out at the ESRF, (Grenoble, France) with a ~1 micron spot size which allowed mapping of the residual stresses and texture in cross-sectional samples across the interface between the metal and the oxide. Therefore, texture of the oxide could be related to that of the metal and the residual stresses with distance from the interface.

12:00 PM Invited
Spatially Resolved Elastic Strains within Bulk Dislocation Cell Structures: Measurements and Models: *Lyle Levine*[1]; Ben Larson[2]; Francesca Tavazza[1]; Jon Tischler[2]; Peter Geantil[3]; Michael Kassner[3]; Wenjun Liu[4]; [1]National Institute of Standards and Technology; [2]Oak Ridge National Laboratory; [3]University of Southern California; [4]Argonne National Laboratory

The existence and magnitude of long range elastic strains (and thus stresses) in dislocation cell interiors and walls in deformed metals have been the subject of extensive investigation for more than 20 years. We have used depth-resolved, submicrometer X-ray beams to directly measure the axial elastic strains within numerous *individual* dislocation cell interiors and cell walls in plastically deformed copper single crystals. As previously reported, the average cell interior strains are tensile in unloaded compression specimens and compressive in unloaded tensile specimens. Recent measurements from individual, buried

cell walls show that these have the reverse average strains. Most significantly, all of these cell interior and cell wall strains exhibit large cell-to-cell variations reminiscent of the misorientations across cell walls. The experimentally determined distribution functions describing these strain fluctuations will be presented along with theoretical models that explain their origin. Finally, local spatial correlations in the elastic strains will be discussed.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Fundamental Properties, Interfacial Reactions and Phase Transformation

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee

Program Organizers: Sung Kang, IBM Corp; Iver Anderson, Iowa State University; Srinivas Chada, Medtronic; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Albert Wu, National Central University

Monday AM — Room: 2020
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Sung Kang, IBM Corp; Jeng-Gong Duh, National Tsing-Hua University

8:30 AM Introductory Comments by Sung K. Kang

8:35 AM Invited

The Next Phase in Pb-Free Solder Development in Electronic Packaging: *Darrel Frear*[1]; [1]Freescale Semiconductor

Electronic Packaging has undergone tremendous change as a result of legislative actions restricting hazardous materials. One of the key elements targeted is Pb in eutectic Sn-Pb solder used to attach electronic packages to circuit boards. Through extensive efforts \a variety of Pb-free solder alloys were successfully implemented for board attach applications. The next challenge is the extension of Pb-free solders for flip chip and die attach applications that use Pb-rich Sn-Pb solder and are currently exempted from legislative bans because there were no identified Pb-free solutions. There are still no drop-in Pb-free replacements for Pb-rich Sn-Pb but there are a number of innovative potential solutions ranging from new conductive adhesives to composite solder alloys to the complete elimination of solder as an interconnect. An overview of these materials and their performance will be presented along with a summary of future work required to eliminate all Pb from electronic packages.

8:55 AM

Preferred Orientation Relationships between Intermetallic Compounds and Substrate Metals in Reactive Wetting Reactions between Molten Sn-Based Solders and Metals: *Jong-ook Suh*[1]; King-Ning Tu[2]; Nobumichi Tamura[3]; [1]Jet Propulsion Laboratory, California Institute of Technology; [2]University of California, Los Angeles; [3]Lawrence Berkeley National Laboratory

Crystallographic orientation relationships between intermetallic compounds and substrate metals are discovered by synchrotron micro x-ray diffraction studies. Cu6Sn5 has six different preferred orientation relationships with Cu, which can be categorized into two groups due to pseudo-hexagonal crystal structure of the Cu6Sn5. Ni3Sn4 has two types of preferred orientation relationships with Ni. If a single crystal metal with proper orientation is used as a substrate, morphology of intermetallic compounds can be greatly altered due to the preferred orientation relationships. Amount of the misfit between Ni3Sn4 and Ni is greater than the misfit between Cu6Sn5 and Cu. The difference in misfit explains morphological difference between Cu6Sn5 and Ni3Sn4. The present study suggests that the existence of preferred orientation relationships can be a general mechanism in intermetallic compound formation by reactive wetting.

9:10 AM

Interfacial Reaction and Thermal Cycling Reliability of Zn-Sn High Temperature Lead-Free Solders: *Seongjun Kim*[1]; Keun-Soo Kim[1]; Goro Izuta[2]; Katsuaki Suganuma[1]; [1]Osaka university; [2]Mitsubishi Electric Corporation

High temperature solders containing 85~97 wt.% Pb have been widely used as die-attach solders in the power electronics packaging. In contrast to the middle temperature solders such as Sn-Ag-Cu alloy, however, little research has been done on high temperature lead-free solders to replace the high Pb bearing solders. In the previous study, we suggested Zn-xSn (x=20, 30, and 40 wt.%) solders as one of the best candidates. To evaluate the possibility of this alloy further, we have investigated the interfacial reactions and thermal cycling reliability of a Si die attached joint with Zn-xSn solders. Si die (Au/TiN/Si) attachment was carried out on the direct copper bonded (DCB) substrates and direct aluminum bonded (DAB) substrates, which are used in a variety of power electronic systems. The changes of interface microstructure and joining strength were examined throughout the thermal cycling test up to 2000 cycles.

9:25 AM

Modification of the Interface Microstructures of Sn-3.5Ag/Cu Solder Joints by Zn Electroplating: *Youngkun Jee*[1]; Jin Yu[1]; [1]KAIST

A Cu UBM is widely used as a surface finish for lead-free solder joints, however fast consumption of Cu, rapid growth of IMC and formation of Kirkendall voids pose serious reliability concern. In our previous study, additions of Zn to Sn-3.5Ag solder increased the reliability by replacing Cu-Sn IMC by Cu-Zn IMCs. The beneficial effects of Zn can be also achieved by modifying UBM rather than the solder composition, which is simpler and cheaper. In this study, the interface microstructure of Sn-3.5Ag / Cu joint was modified by electroplating varying amount of Zn on Cu UBM. As the amount of Zn dissolved in Sn-3.5Ag solder increased with the electroplated Zn thickness, Cu-Sn IMCs such as Cu6Sn5 and Cu3Sn were replaced by Zn-containing IMCs such as Cu5Zn8 and Ag5Zn8, which increased the drop reliability of solder joints significantly. Then, the results were compared with those of Sn-3.5Ag-xZn / Cu solder joints.

9:40 AM

Diffusion Behaviour of Zn during Reflow of Sn-9Zn Solder on Ni/Cu Substrate: Jagjiwan Mittal[1]; Shih-Ming Kuo[1]; Yu-Wei Lin[1]; *Kwang-Lung Lin*[1]; [1]National Cheng Kung University

Reflow behaviour of Sn-Zn solder on Ni/Cu substrate was investigated using different reflow conditions of 230°C in SMT scope. EDX studies of the reflowed samples showed high diffusion of Zn from solder to the intermetallic compounds (IMC) layer from 9 to 41 atomic percentages with the decrease in heating rates from 180°C/min to 90°C/ min. Results demonstrated high interfacial activity and affinity of Zn in the formation of IMC. Reasons of this diffusion are related to the higher reactivity and smaller size of zinc in comparison to tin, phase separation during heating and affinity of Zn to form NiSnZn and NixZny intermetallic compounds. Detailed process study, possible mechanism and other related aspects will be presented

9:55 AM

The Effect of Thickness of Cu-Zn Solder Wetting Layer on the Intermetallic Growth: *Youngmin Kim*[1]; Changyul Oh[1]; Hee-Ra Roh[1]; Young-Ho Kim[1]; [1]Hanyang University

Cu-Zn solder wetting layers developed recently were proven to suppress the excessive growth of intermetallic compound and formation of Kirkendall voids in Sn-Ag-Cu/Cu-Zn system. In this study, the effect of the thickness of the Cu-20wt% Zn layer has been investigated by varying the thickness of Cu-Zn layers ranging 2 to 10 μm. After reflowing Sn-4.0Ag-0.5Cu solder balls onto Cu-Zn layers, these were aged. The granular-like Cu6Sn5 was formed at the Sn-4.0Ag-0.5Cu/Cu-Zn interface after reflow. As the thickness of Cu-Zn wetting layer increased, the IMC growth rate decreased. A typical bi-layer (Cu6Sn5 and Cu3Sn) was formed on the Cu or Cu-Zn (2 μm) layer during aging. When the 6 or 10 μm-thick Cu-Zn layer was used, it was not completely dissolved and Cu3Sn, Kirkendall voids were not formed even after aging up to 1000 hrs. Since the interdiffusion between Sn and Cu was suppressed due to Zn in the wetting layer.

10:10 AM

Effect of Amount of Cu on the Intermetallic Layer Thickness between Sn-Cu Solders and Cu Substrate: *Md. Alam*[1]; S. M. L Nai[1]; Manoj Gupta[1]; [1]National University of Singapore

In the present study, Sn-Cu solders were synthesized using pure tin with varying volume fraction of nano size copper (0% vol., 0.2 % vol., 0.35% vol., 0.7% vol. and 1.1 % vol.) by powder metallurgy route incorporating microwave assisted sintering. Intermetallic compound (IMC) layer formation between Sn-Cu solders and Cu substrate were investigated following reflow process. Samples were prepared by heating at 250 °C using hot plate. Results revealed that IMC layer thickness decreases with the addition of nano copper up to 0.35 vol. %. Beyond 0.35 vol. % copper addition, IMC layer thickness started to increase and maximum IMC layer thickness was found for Sn with 1.1 vol. % Cu. An attempt

is made in this study to correlate the effect of nano copper addition on the IMC layer thickness.

10:25 AM Break

10:40 AM Invited
A Mechanism of Kirkendall Void Formation in Cu/Sn-3.5Ag Solder Joint: *Jin Yu*[1]; Jong Yeon Kim[1]; [1]KAIST

In our previous work, residual S from the SPS additive in Cu electroplating bath was shown to play critical roles in Kirkendall void formation. Segregation of S to Cu/Cu3Sn interface lowered the interface energy thereby localizing Kirkendall voids at the interface. Once nucleated, Kirkendall voids can grow without the presence of external load leading to catastrophic drop failure of joints. In this work, a quantitative model on the Kirkendall void growth is presented based on classical Darken's analysis and the diffusive growth of cavities.

11:00 AM
Reaction Mechanism and Mechanical Property for the Flip Chip Sn-3.0Ag-0.5Cu Solder Bump with Ti/Ni-Cu/Cu under-Bump Metallization after Various Reflows: *Chung-Nan Peng*[1]; Jeng-Gong Duh[1]; [1]National Tsing Hua Univ

Ni under bump metallization (UBM) has been widely used as the diffusion barrier between solder and Cu pad. In order to retard the fast dissolution rate of Ni UBM, the cupper was added into Ni thin film. It is expected that the Ni-Cu UBM could provide extra Cu into solders to maintain Cu6Sn5 IMC in the interface. Therefore, it could significantly decrease the Ni dissolution rate.In this study, the Cu content of the sputtered Ti/Ni-Cu/Cu UBM was varied from 0 to 20 at.% in Ni-Cu UBM. Sn-3Ag-0.5Cu solder was reflowed with Ti/Ni-Cu/Cu UBM for 1, 5, and 10 times. The amount of (Cu,Ni)6Sn5 increased with increasing Cu contents in the Ni-Cu film. Cu concentration of the IMC was strongly dependent on the composition of the Ni-Cu films. The relationships between microstructure and strength of the solder with various reflow times were investigated and discussed.

11:15 AM
Interfacial Reactions in the Au/Sn/Cu Sandwich Couples: *Ching-feng Yang*[1]; Sinn-wen Chen[1]; [1]National Tsing-Hua University

Au bumps are commonly used in the flexible electronic products. Cu tracks on flexible substrates protected by Sn surface finish are attached to the Au bumps, and the Au/Sn/Cu three-layer structure is thus frequently encountered. Interfacial reactions in the Au/Sn/Cu sandwich couples at 210°C were examined. The thickness of the Sn layer varied from 5, 7, 27 to 31μm. At the Sn/Cu interface, the phases formed were $(Cu,Au)_6Sn_5$ and Cu_3Sn. The phases formed at the Au/Sn interface were affected by the Sn layer thickness. Initially, AuSn, $AuSn_2$, and $AuSn_4$ phases were formed when the Sn layer thickness was 7μm. When the Sn layer was 27 and 31μm, $(Cu,Au)_6Sn_5$ phase was observed not only on the Sn/Cu side but also on the Au/Sn side. Besides, a ternary $Au_{25}Sn_{50}Cu_{25}$ phase is found in the $AuSn_4$ matrix. Electromigration effect on Au/Sn/Cu interfacial reations is studied as well.

11:30 AM
Interfacial Reactions between In-Sn Solder and Ni-Fe Platings: *John Daghfal*[1]; J. Shang[2]; [1]Institute of Metal Research; [2]University of Illinois at Urbana-Champaign

Ni-Fe platings are attractive device metallizations because of their unique thermal properties and good solderability. In this study, the interfacial reactions between the eutectic In-Sn solder alloy and Ni-Fe platings were examined as a function of reaction temperature, time and Fe content. Both the type and thickness of the reaction phases were found to depend strongly on Fe content in the Ni-Fe platings. Upon thermal aging, Fe-Sn intermetallic compound showed much slower growth kinetics than Ni-Sn compound. The reaction products were analyzed by considering both nucleation and growth of intermetallic compounds.

11:45 AM
Application of Cu-RuN Film as a Diffusion Barrier for UBM in the Electronic Packaging: *H. Y. Chuang*[1]; C. H. Lin[2]; J. P. Chu[3]; C. Kao[1]; [1]National Taiwan University; [2]Chin-Min Institute of Technology; [3]National Taiwan University of Science and Technology

This paper reports the dissolution behavior of the RuN-bearing Cu film deposited by cosputtering. This copper film exhibits an extremely low dissolution rate in SAC solder. The consumption of the Cu-RuN film after 120 sec. is about 10 times lower than that of pure Cu. The dissolution resistance of Cu-RuN is even better than that of the commercial Ni-P layer. Consequently, this film is potentially useful as a diffusion barrier for under bump metallization. For a better understanding of the new method, thermal aging and wettability tests are also studied in the present work.

12:00 PM
The Peltier Effects upon Interfacial Reactions in the Soldering System: *Chao-hong Wang*[1]; Sinn-wen Chen[2]; [1]National Chung Cheng University; [2]National Tsing Hua University

Interfacial reactions in the sandwich-type Sn/Co/Sn couples at 180°C were examined with and without passage of electric current. Only the $CoSn_3$ phase was formed at both the Sn/Co and Co/Sn interfaces. The thickness of the reaction layer at the Sn/Co interface where electrons flew from Sn to Co was similar to that in the couple without current; however, it was thinner than that at the Co/Sn interface where electrons flew from Co to Sn. Since the Sn flux resulted from electromigration was not in the same direction as that caused by the chemical potential gradients, the fact that it was thicker at the Co/Sn interface could not be explained by the electromigration effect. It was found that the temperature was higher at the Co/Sn interface and its difference was caused by the Peltier effect. It should be the reason why the reaction layer was thicker at the Co/Sn interface.

12:15 PM
Interfaces of Tin with Al-Cu-Fe Quasicrystalline Phase and Its Effect on Solidification Behavior of Tin: *Alok Singh*[1]; Hidetoshi Somekawa[1]; An Pang Tsai[2]; [1]National Institute for Materials Science; [2]Tohoku University

Tin particles of micron size were embedded in $Al_{63}Cu_{25}Fe_{12}$ quasicrystal matrix by rapid solidification and annealing. Interfaces were studied by TEM. Each tin particle made faceted interfaces with several quasicrystal grains in various orientations. At least five orientation relationships were determined, which matched close packed Sn planes 200, 101, 220 or 211 with fivefold or twofold planes of the quasicrystalline phase. Solidification behavior of the tin particles was studied by DSC. Tin particles showed multiple, mainly three, solidification peaks in the range 205°C to 185°C. Solidification nucleus contact angles were calculated to be 9.5°, 11° and 14°. In contrast, when the matrix phase was transformed to a microcrystalline phase, solidification involved only one peak, at about 192°C, with a contact angle of 11°. Interface structures and solidification behavior are compared with those reported for aluminum matrix. Interface formation and its effect on solidification behavior will be discussed.

Peirce-Smith Converting Centennial Symposium: Historical Foundations/Refractory Practices

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
Program Organizer: Joël Kapusta, Air Liquide

Monday AM — Room: 2009
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Joël Kapusta, Air Liquide; Tony Eltringham, BHP Billiton Base Metals

8:30 AM Welcome Address

8:40 AM Keynote
William Peirce and E.A. Cappelen Smith and Their Amazing Copper Converting Machine: *Larry Southwick*[1]; [1]LM Southwick & Assoc

This Peirce-Smith Converting Centennial symposium is celebrating the contributions of two men, William Peirce and E.A.C. Smith, in advancing technology allowing the copper industry to realize its full potential. However, theirs is representative of a larger story: New, simpler technology releasing the stranglehold of older, more complicated technology, new developments in vessel configuration and design finding alternate routes around dead ends in operability, advances in technique and concepts removing roadblocks of cycle time and capacity, and overall innovation opening up vast new reserves around the world to those companies willing to embrace those improvements. Our story is also one of personalities, stubborn smeltermen versus the innovators, those inside the industry versus those from outside. It is a story of an initial borrowing from

the steel industry, but also Peirce and Smith, from the refining end of the copper business, taking the ideas of Baggaley from Pittsburgh's steel and airbrake industry, who built on what Hollway, Manhès and David, Douglas, and others had done in smelter tests, further to compete with the previous primacy and closely held expertise of the copper smeltermen in Wales. These advances have been well recorded in photographs taken over the last 150 years. A number were submitted especially for this symposium. Many others were published in the mining and metallurgy press of the times. Several mining schools also retained copies of photos either taken during field trips, or donated to the school by alumni and operating companies. This presentation will provide a selection and discussion from these sources of "converters in action", describing the various designs, operations and developments depicted. The focus here will be on the first roughly 60 years of copper converter development, from Bessemer's days at the dawn of this new idea, up to and slightly past the time of initial adoption of Peirce and Smith's designs.

9:10 AM

Before Peirce and Smith - The Manhes Converter and the Story of Its Development and Some Reflections for Today: Albert Pelletier[1]; Phillip Mackey[2]; Larry Southwick[3]; Albert Wraith[4]; [1]Late of Montreal, Quebec; [2]Xstrata Nickel; [3]LM Southwick & Assoc; [4]A.E. Wraith

In 1881, the first commercial pneumatic Bessemer-type converter treating copper matte was successfully introduced by Pierre Manhes at the Eguilles copper plant near Vedène in France. This development followed over a year of testing on a smaller scale at a foundry in Vedène, during which the vertical tuyeres of the conventional Bessemer converter had been successfully replaced by horizontal tuyeres in an effort to avoid the freezing of copper which had occurred in the bottom of the vessel having vertical tuyeres. The Manhes Converter, as it became known, was the first successful large-scale adaptation of the Bessemer concept of using compressed air blown into a melt for metal refining and treatment. Within a few years, Manhes Converters utilizing horizontal tuyeres in either the original type of vertical converter, or a later and more enduring horizontal barrel converter, were in operation at over a dozen copper smelters around the world. It was this process that Messrs Peirce and Smith essentially improved with the introduction of the larger converter which bears their name and the development celebrated at this Symposium. The present paper briefly traces the original development by Pierre Manhes in the early 1880s and which set the stage for the later Peirce-Smith adaptation. The paper observes that the original development by Manhes was in part driven by the need to reduce coal (energy) consumption and hence the production cost in order for Eguilles to remain competitive with the larger plants in Swansea, Wales, where a ready supply of cheap coal helped keep smelter treatment terms of the day low. These themes are eerily resonant of today where booming copper production in China has impacted the industry, and also where energy consumption, closely linked to today's more familiar theme of 'climate change' remains an important challenge currently confronting the entire industry. The paper concludes with comments on these seemingly parallel situations today and offers some thoughts for the future.

9:30 AM

Conflicts over Designs, Refractories and Awards: The First 20 Years of PS Experience: *Larry Southwick*[1]; [1]LM Southwick & Assoc

This paper covers technical and other developments during the first 20 years of Peirce-Smith converting. There were numerous mechanical and process ideas and concepts already being developed and used by others that had possible application within the converter that Peirce and Smith were moving ahead with. There were also other converter configurations in which the basic lining and larger size concepts could function. Finally, as awards and honors began to accumulate, there were those who felt that contributions of other early workers were being ignored. The working out of these conflicts and differing directions will be described.

9:50 AM

Converting and Refining – Experience in Ferrous and Non-Ferrous Metallurgy: *Theo Lehner*[1]; Caisa Samuelsson[2]; [1]Boliden Mineral AB; [2]Lulea University of Technology

Converting and refining play a central role in the extraction of metals. Sweden has a long tradition as an experimental play ground for metallurgists, chemists and alchemists, developing new methods and technologies. Presenters of papers are usually happy in recording successful developments. But reporting failures can be as great a value as the former. In the paper experiments and experience in process development are illustrated, differences between ferrous and non-ferrous developments are highlighted and industrial learning curves are discussed.

10:10 AM Break

10:30 AM

Chrome-Magnesite Refractory Corrosion with Olivine Slag of High Cuprous Oxide Content: *Carolina Ramirez*[1]; Patricio Ruz[2]; Gabriel Riveros[1]; Andrzej Warczok[1]; Robert Treimer[3]; [1]Universidad de Chile; [2]RHI Chile; [3]RHI AG

Continuous converting of copper matte produces slag with high cuprous oxide content. The corrosion of chrome-magnesite refractories with slag of high cuprous oxide content is a major problem. Properties of olivine type of slag make the slag very attractive for matte smelting and converting. Analysis of phenomena determining the slag infiltration into the porosity of a refractory and dissolution of refractory components in the slag allowed for better understanding of the mechanisms of refractory destruction. The results of laboratory scale tests of the olivine slag infiltration into open porosity and corrosion showed dominating effects of slag temperature and cuprous oxide content. The optical and X-ray examination of slag samples permitted the definition of the mechanism of formation of new phases and refractory deterioration.

10:50 AM

Development of Refractory Practices for Peirce Smith Converters: *Jan Bäckström*[1]; *Martin Johann Hansel*[2]; [1]Boliden Mineral AB; [2]RHI AG

Starting from historic perspectives on Converting at the Rönnskär Smelter, the continuous developments of refractory practice is described. In the development the joint efforts of operators and suppliers of refractory have lead to significant achievements. This paper comprises the development of the converting process and the refractory lining as well as future prospects regarding operation and refractory lining design, especially in the face of equipment and raw material availability.

11:10 AM

Wear of Magnesia and Magnesia-Chromite Bricks in Vessels of the Non Ferrous Industry: *Robert Treimer*[1]; [1]RHI AG

For all high temperature processes refractory linings are used, which are influenced by a number of different factors and mechanisms causing wear of these refractory linings. In vessels of the Non-Ferrous-Industry mainly magnesia and magnesia-chromite bricks are used where the wear behaviour is influenced by a number of different chemical, thermal or mechanical factors. Chemical wear factors are e. g. corrosion through infiltrated slag, matte or metal, SO2-diffusion, redox-reactions and hydration. Thermal wear factors are the temperature level and thermal shocks and mechanical wear factors are primarily erosion due to the movement of the metallurgical bath, impacts by charging and tuyere-punching. Mostly however, not only single wear factors are dominating, but rather a combination of different factors like thermo-mechanical impacts or thermo-chemical stresses are interacting simultaneously. The detailed knowledge of the different wear factors and their impacts to the refractory material is essential for recommendations of the suitable refractory material for a great variety of applications in the Non Ferrous Industry and for a successful further development of refractory solutions. This paper presents in detail the process steps of a PS-converter and the wear and impact to the refractory lining. Based on the knowledge of these operational parameters and the single wear factors the most suitable lining concept of RHI is presented.

11:30 AM

Corrosion of Refractories in Peirce Smith Converters: *George Oprea*[1]; Waiman Lo[1]; Tom Troczynski[1]; Joe A. Rigby[2]; [1]University of British Columbia; [2]RHI Canada

The refractory lining of the tuyere line in Peirce Smith converters have usually much shorter life in service than the rest of the lining, above or below it. There are already recognized chemical, thermal and mechanical factors which contribute to the wear of the lining. Our study presents the experimental results on microstructural changes occurring on these tuyere line bricks in a nickel-copper converter. The experimental bricks, after use at the tuyere line, were investigated using SEM/EDS and XRD techniques, to identify the mineralogical changes due to interactions with the nickel-copper matte or fayalite type slag at the process temperatures. The microstructural changes were correlated with physical and mechanical properties of the brick before and after use and a wear mechanism was suggested. Although the matte does not theoretically react with the mineralogical components of the brick, a partial oxidation of the Ni-Cu sulphides was identified using a line analysis by SEM/EDS in the densified layer

at the hot face, which allows for mineralogical changes at the matte-refractory interfaces, with a high probability of micro-crack formation due to the thermal cycling during use. These microcracks were also identified to open through inter-granular fractures parallel to the hot face, which appeared to be the main cause of lost lining during use. The reactions between the fayalite slag and refractory brick could eventually play a role in chemical dissolution of the brick only in the superficial layer at the hot face, as no slag components were identified deep into the brick.

11:50 AM Discussion

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials VIII: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Chih-ming Chen, National Chung-Hsing University; Srinivas Chada, Medtronic; Sinn-wen Chen, National Tsing-Hua University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-ho Lee, Hongik University; Daniel J. Lewis, Rensselaer Polytechnic Institute; Kejun Zeng, Texas Instruments; Wojciech Gierlotka, AGH University of Science and Technology; Yee-wen Yen, National Taiwan University of Science and Technology

Monday AM Room: 2022
February 16, 2009 Location: Moscone West Convention Center

Session Chairs: Chih-ming Chen, National Chung-Hsing University; Sinn-wen Chen, National Tsing Hua University

8:30 AM Invited
Whisker Growth Behavior of Tin and Tin Alloy Lead-Free Finishes: *Katsuaki Suganuma*[1]; Keun-Soo Kim[1]; Sun-Sik Kim[1]; Alongheng Baated[1]; Kyoko Hamasaki[1]; [1]Osaka Univ

As a result of the global transition to lead-free electronics, the majorities of the electronic component manufacturers are now using pure tin or tin-rich alloys for terminal and lead finishes. Not only because of lead-free, tin whiskers have been one of the serious failure causes for electronics and aerospace equipments. Tin whiskers spontaneously grow from tin based lead-free finished surfaces even at room temperature. Recent researches have revealed the mechanisms of Sn whisker formation and growth from pure tin plating at room temperature. Comparing with pure tin plating, some tin alloy plating was found to be rather immune to whisker formation. However, the mechanism of alloying effects on whisker formation is still unclear. In this study, tin whisker growth process was examined on the various tin alloy platings at room temperature. Sn-Bi plating on Cu substrate significantly suppressed the tin whisker formation compared with pure tin plating.

8:50 AM
Whisker Growth on Sn Plating with or without Surface Treatment during Heat and Humid Environments: *Keun-Soo Kim*[1]; Sun-Sik Kim[1]; Alongheng Baated[1]; Kyoko Hamasaki[1]; Katsuaki Suganuma[1]; Masanobu Tsujimoto[2]; Isamu Yanada[2]; [1]ISIR, Osaka University; [2]C. Uyemura & Co., Ltd.

Establishment of lead-free plating technology and whisker countermeasures is one of the critical problems remaining to be solved for lead-free electronics packaging. In our previous study, we reported on mitigation method of the Sn whisker by thin metal layer formation, such as Ni, Au, Pd, on pure Sn plating. Comparing with pure Sn plating, metal layer/Sn plating was much stable against Sn whisker formation in room ambient environment. In the current work, Sn whisker growth behavior of pure Sn plating and metal layer/Sn plating samples during 55°C/85% and 85°C/85% relative humidity (RH) exposure and thermal fatigue tests were investigated. Ni, Au and Pd layers with the thickness from 50nm to 200nm were deposited on matte Sn plating by flash-coating process. Ni, Au and Pd metal layer on Sn plating significantly suppressed the Sn whisker formation under the severe thermal and humid conditions.

9:05 AM
Study of Surface Oxidation of Sn(Ni, Ag, Si, In, Cu) Alloys: *Yan You Li*[1]; Cheng-Yi Liu[1]; [1]National Central University

Surface oxidation of molten solder plays a very important role for the soldering wettability and wave soldering. Oxidation of molten Sn-base alloys is investigated. Five different metallic elements (Ni, Ag, Si, In, Cu) were alloyed with pure Sn and form Sn(M) alloys. The M doping ranges from 0.5wt.% to 10 wt.%. Sn(M) alloys were annealed at 600° for certain times. Then, the weight gain of Sn(M) were measured, which indicate the degree of oxidation of molten Sn(M) solder. Our preliminary results show that the Ag and In additive can retard the oxidation rate of Sn (M) solder. And, Cu additive can enhance the oxidation rate of Sn(Cu) molten solders, because the surface oxide scale has larger number of voids, which encourage oxygen diffuse inward easily. In this talk, the detail oxidation mechanism of kinetics on Sn(M) molten solders will be present and discussed.

9:20 AM
Fundamental Study on the Inter-Mixing between 95/5 High Lead Solder Bump and the 37/63 Pre-Solder on Chip-Carrier Substrates: *Chih-Chiang Chang*[1]; [1]National Taiwan University

The microstructure of solder, as well as the formation and growth of intermetallic compounds, plays a critical role in the reliability of electronic packaging. In this study, we study the inter-mixing between the eutectic solder deposited over chip-carrier substrates and 95Pb5Sn solder bump. The interfacial reactions on the bump interface and the substrate interface are also studied. The reaction conditions include aging at 100, 130, 150, 175°C for 100, 500, 1000, 1500, or 2000hr. It is found that de-wetting occurred between UBM and high lead solder interface after five reflow. After 100 hr of aging, $(Cu,Ni)_6Sn_5$, Cu_3Sn, and $(Ni,Cu)_3Sn_4$ IMCs formed. Micro-voids formed between Cu_3Sn and Cu interface were also identified at 150 and 175°C. At 100°C, however, micro-void formed only after aging for more than 1500 hr. The activation energy values for the growth of Cu_6Sn_5, Cu_3Sn and Ni_3Sn_4 were found to be 116, 67, and 88 kJ/mol, respectively.

9:35 AM
Sn Concentration Effect on the Massive Spalling in High-Pb/Cu Reaction: *M.H. Tsai*[1]; C.R. Kao[1]; [1]National Taiwan University

Massive spalling of Cu_3Sn intermetallic compound in high-Pb solders on bare Cu substrate during soldering reaction was investigated to understand the spalling phenomenon of intermetallics in various Sn-containing solders. High-Pb solder alloys of four compositions (0.5Sn-99.5Pb, 1Sn-99Pb, 3Sn-97Pb and 5Sn-95Pb) were soldered at 350°C for 1 to 20 minutes. For all solder compositions only Cu_3Sn was observed at the interface between the solder and the substrate during soldering. When the Sn concentrations (0.5Sn and 1Sn) were low, the massive spalling occurred. However, spalling was not observed when the Sn concentrations (3Sn and 5Sn) were high until the soldering time was more than 10 minutes. The Cu-Sn-Pb phase diagram is used to rationalize this Sn concentration effect.

9:50 AM Break

10:10 AM Invited
Assessment of Electromigration Effects at Copper Wire-Bonds: C. Wang[1]; H. Goddin[2]; *A. Greer*[1]; [1]University of Cambridge; [2]TWI Ltd

The interfacial reactions of Cu wire-bonds on Al-based metallization have been studied. A sequence of intermetallic phases forms, the phase selection being influenced by the limited supply of Al. Ultimately, the bond is dominated by Cu with a graded concentration of Al in solution. There is no evidence for any formation of Kirkendall voids. To investigate electromigration effects, electrical currents of 500 mA were imposed on the Cu ball-bonds. Compounds formed at the Cu/Al interface in a sequence similar to that without currents. Electromigration appears to have a negligible effect on intermetallic growth at Cu/Al interfaces, in contrast to the strong electromigration effects found for reactions at Au/Al interfaces. Computational simulations of resistance changes of the Cu ball-bonds are consistent with the experimental results.

10:30 AM
Current-Induced Growth of Reaction Phases at Electroless Nickel/Tin Interfaces: Q. Yang[1]; P. Shang[1]; J. Guo[1]; Z. Liu[1]; *J. Shang*[2]; [1]Institute of Metal Research; [2]University of Illinois at Urbana-Champaign

The microstructural transformations at the electroless nickel/tin interfaces following current stress were observed by scanning and transmission electron

microscopy. A clear polarity effect was found in the growth of the interfacial reaction phases. At the cathode side, intermetallic compounds (IMC) delaminated from the electroless Ni (EN) layer and drifted into the Sn phase with the electron wind force. While the total thickness of Ni-P and EN decreased at the cathode side, the Ni-P layer grew in thickness between the EN layer and Sn. At the anode, only slight increases in the IMC and Ni-P thickness were observed. Such polarity effect is shown to result from current-driven migration of reactive species.

10:45 AM

Electromigration of Sn-Zn-Based Lead-Free Solders: *Chih-ming Chen*[1]; Yu-min Hung[1]; Chi-pu Lin[1]; [1]National Chung-Hsing Univ

Sn-Zn-based alloys are promising lead-free solders. Electromigration is an important reliability issue of solder systems. Electromigration of Sn-Zn-based solders were investigated under current stressing with a density of about 105 A/cm2 at 80 to 140°. Two different cooling conditions, furnace and fan cooling, were used in the cooling process of reflow, and by which different microstructures developed in the solders. The furnace and fan-cooled solders displayed distinct electromigration behaviors, where lots of Sn extrusion sites were found in the furnace-cooled solder but the fan-cooled solder displayed a nearly unchanged microstructure. The effects of Bi and Cu addition into the Sn-Zn solder on the electromigration behavior were also investigated.

11:00 AM

Effect of Current Density Distribution on the Formation of Intermetallic Compounds in Pb-free Solder Joints: *Jung-Kyu Han*[1]; Luhua Xu[1]; Shih-Wei Liang[2]; King-Ning Tu[1]; Yi-Shao Lai[3]; [1]UCLA; [2]National Chiao Tung University; [3]Advanced Semiconductor Engineering

An investigation was carried out of the role of current density distribution in determining the formation of intermetallic compounds, using Pb-free flip-chip SnAgCu solder joints. In general, Cu atoms diffuse to the anode side in solder joints due to electromigration and evenly form intermetallic compounds along the contact area. When Cu trace on the substrate side was irregularly consumed, however, the shape of intermetallic compounds was changed to arc-shape at anode side. In this case, abnormal Cu trace consumption causes current density re-distribution in solder joints. Since Cu atoms migrate faster along high current density regime than low current density regime, the change of current density distribution affects the shape of intermetallic compounds. The simulation data of current density distribution is also in good agreement with the experimental results.

11:15 AM

Critical Product of Electromigration in Cu-Sn Intermetallic Compounds: *Luhua Xu*[1]; Jung-Kyu Han[1]; Shih-Wei Liang[1]; Di Xu[1]; Masaru Fujiyoshi[2]; K.N. Tu[1]; [1]University of California, Los Angeles; [2]Hitachi Metal Ltd, Japan

The properties of intermetallic compound become more important with the trend in reducing solder joint size. This is because the thickness of UBM as well as the reflow temperature will remain the same while the bump size decreases. Thus the volume fraction of IMC formation in the solder joint will increase greatly. For example, when the solder joint size decreases from 100 to 50 and to 25 micron, the volume will decrease 8 to 64 times, respectively. As a result, intermetallic compound (IMC) could occupy the entire solder joint which can become a pure intermetallic joint. The properties of intermetallic(i.e., Cu6Sn5 + Cu3Sn) were characterized in this study. Electromigration behavior and critical product of the intermetallic compound is investigated by employing the V-groove solder joint samples. It was found that the critical product of Cu6Sn5 IMC is at least one order of magnitude higher than that of SnAgCu solder.

11:30 AM

Polarity Effect of the Growth of Intermetallic Compound in SnAgBiIn Pb-Free Solder under Electromigration: Albert Wu[1]; *Kuo-Hao Sun*[1]; [1]National Central University

The Pb-free SnAgBiIn solder strips were prepared in the Si(001) U-grooves to investigate the behaviors under electromigration. The Cu electrodes were electroplated in the grooves and the solders were consequently reflowed between the electrodes. The samples were tested under various temperatures and current densities. The thickness of the compounds at both interfaces between the solders and the Cu electrodes were measured. The changes of the thickness with times were recorded. The compositions of the intermetallic compounds were analyzed by EPMA. The kinetics of the growth of the compounds is discussed in this paper.

Recent Advances in Thin Films: Process-Property Correlations

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Thin Films and Interfaces Committee
Program Organizers: Nuggehalli Ravindra, New Jersey Institute of Technology; Gregory Krumdick, Argonne National Laboratory; Choong-un Kim, University of Texas; Narsingh Singh, Northrop Grumman, ES

Monday AM — Room: 3011
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Nuggehalli Ravindra, New Jersey Institute of Technology; Choong-un Kim, University of Texas

8:30 AM Introductory Comments

8:40 AM Invited

A Microstructural Characterization of Ta-Based Thin Films for Cu Metallization: Julien Nazon[1]; Marie-Hélène Berger[2]; Thierry Sauvage[3]; Jean-Claude Tedenac[1]; *Nicole Fréty*[4]; [1]Institut Charles Gerhardt - Université Montpellier II; [2]Centre des Matériaux P.M. Fourt - Ecole des Mines de Paris; [3]CEMHTI - UPR3079 CNRS; [4]Institut Charles Gerhardt - Université Montpellier II

Advances in microelectronic and thermoelectric device technologies are dependent on the development of new barriers against the diffusion of electrical wiring copper. In the present work, the efficiency of tantalum-based thin films was investigated through a microstructural approach associated to the measurement of the electrical resistivity. The properties of TaN(75nm)/Ta(75 nm) and TaN(50 nm)/Ta(50 nm)/TaN(50 nm) multilayer thin films have been studied and compared to that of TaN(150 nm) single layers. These thin films were deposited onto silicon substrates by radio-frequency reactive sputtering. The microstructure was characterized using Glancing Angle X-Ray Diffraction, Scanning and Transmission Electron Microscopies and Rutherford Backscattered Spectrometry. The diffusion mechanisms were studied after vacuum annealing in the 773-973K temperature range using these characterization techniques. The results pointed out the interest of a multilayered thin films structure in the improvement of the diffusion barrier properties.

9:10 AM

A Statistical-Thermodynamic Modeling of Behavior and Properties in Thin-Film Intermetallic L12- and D019-Structures: *Olga Semenova*[1]; Regina Krachler[1]; Sabine Knott[1]; [1]University of Vienna

Modeling of behavior of thin films under various technological states is of paramount importance, since these compounds are fascinating group of materials, from point of view of fundamental properties and practical applications. Statistical-thermodynamic modeling based on Ising approach and Bethe-Bragg-Williams random-mixing approximations is proposed for description of thermodynamic behavior and ordering phenomena in many layers nano-crystalline materials with L12- and D019-structure. It includes description of Long-Range and Short-Range Ordering in crystal lattice. Obtained theoretical results are tested using experimental data on thermodynamic and structural properties of bulk intermetallics Ni3Ga, Ni3Al, Ti3Al. Degree of long-range order and critical transition temperatures were predicted and compared to experimental data. Proposed approach and obtained results can be applied to process of new advanced materials discovery and development, these may help address a challenge of rapid and accurate optimization in solution of many fundamental and technological problems, which are not able to be solved experimentally.

9:30 AM Invited

Corrosion Properties of Chromized Tungsten Carbide Materials: *Jyh-Wei Lee*[1]; Jai-Lin Li[1]; Yu-Ting Lin[1]; [1]Tungnan University

Tungsten carbide (WC) materials have been widely used in industries due to their high hardness and excellent wear resistance. However, poor corrosion resistance of WC material in the acid solution is found due to the selective corrosion of cobalt binder. A novel chromized thin film has been produced on the surface of tungsten carbide by pack cementation process at 900°C. A thin film mixture containing chromium nitride and chromium carbides was revealed. The corrosion resistances of untreated substrate and chromized WC materials

under various chromizing conditions in sulfuric acid solution were investigated. It is observed that the novel chromizing thin film provides excellent corrosion resistance to the cobalt contained WC substrate. Nevertheless, selective corrosion attack on the surface defects of chromizing thin film was found.

10:00 AM

Crystallization Structure and Annealed Effect of Ni-Doped Sn-Al Thin Films on Electromagnetic Interference Shielding Characteristics: *Hung Fei-Shuo*[1]; Fei-Yi Hung[2]; Chiang Che-Ming[1]; Lui Truan-Sheng[3]; [1]Department of Architecture, National Cheng Kung University, Tainan; [2]Institute of Nanotechnology and Microsystems Engineering, Center for Micro/Nano Science and Technology, National Cheng Kung University, Tainan; [3]Department of Materials Science and Engineering, National Cheng Kung University, Tainan

Electromagnetic interference (EMI) is a new form of pollution discovered in recent years. The elements Sn and Al not only possess EMI shield efficiency, but also have acceptable costs. In this study, sputtered Sn-Al thin films with Ni doped (0~9 at.%) were used to investigate the effect of the crystallization mechanism and film thickness on the electromagnetic interference (EMI) characteristics. In addition, the annealed microstructure, electrical conductivity and EMI of the Sn-Al films and the Ni-doped Sn-Al films were compared. The results show that Sn-Al film increased the electromagnetic interference (EMI) shielding after annealed. For the Ni-doped Sn-Al films with higher Ni atomic concentration, the low frequency EMI shielding could be improved. After annealing, the Sn-Ni and Al-Ni intermetallic compound (IMC) of thin film distributed in the matrix. This metallurgical effect not only enhanced the diffusion of atoms to the grain boundaries, but also promoted the high frequency EMI shielding.

10:20 AM Break

10:40 AM

Effect of Sputtering Conditions and Post-Annealing on Internal Structure and Electrical Properties of Titanium-Oxide Thin Films: *Masanari Tomozawa*[1]; Masashi Mikami[1]; Kimihiro Ozaki[1]; Keizo Kobayashi[1]; Toshimasa Miyazaki[2]; [1]National Institute of Advanced Industrial Science and Technology; [2]Tayca Corporation

Titanium-oxide thin films were deposited onto glass substrates by magnetron-sputtering. A target material consisted of TiO and Ti6O11. Deposition was carried out under various applied voltage, Ar gas pressure and sputtering time. Thickness, internal structure and electrical resistivity of the thin films were investigated. Thickness of the thin films increased with increasing applied voltage, Ar gas pressure or sputtering time under our experimental conditions. The thin films deposited under lower Ar gas pressure were mainly composed of Ti6O11. On the other hand, those deposited under higher Ar gas pressure were composed of Ti6O11 and TiO. Vacuum annealing after deposition also produced TiO phase in the thin films. Formation of TiO phase lowered electrical resistivity of the thin films.

11:00 AM

Evolution of Annealing Twins in Thin Film Microstructures: *ChangKyu Yoon*[1]; David Field[1]; [1]Washington State Univ

The characteristic microstructure of a thin film affects its function in electronic applications. Modern Cu films and line structures often contain a large fraction of annealing twins that change the crystallographic texture and grain boundary character distribution in these structures. Annealing twin evolution is often overlooked or ignored in models of grain growth such as the conventional Monte Carlo simulation (Potts model). In this work, texture and grain structure evolution is modeled using Potts model according to the overall energy minimization model. Specific criteria are introduced to nucleate and grow twin boundaries. Simulation results are compared with observation of structure evolution in Cu and Ni films as a function of film thickness and annealing temperature.

11:20 AM

Phase-Field Modeling of Thin Film Growth: Applications to Step and Island Dynamics: *Zhengzheng Hu*[1]; Shuwang Li[1]; Steven Wise[2]; John Lowengrub[1]; Axel Voigt[3]; [1]University of California, Irvine; [2]University of Tennessee; [3]TU Dresden

A phase-field model is presented to simulate the dynamics of step flow and small islands during epitaxial growth. Asymmetric kinetics rates and edge diffusion are incorporated. Moreover, a modified free energy function and a corrected initial phase variable are given to efficiently capture the morphological evolution. Our recent study on step flow matches with results presented by Frank Haußer et al., based on a front tracking method. In the long wavelength regime, we observe meandering with a wavelength being determined by the linear instability and endless growth of the amplitude. Whereas in the shorter wavelengths, we observe coarsening due to the competition between different wavelengths. We also observe mushroom formation, subsequent pinch-off leading to the formation of a vacancy island. When apply our model to the island dynamics, our simulations confirm the linear stability results of Hu et al., and reveal the possibility of shape control in nanoscale.

11:40 AM

Stress-Driven Surface Instabilities in Solids with Diffusing Charged Defects: *Steven Henke*[1]; P. Chung[2]; A. El-Azab[1]; M. Grinfeld[2]; [1]Florida State University; [2]US Army Research Laboratory

Stress-driven rearrangement instability (SDRI) theory postulates that diffusion in stressed solids can lead to surface morphological instability, an effect that is currently believed by many physicists to be real and important for elevated-temperature deposition or annealing of thin films. Both atomic surface diffusion and bulk diffusion of point defects contribute to the instabilities. The stress-driven diffusion of mobile oxygen vacancies in the bulk is especially important in ferroic perovskite films (e.g., Barium Strontium Titanate), which have desirable optical and electric properties for device and sensor applications, and often require well-controlled surfaces and interfaces. We present a continuum reformulation of the SDRI theory that includes the coupled electro-elastic diffusion of oxygen vacancies and introduce a 3D finite-element scheme to solve the equations for film surface evolution. We also explore the stability of the film boundary due to perturbations and attempt to characterize the incipient instabilities in terms of the model parameters.

Recycling of Electronic Wastes: Life Circle Analysis and Environmental Issues

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS Materials Processing and Manufacturing Division, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Lifeng Zhang, Missouri University; Fay Hua, Intel Corp; Oladele Ogunseitan, University of California, Irvine; Gregory Krumdick, Argonne National Laboratory

Monday AM — Room: 2024
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Gregory Krumdick, Argonne National Laboratory

8:30 AM Introductory Comments

8:35 AM

Closed Loop WEEE Recycling? Challenges and Opportunities for a Global Recycling Society: *Christina Meskers*[1]; Christian Hagelueken[1]; [1]Umicore

End-of-life Electronic and Electrical Equipment (WEEE) is unjustly regarded as mainly a waste management problem. What is structurally overlooked is the enormous resource impact of these devices. EEE represents a tremendous metal resource, which should be utilized through effective recycling, which has a much lower environmental footprint than primary production. The actual amount of end-of-life EEE recycled today is embarrassingly low since insufficient EEE is collected and part of the collected EEE is exported to developing countries, where it is largely not entering official recycling systems. To achieve a global recycling society issues like technology, economics, life cycle structure, stakeholder awareness and legislation have to be addressed in a global, co-operative manner so that sustainable closed product cycles can be obtained. Requirements for a "global recycling society" are defined to address today's reality of global flows of used consumer products, taking into account the likely needs of the future.

8:55 AM Question and Answer Period

9:05 AM

Life Cycle Analysis for Recovered E-Wastes: *Ocileide Custódio da Silva*[1]; Diego Blanco[2]; [1]INdT; [2]UEA

The use of recovered e-waste has been evaluated in order to reach environmentally friendly solutions for the end of life of electronic products. However, to assure the performance of these recovered products is necessary to understand the critical points affecting the reliability and final characteristics

of them. This work uses life cycle assessment as methodology to evaluate the performance of e-waste from electronic devices. The following materials were evaluated: polymers and metals. The life cycle of these materials were assessed from cradle to grave. The degradability of the materials was analyzed by environmental tests to complement the evaluation of the end of life of the materials studied. According to the results, the materials analyzed may be considered to be a good option for manufacturing different products if obstacles, such as processing parameters, can be overcome.

9:25 AM Question and Answer Period

9:35 AM

Toxicity Screening for Materials Selection in the Printed Wiring Board Industry: *Carl Lam*[1]; Julie Schoenung[1]; [1]University of California, Davis

In order to support the decision process of toxics use reduction in manufacturing and design of product systems, it is pertinent to provide a rigorous and up-to-date method in efficiently screening and scoring a material's hazard based upon its potential human health toxicity and exposure. Similar to that of EPA's Use Cluster Scoring System (UGSS) and Risk Screening Environmental Indicators (RSEI) models, publicly available toxicity data sources are utilized in this screening model. Example analyses of metals commonly used in the printed wiring board manufacturing industry (such as lead, copper, tin, zinc and others) are presented to illustrate the evaluation methodology. Statistical analysis on the reliability of the various toxicity data will provide better insight and quantification of uncertainties in the scoring methodology. The end result has potential to expand traditional life cycle analysis in the human health effect categories to provide error reporting at its foundation.

9:55 AM Question and Answer Period

10:05 AM

Engineering Environmentally-Benign Electronics: Convergent Optimization of Materials Use, Consumer Participation, and Government Regulation: *Oladele Ogunseitan*[1]; Jean-Daniel Saphores; Julie Schoenung[2]; Andrew Shapiro[1]; [1]University of California, Irvine; [2]University of California, Davis

Sustainable strategies to reduce the public and environmental burden of hazardous materials associated with discarded electronic products will require coordination of efforts ranging from selective use of materials in product design, consumer behavior toward recycling, to trans-boundary regulatory incentives. Our project adopted the cellular phone for a model for addressing the knowledge gaps and policy discrepancies that contribute to continuing risks associated with electronic waste. This presentation focuses on progress and emerging solutions according to five specific objectives: (a) Development of an integrative environmental burden model (EBD) for chemicals associated with the designation of these products as post-consumer hazardous waste. (b) Identification of alternative materials to replace hazardous constituents according to the EBD and current policies. (c) Estimation of the cost and performance differentials between the alternative materials and current CE constituents. (d) Survey of consumer willingness to participate in e-waste management practices (e) Comparative assessment of regulatory policy designs.

10:25 AM Break

10:45 AM

Modeling the Impact of Physical System Architecture on Recycling System Performance: *Jeffrey Dahmus*[1]; Elsa Olivetti[1]; Susan Fredholm[1]; Jeremy Gregory[1]; Randolph Kirchain[1]; [1]Massachusetts Institute of Technology

As recycling systems for waste electronics become more widespread, understanding and characterizing the key determinants of economic and environmental performance becomes critical. One such determinant is physical system architecture, which in turn has a profound impact on material recovery rates. The work presented here examines the effect of system architecture on the economic and environmental performance of electronics recycling systems. Such architectural decisions greatly impact the economic performance, affecting costs, such as those associated with collecting and transporting end-of-life electronics, as well as revenues, such as those associated with the amount of saleable material recovered. Environmental performance, including the trade-off between the burdens of collecting and transporting waste electronics and the benefits of recovering and recycling materials, is also greatly affected by system architecture. The work presented here uses network models, process-based cost models, and lifecycle analysis tools to evaluate the impact of system architecture on recycling system performance.

11:05 AM Question and Answer Period

11:15 AM

Human Health and Ecosystem Toxicity Potentials of Waste Electronic Devices: *Seong-Rin Lim*[1]; Julie Schoenung[1]; [1]University of California, Davis

The objective of this study is to evaluate human health and ecosystem toxicity potentials from heavy metals in e-waste, i.e., laptop computers, LCD monitors, LCD TVs, plasma TVs, and CRT TVs. These toxicity potentials are evaluated by using heavy metal contents from the literature (California Department of Toxic Substances Control, 2004; Matsuto et al., 2004) and their toxicity characterization factors from the US EPA Tool for the Reduction and Assessment of Chemical and other environmental Impacts (TRACI). The toxicity potential from the plasma TVs are more significant than those from the LCD-related devices but less than those from the CRT TVs with the exception of ecotoxicity through water. The cancer potential is primarily from lead; the noncancer potential is primarily from lead and copper; and the ecotoxicity is primarily from copper. Therefore, e-waste management policy should focus on recycling and elimination of lead and copper.

11:35 AM Question and Answer Period

11:45 AM

A Review of Electronic Waste (e-Waste) Recycling Technologies "Is e-Waste an Opportunity or Treat?"": *Muammer Kaya*[1]; [1]Osmangazi University

The electronic industry is the world's largest and fastest growing manufacturing industry. As a result of this growth, combined with rapid production obsolescence, discarded electronics or electronic waste begin a serious solid waste problem in the world. Electronic waste is the most rapidly growing waste and contains over 1000 different substances. It is a crisis not only of quantity but also a crisis born from toxic ingredients- such as Pb, Be, Hg, Cd, Cr+6 and BFRs that pose both occupational and environmental health treat upon disposal. E-waste market will exceed 11 billion $ in 2009 in the world. This paper reviews the e-waste problem in the world, describes existing collection, dispose and recycle techniques for glass, plastics and metals and covers the legislations and finance for environmental friendly solutions and for sustainable development. Current situations of scrap automobile accumulators/household batteries recycle in Turkey are presented as a case study.

12:05 PM Question and Answer Period

Shape Casting: Third International Symposium: Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Solidification Committee, TMS: Aluminum Processing Committee
Program Organizers: John Campbell, University of Birmingham; Paul Crepeau, General Motors Corp; Murat Tiryakioglu, Robert Morris University

Monday AM — Room: 2011
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Glenn Byczynski, Nemak Europe GmbH

8:30 AM Introductory Comments

8:35 AM

Intrinsic and Extrinsic Metallurgy: *John Campbell*[1]; [1]University of Birmingham

Physical metallurgy has been highly successful in describing the nucleation and growth of dense phases occurring during solidification of metals. The limitations of intrinsic metallurgy are seen mainly in the presence of pores and cracks that lead to various kinds of failure in materials, but which cannot be explained by intrinsic mechanisms. For instance solidification as a simple phase change is unable to nucleate a pore or a Griffiths crack (by either homogeneous or even by heterogeneous nucleation) because of the extremely high interatomic forces, as supported by much excellent theoretical and experimental evidence. Only defects entrained from the outside can explain the occurrence of volume defects such as pores and cracks, and therefore provide understanding of the fundamental causes of failures in tensile, creep, fatigue modes, and probably some types of corrosion pitting failures. An accurate metallurgical understanding of cast and wrought alloys requires both intrinsic and extrinsic contributions.

9:00 AM
Quality Indices for Cast Aluminum Alloys: *Murat Tiryakioglu*[1]; John Campbell[2]; [1]Robert Morris University; [2]University of Birmingham

Several indices are available in the literature to assess the structural quality of cast Al alloys, especially Al-7%Si-Mg alloys, based on tensile test results. Some of these indices, most notably the one developed by Drouzy et al. provide a number that do not necessarily have a physical meaning, while the others are a measure of what fraction of the expected tensile property is achieved. These indices are discussed and the concept of maximum potential ductility is introduced. A new quality index that uses this maximum ductility potential concept is introduced for Al-7%Si-Mg as well as Al-Cu alloys.

9:25 AM
Use of 'Standard' Molds to Evaluate Metal Quality and Alloy Properties: *Geoffrey Sigworth*[1]; Tim A. Kuhn[1]; [1]Alcoa Primary Metals

Several mold designs have been proposed as standards for aluminum castings. The two most commonly used in North America are the ASTM B108 test bar, and a 'step' casting proposed by the Aluminum Association (AA). The history of these molds is reviewed briefly and mechanical properties are presented for A356-T6 alloy castings. The B108 test bar is prone to shrinkage. Measures that help to minimize this shrinkage are discussed. The AA mold is also prone to shrinkage, but a judicious selection of sample locations avoids much of the problem. In spite of their limitations, the two molds can be used to evaluate melt treatment procedures and metal quality in the foundry. Data from casting trials are presented for both molds.

9:50 AM
Properties of B356-T6 Aluminum Cast via Permanent Mold and Advanced Squeeze Cast (ASC) Processes: *Gerald Gegel*[1]; David Weiss[2]; William Edney[3]; [1]Materials & Process Consultancy; [2]Eck Industries; [3]Prototype Cast Manufacturing Inc.

The mechanical properties of cast products are a function of alloy composition, solidification rate and porosity content. The tensile and fatigue properties of AA B356 cast using low pressure permanent mold and advanced squeeze casting processes are compared to illustrate the mechanical property advantages accrued as a result of solidification under pressure. The ASC method uses low pressure to fill the die and then applies squeeze pressure directly to the entire volume of the component. As this technology is new, we will describe the design and operation of this production-viable 600-ton machine. The design of the machine permits the use of the same tooling to produce both LPPM and ASC castings. This DOE sponsored research and development project has provided a production-viable machine and process technology that will improve the strength and reliability of cast components.

10:15 AM Break

10:25 AM
The Relationship between Defect Size and Fatigue Life Distributions in Al-7% Si-Mg Alloy Castings: *Murat Tiryakioglu*[1]; [1]Robert Morris Univ

Fatigue life of cast Al alloys is dictated by the largest defect in the casting. The size distribution of largest defects can be modeled by extreme value distributions. When the defect size statistics are combined with equations that link the failure-initiating defects with fatigue life, the statistical distribution can be estimated. This technique is demonstrated on several datasets from the literature.

10:50 AM
Improvement of an Existing Model to Estimate the Pore Distribution for a Fatigue Proof Design of Aluminium High-Pressure Die Casting Components: *Christian Oberwinkler*[1]; Heinz Leitner[1]; Wilfried Eichlseder[1]; [1]University of Leoben, Institute of Mechanical Engineering

The estimation of the fatigue life time of aluminium high-pressure die casting components requires the knowledge of the pore distribution. A basic model was derived from a hpdc plate using Self-Organizing Maps and statistical tools to compute a statistical distribution of the porosity within a well defined area. A new component (especially designed for this project) has been used to extend the applicability of the existing model, including influences like the hydrogen content, the wall thickness, different feeding times, and the mold temperature. An example will be presented to visualize how the estimated porosity distribution can be included into the computation of the safety against dynamic loading using a simplified Monte-Carlo simulation together with the Kitagawa-Haigh diagram as a material model.

11:15 AM
Advanced Cast Aluminum Alloys: *Alan Druschitz*[1]; John Griffin[1]; [1]University of Alabama at Birmingham

A recent advancement in aluminum casting has demonstrated that complex shapes can be cast from microalloyed Al-Cu alloy in dry sand molds with chills and that these castings can be heat treated to produce mechanical and physical properties nearly comparable to wrought 2519 aluminum alloy. Given this initial level of success, further research has been focused on improving this microalloyed Al-Cu alloy so that the mechanical properties consistently meet or exceed those of wrought 2519 alloy. Further, new research has been initiated on ultra-high strength, microalloyed Al-Zn-Mg-Cu alloys with the goal of producing complex castings with properties significantly better than wrought 2519 aluminum alloy and equivalent to or better than the best 7000 series wrought alloys. The development of the appropriate chemistries, casting practices and heat treatments are described in this paper.

Structural Materials Division Symposium: Advanced Characterization and Modeling of Phase Transformations in Metals in Honor of David N. Seidman on his 70th Birthday: Driven Alloy Systems

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Robert Averback, University of Illinois, Urbana-Champaign; Mark Asta, University of California, Davis; David Dunand, Northwestern University; Ian Robertson, University of Illinois at Urbana-Champaign; Stephen Foiles, Sandia National Laboratories

Monday AM — Room: 3000
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Ian Robertson, university of illinois

8:30 AM Introductory Comments

8:40 AM Keynote
Energy Security, Climate Change and Materials Science: Requirements and Strategies for Sustainability in the 21st Century: *Tomas Diaz de la Rubia*[1]; [1]Lawrence Livermore National Laboratory

Increasing energy demand and levels of CO2 in the atmosphere are placing enormous pressure on natural resources, the global ecosystem, and international political stability. Alternative sources of energy are required in order to meet increased energy demand, stabilize the increase of atmospheric carbon dioxide, and mitigate the concomitant climate change. In response, governments are urgently trying to develop new economical, sustainable, and environmentally friendly energy technologies. In this talk, I will present an overview of a new approach that combines inertial confinement fusion and fission into a simple, safe, cost-effective technology that promises to provide sustainable energy while minimizing proliferation concerns and nuclear waste disposition issues and cost. I will survey some of the key research challenges associated with the accelerated development of new materials with properties tailored to meeting this energy technology.

9:20 AM Invited
The Dislocation Network under Irradiation: *Georges Martin*[1]; Dan Mordehai[1]; [1]CEA

In crystalline metals, the dislocation network is the main source of internal strain. Irradiation, steadily injects new sources of internal strain in the metal (point defects, defect clusters): as a consequence, the evolution of the dislocation network is driven by irradiation. Examples are irradiation enhanced dislocation annealing, irradiation driven re-crystallization and irradiation induced plasticity at temperatures and stress levels where plastic strain does not show up in the absence of irradiation. The atomistic mechanisms by which the forcing proceeds have long been recognized: the partitioning of defect elimination between dislocations and other defect sinks, both in stationary or transient regimes, cascade effects… As a result, under irradiation, dislocations climb "for free". However dislocation annealing requires the coordinated climb of dislocation pairs. We show that polarisability effects, at the root of SIPA creep (Stress

Induced Preferential Absorption), provide the mechanism for coordinated climb, which eases dislocation annealing.

9:50 AM Break

10:15 AM Invited
Atom Probe Tomography Characterization of Multiple Phase Separations in PM 2000 Ferritic ODS Steels: *Michael Miller*[1]; Carlos Capdevila[2]; Kaye Russell[1]; [1]Oak Ridge National Laboratory; [2]Centro Nacional de Investigaciones Metalúrgicas

Atom-probe tomography has been used to quantify the scale and composition parameters of the α-α' and Fe(Ti,Al) phases that are produced in a PM 2000 oxide dispersion strengthened ferritic alloy during low temperature isothermal annealing between 400 and 475ºC. Atom probe tomography has revealed that both the scale and concentration amplitude of the chromium-enriched α' regions increase with ageing time. The morphology of the α' regions also changes from an interconnected network structure to isolated particles as ageing proceeds. These fine scale phases are responsible for a significant increase in the hardness of the alloy with ageing time. The influence of aluminum on the position of the miscibility gap has also been determined. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

10:45 AM
Austenite Precipitate Kinetics and Ballistic Property of Low-Carbon Ni Steels: *Xian Zhang*[1]; [1]Naval Surface Warfare Center

A comprehensive study on the phase transformation kinetics of austenite reversion in a series of VIM (vacuum induction melt) Low-Carbon Ni steels, containing five different Ni contents ranging from 2.5 % to 10 %, was performed to investigate key microstructural contributors to the ballistic resistance of steel. A wide range of heat treatments, static and dynamic mechanical tests, and various analytical techniques including SEM, TEM, EELS, EBSD, and X-ray diffraction, were employed to characterize microstructure and processing-structure-property correlations. This paper focuses on the control of austenite precipitate kinetics and morphology during the QLT (quenching-lamellarizing-tempering) process and its effect on FSP ballistic resistance V50. We conclude that highly dispersed fine austenite particles (on the nanometer scale) embedded in a ductile ferrite matrix appear to be the optimum microstructure for obtaining the best combination of strength, toughness, and ballistic property of Low-Carbon Ni steels.

11:00 AM Invited
Synthesis of New Materials via Self-Organization Driven by External Forcing: *Pascal Bellon*[1]; Robert Averback[1]; Pavel Krasnochtchekov[1]; See Wee Chee[1]; Brad Stumphy[1]; [1]University of Illinois

Materials, either during their processing or in service, are often subjected to sustained dynamical forcing, for instance plastic deformation during extrusion, and irradiation by energetic particles in nuclear reactors. These non-equilibrium dissipative material systems display a tendency to self-organize. Using atomistic simulations and continuum modeling, we will show that, for alloys irradiated with energetic particles, this self-organization results from the competition of dynamical processes acting at different length scales. Furthermore, the characteristic length scale of these self-organized structures, which is typically in the range of 1 to 100 nm, varies continuously as the irradiation-induced displacement rate and the temperature are varied. These predictions are tested using experiments on Cu-base alloys using transmission electron microscopy and atom probe tomography. This approach opens a new route for the synthesis of nanostructured materials with tunable scale, a property that can then be used to design radiation-resistant materials.

11:30 AM Invited
Atom Probe Tomography of Materials for Energy Applications: *Thomas Kelly*[1]; [1]Imago Scientific Instruments

Atom probe tomography (APT) produces three-dimensional structural and compositional images of materials at the atomic scale. These data have proven invaluable for a wide range of materials used in energy applications. Specimen preparation advances have made it routine now to extract and analyze specimens from bulk materials including advanced alloys, device wafers and even finished components. Major developments in atom probe technology have led to greater facility for running specimens and greater detail in quantitative analysis. In this talk, examples will be given of how this capability is having impact on metals, semiconductors, ceramics, and even synthetic organics and polymers.

12:00 PM
Radiation Resistant Alloys for Use at High Temperatures: See Wee Chee[1]; Brad Stumphy[1]; *Robert Averback*[1]; Pascal Bellon[1]; [1]University of Illinois

It has been known for several decades that materials that include high concentrations of nanoscale features within their microstructures provide excellent resistance to irradiation damage. Such highly non-equilibrium structures, however, are generally unstable to coarsening during exposures to long term irradiation and operation at very high temperatures. We exam here the potential for developing alloys that self organize on an ultrafine length scale during high temperature irradiation, and thus maintain their radiation tolerance. By using a combination of x-ray diffraction, transmission electron microscopy, and atom probe tomography measurements analyze a series dilute Cu alloys, we show that ultrafine microstructures can indeed be preserved at irradiation temperatures exceeding 650 °C (i.e., > 0.62TM, where TM=melting temperature of Cu) and to irradiation doses greater than 100 dpa. Molecular dynamics computer simulations provide a clear explanation for the stability of some of these microstructures.

12:15 PM
Self Organization in Irradiated Cu-10at%Fe Alloys: An Atom Probe Tomography Investigation: *Brad Stumphy*[1]; See Wee Chee[1]; Robert Averback[1]; Pascal Bellon[1]; [1]University of Illinois

Atom probe tomography (ATP), in combination with magnetization measurements, was employed to investigate irradiation-induced precipitation in dilute Cu-Fe alloys. Thin-film specimens, ˜200 nm thick, were fabricated using magnetron sputtering. For the magnetization measurements, the alloys were deposited directly on heavily oxidized Si wafers, while for the APT, the alloys were deposited onto the tips of Mo wires, using a Ti layer to enhance the cohesion of the film. The ATP specimens were shaped using a focused ion beam. Irradiation at 80 K led to the complete dissolution of Fe precipitates. Between room temperature and ˜ 250°C, precipitation was observed, but the sizes of the precipitates saturated with dimensions less than ˜ 5nm. Solubilities of Fe in the Cu matrix and Cu in the Fe precipitates, and the interface diffuseness, were determined as a function of temperature in both the irradiated and unirradiated samples.

Synergies of Computational and Experimental Materials Science: Three-Dimensional Materials Science I

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Katsuyo Thornton, University of Michigan; Henning Poulsen, Risoe National Laboratory; Mei Li, Ford Motor Co

Monday AM — Room: 3003
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Katsuyo Thornton, University of Michigan; Henning Poulsen, Risoe National Lab

8:30 AM Introductory Comments

8:35 AM Invited
Microstructure Evolution and Fundamental Characteristics in Solidification of Metals and Alloys - A Comparison of Modelling and Experiments: *Ragnvald Mathiesen*[1]; [1]NTNU

Alloy solidification processes generally evolve under non-equilibrium conditions, where the solid grow as intricate self-assembly structures controlled by complex interplays of diffusive and hydrodynamic heat and mass transport, with the solid-liquid interface both as an internal bondary and a solution to the problem itself. While modelling of solidification microstructures and fundamentals has advanced considerably over the last decades, provision of new experiments for guidance has fallen behind. In the past in-situ studies were limited to video microscopy on optically transparent model systems. However, these are severely limited as analogues to real alloys, and can only be used to realize a few cases. Recent improvements in sources and detectors have opened for X-ray investigations at spatiotemporal resolutions approaching those of

video microscopy. Here, in-situ X-ray imaging observations from solidification studies in Al-based alloys will be presented, and compared qualitaviely and quantitatively with results from modelling.

9:15 AM Invited
Using Experimental Data in Simulations of Grain Growth: I. McKenna[1]; D. Rowenhorst[2]; E.M. Lauridsen[3]; *Peter Voorhees*[1]; [1]Northwestern University; [2]Naval Research Laboratory; [3]RISO Laboratory

Recent advances in computational and experimental techniques allows for the routine visualization of the three-dimensional grain structure of materials. This opens new routes to explore the relationship between materials processing, structure, and properties. Using experimentally measured three-dimensional grain structures we have followed the evolution of grains using a phase field model that accounts for all five degrees of freedom that determine the grain boundary energy. We show that a multiorder parameter model can be used to explore the topological changes of individual grains during grain growth. This model employs quarternions to account for the dependence of the grain boundary energy on the misorientation and a tensor gradient energy coefficient to account for the change in grain boundary energy with boundary normal. Results of the simulations and the development of the models will be discussed.

9:55 AM
Modeling and In-Situ X-Ray Video Microscopy of Confined Equiaxed Grain Growth and Buoyant Motion in Al-Cu: *Pierre Delaleau*[1]; Ragnvald Mathiesen[2]; Paul Schaffer[1]; Lars Arnberg[1]; Martin Bellmann[1]; Christoph Beckermann[3]; [1]NTNU, Department of Materials Science and Engineering; [2]NTNU, Department of Physics; [3]The University of Iowa, Department of Mechanical and Industrial Engineering

Equiaxed dendritic growth in grain refined Al-x%wtCu (x=15-25) has been studied in situ during directional solidification by means of synchrotron X-ray video microscopy. At these compositions, the α-Al grains have a lower density than the surrounding melt and experience buoyant forces which affect their growth rates and morphologies. As the samples are concealed into a thin container, the walls severely influence grain motion. A model, based on a spherical envelope approximation to the dendrite morphology in order to simplify both the interface geometry of the growing crystals and the Stokes drag exerted upon them during motion, has been derived taking into account the influence of the sample confinement. The model is compared with the in situ experiments both to evaluate its present merits and to devise possible routes for improvement in order to develop it further to a model description for α-Al dendritic growth during buoyant motion.

10:15 AM
Predicting the Evolution of Interfacial Morphology during Coarsening: *Larry Aagesen*[1]; Julie Fife[1]; Peter Voorhees[1]; Erik Lauridsen[2]; Marco Stampanoni[3]; [1]MSE Dept., Northwestern University; [2]Riso National Laboratory; [3]Swiss Light Source

The process of coarsening in two-phase systems is governed by interfacial morphology. A new method of predicting the evolution of interfaces and their morphologies was developed. The method uses a phase-field model of a binary alloy which allows for unequal diffusivities between the liquid and solid phases, and accounts for changes to interfacial morphology due to the motion of the interface itself. To validate this method, experimental data was used as input to the phase-field model, and simulation results were compared to data at later times. The experimental data was from a directionally solidified Al-Cu alloy which was coarsened just above the eutectic temperature and observed using in-situ X-ray tomography at the Swiss Light Source. This provided three-dimensional data of the microstructure throughout the coarsening process. The comparison allowed the fine-tuning of simulation parameters to more closely match experimental results, and thus improved the accuracy of the predictions.

10:35 AM Break

10:50 AM Invited
3DXRD Characterization and Modelling of Recrystallization: *Dorte Jensen*[1]; [1]Riso - DTU National Lab

3D x-ray diffraction (3DXRD) allows non-destructive characterizations of bulk microstructures and strains. The method is described briefly with focus on recent developments. 3DXRD results obtained so far have in particular highlighted the importance of LOCAL phenomena which are typically not at all (or incorrectly) incoorporated in excisting models. This is illustrated for recrystallization of metals. It is shown how 3DXRD measurements have led to new 3D geometrical modelling and 3D MD simulations nessesarry to explain the experimental results and in turn how the modelling and simulations have guided new 3DXRD experiments.

11:30 AM
Phase Field Simulations of Coarsening of Al6Mn Precipitates Located on Grain Boundaries in Al Alloys: *Nele Moelans*[1]; Alexis Miroux[2]; Erica Anselmino[2]; Sybrand van der Zwaag[3]; Bart Blanpain[1]; Patrick Wollants[1]; [1]K. U. Leuven; [2]M2i; [3]Delft University of Technology

In-situ observations show that grain boundary movement during recrystallization in aluminum alloys is not smooth but jerky on a microscopic scale. There is experimental and theoretical evidence that the jerky motion is due to the pinning effect of small Al6Mn precipitates. The pinning precipitates are however too small to study this effect in detail from in-situ observations. Therefore phase field simulations are performed of the recrystallization in Al-alloys that account for the interaction between precipitates and grain boundaries. Model parameters are, as far as possible, determined based on experimental information. Comparison of the phase field simulations with the in-situ observations will give a better understanding of the mechanisms behind and conditions for jerky grain boundary motion.

11:50 AM
Influence of Grain Boundary Misorientation on Nucleation of Twins in Textured Zr: *Dhriti Bhattacharyya*[1]; Rodney McCabe[1]; Carlos Tome[1]; [1]Los Alamos National Laboratory

Deformation twinning is a major method of strain accommodation in hcp metals like Zr. It is well known that grain orientation with respect to the applied load (Schmid factor) has a significant effect on the formation of twins in any grain. Another important factor for the nucleation of twins in a grain may be its misorientation with neighboring grains. In this study, we have used electron-backscatter diffraction (EBSD) data to investigate the latter effect, by correlating the misorientation between the active twin and slip systems in the neighboring grains on twin-formation in a given grain. Specifically, we investigate whether dislocation or twin induced stress concentrations at a grain boundary can cause twin nucleation in a neighboring grain. Extensive studies made over hundreds of grain-boundaries indicate that misorientation with the neighboring grain is an important factor for nucleation of twins at the boundaries.

Transformations under Extreme Conditions: A New Frontier in Materials: Keynote: Melting and Solidification I

Sponsored by: The Minerals, Metals and Materials Society, ASM International, ASM Materials Science Critical Technology Sector, TMS Materials Processing and Manufacturing Division, TMS/ASM: Phase Transformations Committee
Program Organizers: Vijay Vasudevan, University of Cincinnati; Mukul Kumar, Lawrence Livermore National Laboratory; Marc Meyers, University of California-San Diego; George "Rusty" Gray, Los Alamos National Laboratory; Dan Thoma, Los Alamos National Laboratory

Monday AM — Room: 3001
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Mukul Kumar, Lawrence Livermore National Laboratory; Srikumar Banerjee, Bhabha Atomic Research Center

8:30 AM Introductory Comments

8:40 AM Keynote
Phase Transformation Kinetics and Mechanisms in Shocked Condensed Matter: Challenges and Opportunities: *Yogendra Gupta*[1]; [1]Washington State University

Shock wave experiments provide a unique approach to examine compression induced phase transformations in real time. Continuum measurements under shock loading (either peak state values or wave profile measurements) in conjunction with static pressure results are most commonly used to infer thermodynamic states, transformation mechanisms, and kinetics. Challenges associated with these traditional approaches will be discussed using representative examples. Understanding the role of crystal orientation, material microstructure, and stress deviators on phase transformations constitute long standing needs.

New experimental capabilities that have the potential to provide an in-depth understanding of transformation mechanisms and kinetics will be outlined. The combination of dynamic loading capabilities, recent computational developments, and new in-situ microscopic measurements presents an exciting opportunity to understand transformation of materials at extreme conditions.

9:30 AM Invited
First Principles Calculations of Shock Hugoniot and Shock Induced Melting of Osmium: Keshaw Joshi[1]; Satish Gupta[1]; *Srikumar Banerjee*[1]; [1]Bhabha Atomic Research Centre

The stability of crystal structure of osmium under application of high pressure has been examined by carrying out the first principles calculations of total energy at various compressions for hcp, bcc, omega (a three atom simple hexagonal) and fcc structures. Our analysis indicates that the ambient hcp phase remains lowest energy structure up to a hydrostatic pressure of ~ 698 GPa (V/V0 = 0.58). The shock Hugoniot derived from 0 K isotherm by incorporating the thermal lattice and thermal electronic contribution in conjunction with the Rankine Hugoniot relation yields C = 4.49 km/s and s = 1.304 in the Us – Up plot. The melting line has been constructed by applying the theoretically determined pressure dependent Gruneisen parameter in Lindemann criterion of melting. The intersection of melting line with the shock Hugoniot, indicating the melting of osmium under shock compression, occurs at ~ 440 GPa (T = 8944 K).

10:05 AM
Thermodynamics of the γ-α Transition in Cerium with Phonon Contributions: *Yi Wang*[1]; L. Hector[2]; Hui Zhang[1]; Shun-Li Shang[1]; Long-Qing Chen[1]; Zi-Kui Liu[1]; [1]The Pennsylvania State University; [2]GM R&D Center

Thermodynamics of the γ-α transition in Cerium are investigated with a model that accounts for finite temperature mixing of the free energies of the nonmagnetic and magnetic Ce 4f-states. All model inputs are taken from first-principles density functional theory (DFT) calculations with strong correlation of the f-electrons. Vibrational free energies are computed with phonon calculations based upon the direct approach to lattice dynamics. This provides the correct phase transition thermodynamics as demonstrated in our computed free energy curves over 0 to 600 K and temperature-volume phase diagram. We find remarkably close agreement between our computed 0 GPa phase transition temperature, critical point, and 300 K γ-α volume collapse and experiment. Our model, which does not rely upon existing experimental data or other approximations outside of DFT, provides a framework for accurate prediction of the temperature-pressure behavior of other f-state systems, such as Plutonium.

10:25 AM Break

10:40 AM Invited
Dynamic Phase Transitions Compared with Static; Flat Melting Curves and Other Mysteries: *Robert Hixson*[1]; [1]Naval Postgraduate School

In this talk I'll show comparisions of phase transtion locations for selected metals determined using static high pressure techniques with those made using shock wave compression techniques. I will in particular look at available data for melting curves measured using static high pressure techniques, and make comparisons with Hugoniot melting points determined from shock compression experiments. There are only a few metals for which shock wave Hugoniot melting points have been determined, and many of these will be discussed. One focus will be on iron. I'll also briefly review and discuss recent work that shows relatively flat melting curves determined from diamond anvil cell data.

11:15 AM
Pressure Induced Solidification of Ta and Cu: A Comparison: *David Richards*[1]; James Glosli[1]; Fred Streitz[1]; [1]Lawrence Livermore National Laboratory

Using powerful computers such as Blue Gene/L it is now possible to use classical molecular dynamics to simulate pressure induced solidification at size scales that are free of finite size effects. We present a comparison of the nucleation, growth, and coalescence of clusters during pressure induced solidification in large scale MD simulations of liquid Ta and Cu. We extract growth and nucleation rates from our simulations, as well as cluster size distributions that can be compared against the predictions of simple models. This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344 UCRL-ABS-2367881.

11:35 AM
New Phase Diagram of Ta: Bridging Laser Heated Diamond-Anvil Cell and Shock Melting: *Christine Wu*[1]; Per Soderlind[1]; James Glosli[1]; John Klepeis[1]; [1]Lawrence Livermore National Lab

Determination of the melt line of materials under high pressures is essential for establishing its phase diagrams and has important implications for geophysics, material science, and high-pressure physics. So far, melting temperatures at high pressure are primarily measured by in situ laser-heated diamond-anvil cell (DAC) or shock wave experiments. Often, these two methods yield significantly different results, particularly for non close-packed metals, such as bcc metals. For instance, anomalously flat melting slopes were reported for numerous bcc metals by laser-heated DAC. The flatness of the melting slope is in sharp contrast to the classical Lindemann behavior which shock-melting temperatures follow closely. In this presentation, we will report a novel phase diagram of Ta obtained from ab initio methods, and molecular dynamics (MD) simulations, which resolves the long-standing controversy, and has significant impact on our understanding of phase diagrams of bcc metals. This work preformed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

11:55 AM
Pressure-Driven Solidification: Coupling Phase-Field Modeling with Underlying Molecular Dynamics: *James Belak*[1]; Patrice Turchi[1]; Milo Dorr[1]; Bryan Reed[1]; David Richards[1]; Jean-luc Fattebert[1]; Michael Wickett[1]; Fred Streitz[1]; [1]Lawrence Livermore National Lab

Large parallel computers have enabled MD simulations of pressure-driven solidification of sufficient scale to observe the formation of realistic microstructure. Here, we calculate the coarse-grained phase-field order parameter from the local atomic coordinates within the MD. The results are represented within emerging crystallographic phase-field models and validated through overlapping MD and phase-field simulations. Results will be presented for the solidification of tantalum. F. H. Streitz, J. N. Glosli, and M. V. Patel, Phys. Rev. Lett. 96, 225701 (2006). R. Kobayashi and J.A. Warren, Physica A, 356, 127-132 (2005). T. Pusztai, G. Bortel and L. Granasy, Europhys. Lett, 71, 131-137 (2005).This work performed under the auspices of the U.S. Department of Energy by LLNL under Contract DE-AC52-07NA27344.

2009 Functional and Structural Nanomaterials: Fabrication, Properties, and Applications: Low Dimensional Nanostructures II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Nanomaterials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Gregory Thompson, University of Alabama; Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute; Jiyoung Kim, University of Texas at Dallas; Seong Jin Koh, University of Texas at Arlington; Wonbong Choi, Florida International University; Alexander Howard, Air Force Research Laboratory

Monday PM
February 16, 2009

Room: 3018
Location: Moscone West Convention Center

Session Chairs: Seong Jin Koh, University of Texas at Arlington; William Ready, Georgia Tech

2:00 PM Invited

Ferromagnetic Nanoparticles: *J. P. Liu*[1]; [1]University of Texas at Arlington

Most ferromagnetic particles lose their hysteresis when their size is reduced to nanoscale, except few materials like FePt and SmCo compounds with extremely high magnetocrystalline anisotropy can hold a permanent magnetic moment at room temperature in particles of few nanometer size. By applying newly developed "salt-matrix annealing" and "surfactant-assisted milling" techniques, monodisperse ferromagnetic FePt and SmCo nanoparticles have been successfully synthesized. These first-ever-available nanoparticles display various ferromagnetic properties at room temperature which are found to be strongly size dependent. The ferromagnetic nanoparticles are used as building blocks for advanced bulk and thin film magnets, and can be also applied in biomedical technologies.

2:30 PM

Nanoscale Modeling Studies of Magnetic Flux Closure in Cobalt Nanoparticles: *Prabeer Barpanda*[1]; [1]Rutgers University

Soft magnetic rings are promising candidates for nonvolatile random access memory (RAM) devices due to their capacity to support bistable flux closure (FC) domains. Cobalt nanoparticles form one such soft magnetic ring system, having zero magnetostatic energy and can be closely packed for high-density data storage. Flux closure states in Cobalt nanoparticles can be rapidly (picoseconds range) switched by in-plane magnetic fields/ coaxial currents. In the current study, 3D-FFT micromagnetic modeling has been employed to examine magnetic states in nanoparticle rings that are formed from 20-nm-diameter cobalt crystals. We assess the effect of the number of particles in a ring on the formation of flux closure or 'onion' states at remanence after the rings are subjected to out-of-plane fields. We compare the simulations with experimental magnetic induction maps measured using electron holography and assess the influence on the reversal mechanism of a ring of the morphologies of the constituent crystals.

2:45 PM

B/SiOx Nanonecklace Reinforced Nanocomposites by Unique Mechanical Interlocking Mechanism: Xinyong Tao[1]; Jie Liu[1]; Goutam Koley[1]; *Xiaodong Li*[1]; [1]University of South Carolina

Necklace-like nanostructures with SiOx beads on boron strings were self-assembled via a facile environment-friendly method at atmospheric pressure. The electrical conductivity of the boron string is a thousand times higher than that of pure bulk boron (10-6 O-1cm-1). Due to the unique mechanical interlocking between beads and epoxy matrix, the reinforcement effect of the nanonecklaces in epoxy is even better than normal carbon nanotubes. B/SiOx nanonecklaces are expected to exhibit unique electrical and mechanical properties for constructing nanodevices and nanocomposites.

3:00 PM

Synthesis and Magnetic Properties of FePt and FeRh Mixed Nanoparticles: *Naidu Seetala*[1]; Jessica Harris[1]; Joseph Buchanan-Vega[1]; J. W. Harrell[2]; Zhiyong Jia[2]; David Nikles[2]; [1]Grambling State University; [2]University of Alabama

We have examined the properties of FePt and FeRh nanoparticles for the heat assisted magnetic recording (HAMR) media applications. FePt and FeRh nanoparticles (~ 6 nm) were chemically synthesized using simultaneous polyol reduction method. The FeRh nanoparticles were annealed in salt at 800°C to avoid particle segregation and sintering. The XRD results show CsCl-type bcc (B2) phase for FeRh upon salt annealing. The temperature dependent magnetic studies of annealed FeRh nanoparticles showed anti-ferromagnetic to ferromagnetic transition at around 80°C. The high temperature synthesis of FePt nanoparticles provided $L1_0$ phase with a magnetic coercivity of ~ 2000 Oe for as-synthesized particles. The temperature dependence (20 - 230°C heating and cooling cycles) of the magnetic properties were studied individually for FePt and FeRh nanoparticles, and after physically mixing them and annealing at 400°C. Temperature dependent hysteresis behavior is observed in all the samples.

3:15 PM

Preparation of Nanostructered Iron Oxide Particles via Ultrasonic Spray Pyrolysis (USP): *Burcak Ebin*[1]; Sebahattin Gurmen[1]; Cuneyt Arslan[1]; [1]Istanbul Technical University

Scientific and technological attentions have focused on synthesis and characterization of nanostructured iron oxide particles in recent decades due to their interesting physical and chemical properties. Especially novel magnetic properties of nanosized particles could open new practical applications in many fields such as magnetic storage devices, ferro fluids, catalysis, magnetic drug delivery system, and cancer treatment.In this research, nanostructured iron oxide particles were prepared via ultrasonic spray pyrolysis (USP) method using iron (II) chloride solution. The dependence of iron oxide particles size and morphology to the precursor concentration, and reaction temperature were investigated under 1.3 MHz ultrasonic frequency, and 1.0 l/min air flow rate. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to investigate size, morphology and crystal structure of particles. It was observed that decreasing of precursor concentration and temperature cause the reducing in particle size.

3:30 PM Break

3:45 PM

Solution and Low Temperature Synthesis of a Conductive and Porous Metal-Silica Nanocomposite: Tsan-Yao Chen[1]; Yong-Jae Choi[1]; *Tzy-Jiun Luo*[1]; [1]North Carolina State University

In our effort to control and synthesize metallic nanoparticles that exhibit interconnected 3-D network, we have developed a low temperature and solution procedure to fabricate a porous metal-silica nanocomposite. We used silver as a test model and successfully synthesized a metal-silica nanocomposite that is highly conductive (< 2 ohm-cm), low density (~ 2 g/mL), with low weight percentage of silver (2 ~ 5 wt%). This material is nanoporous in nature and was synthesized using polyethyleneglycol blended sol-gel matrix as structural template. It consisted of three major components: nanoporous silica matrix, pore-filled polymers, and silver ions that were later reduced to interconnected silver network at 160°C. Such material will find its applications in fuel cells, biofuel cell, and sensors. Therefore, SEM, XRD, Tapping mode AFM and cyclic voltammetry were utilized to characterize its nanostructure and properties.

4:00 PM

Study on Microstructure and Emission Properties of Scandate Cathode: *Wei Liu*[1]; Jinshu Wang[1]; Yiman Wang[1]; Meiling Zhou[1]; [1]Beijing University of Technology

The sub-micron Sc2O3 doped tungsten powders have been successfully prepared by Sol-Gel and two-step reduction method for the first time. Then, the Scandia doped tungsten mixed matrix impregnated cathodes with the sub-micron structure has been also successfully prepared. 50A/cm2 of Jdiv and 100A/cm2 of J10% at 850°Cb are obtained for the optimally activated cathodes. By using in situ AES, HRSAM and other kinds of analysis methods, it is found that BaSc and O diffuse from the interior porous body to surface simultaneously to form a uniform activator substance layer with the optimal atomic ratio. The comparison experiment between "M"-type cathode and sub-micron Scandate cathodes displays that the thickness of the activator substance layer in Scandate cathodes is larger than that in "M"-type cathodes, indicating that this uniform

activator layer has a multi-layer structure which leads to the excellent emission property of this cathode.

4:15 PM
Gram-Scale Synthesis of Functionalized, Highly Fluorescent, and Non-Toxic Silicon Nanoparticles: Han Zuilhof[1]; [1]Wageningen University

Non-toxic fluorescent nanoparticles are highly desirable for a wide variety of bio-imaging studies. To this aim we developed methods to synthesize functionalized, oxide-free silicon nanoparticles. These are brighly fluorescent, with a narrow emission due to a very narrow size distribution (1.6 +/- 0.2 nm). Since they can be synthesized on a gram scale, this allows for the first time a range of optical properties (both steady state and time-resolved), bio-imaging of yeast cells using functionalized Si nanoparticles, and toxicity studies on different cell lines. The paper discusses this synthesis, opto-electronic properties, preliminary bioimaging studies and detailed quantitative studies on the (lack of) toxicity of these nanoparticles.

4:30 PM
Synthesis of Sb4O5Cl2 Nanobelts by Hydrolysis of Alkoxide and Thermal Decomposition Properties of the Novel Nano-Flame Retardant: *Li Feng*[1]; [1]China University of Mining and Technology

Novel flame retardant of antimony oxychlorides (Sb4O5Cl2) nanobelts have been synthesized via alkoxide hydrolysis. X-ray diffraction (XRD) measurement showed that the samples were pure Sb4O5Cl2 crystals with monoclinic structure. The fibers-like structure of Sb4O5Cl2 nanobelts with 0.3-1.0 μm in length and 10-50 nm in diameter were confirmed by TEM. The thermal analysis (TG/DTG/DSC) revealed that there were three steps of mass loss of the products under nitrogen atmosphere, and the possible mechanisms for the decomposition of Sb4O5Cl2 were discussed.

Alumina and Bauxite: Bayer Process Safety, Enviromental and Sustainability Issues

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Everett Phillips, Nalco Co; Sringeri Chandrashekar, Dubai Aluminum Co

Monday PM — Room: 2002
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Pierre Ferland, Rio Tinto Alcan

2:00 PM Introductory Comments

2:10 PM
The Asia-Pacific Partnership: An Important New Initiative for a Sustainable Alumina Industry: *Markus Gräfe*[1]; Greg Power[1]; Craig Klauber[1]; [1]CSIRO Minerals

The Asia-Pacific Partnership on Clean Development and Climate is an inter-governmental agreement between seven countries, predominantly located around the Pacific Rim: Australia, Canada, China, India, Japan, Republic of Korea and the United States of America. Collectively these countries represent about half the world's emissions and 52% of the world's aluminium production. Aluminium is one of eight key areas covered by the APP and the Task Force is chaired by Australia and co-chaired by the United States of America. Projects within this area are focused on best practice and its deployment across the Partnership economies. Within aluminium there are seven project activities covering benchmarking and linkages to technology providers, plus per-fluorocarbon emissions, bauxite residues, high silica bauxite, fluoride emissions and aluminium recycling. The Partnership program and how it is implemented is described with a particular focus on the bauxite residue management work being undertaken.

2:35 PM
Operations Support in the Alumina Industry – A Valuable Partnership: *Jason Berzansky*[1]; [1]Hatch Associates Consultants

Since 2001, Hatch Associates Consultants has been involved in a highly successful engineering alliance with an alumina supplier for the client's sustaining capital program. As part of Hatch's Operations Support (OpSupport) network, this relationship has flourished over the past seven years, as evidenced by the recent extension of the alliance agreement. While the primary focus of this engineering alliance is on the sustaining capital program, Hatch has added value to the client through a variety of other activities such as maintenance and operations support activities. This paper focuses on the sustaining capital engineering alliance concept and demonstrates how such relationships can achieve success when common goals are established, agreed upon and eventually realized.

3:00 PM
Sustainable Storm Water Management: *Dana Smith*[1]; Jaw Fu[2]; Amanda Ludlow[3]; [1]AWA Atlantic; [2]Alcoa; [3]Roux Associates, Inc

Alcoa's Point Comfort, Texas alumina refinery has deployed a multi-faceted Engineered Natural System (ENS) to capture and recycle bauxite ore, improve runoff water quality and reduce runoff volume from the bauxite storage area at the Site. The ENS is comprised of initial sedimentation trenches and swales to capture and recover course fractions of bauxite eroded from large bauxite storage piles. Decanted stormwater from the trenches is then conveyed to a staged constructed treatment wetland (CTW) for additional cleansing and retention. The staged CTW contains an initial forebay, settling pond, high marsh and low marsh areas and a terminal micropool. Clean effluent from the CTW can either be conveyed to a phytoplot for consumptive elimination or used by the refinery to reduce dependency on potable water.

3:25 PM
Achieving Excellence in Liquid Effluent Treatment at Alunorte: *Jorge Aldi*[1]; [1]Alunorte – Alumina do Norte do Brasil S.A

Alunorte began its operation in 1995 with a capacity of 1.1mi t/y and after three Expansions the production in 2009 will be 6.3mi tpy. As Alunorte use the dry stacking technology to dispose the red mud, the area for the red mud deposit is very big as well the pluvial index in the rain forest region. So in 2003, after the first Expansion, Alunorte have decided to build a new liquid effluent treatment station to guarantee a nominal capacity of 3,600m3/h of treated effluent, ensuring a pH around 8.0, a temperature below 40°C and a NTU below 20. This paper aims to present the concept implemented for such an effluent treatment station as well the results achieved to date after the implementation, emphasizing that all kinds of liquid effluents, no matter if it is contaminated or not, are treated before being discarded in the river.

3:50 PM Break

4:05 PM Invited
Sustainability of Chinese Alumina Production from High Silica Diasporic Bauxite: *Songqing Gu*[1]; Zhonglin Yin[1]; [1]Zhengzhou Research Institute of Chalco

The sustainable development of Chinese alumina production faces great challenges from the low grade bauxite resource, high process energy consumption, product quality and environmental issues. Improving flotation-Bayer process and developing the hydro-chemical processes to produce suitable DSP will be the key solutions for efficiently processing high silica bauxite with lower consumptions and more competitive cost. Developing new technologies for energy savings and high alumina recovery to reduce residue disposal, enhancing circulation efficiency and productivity in the various production stages for high output and low consumptions, realizing dry residue disposal and zero- waste water discharge, reusing red mud for new materials manufacture and valuable elements recovery will provide a vital basis for longer term sustainability of the Chinese alumina industry.

4:30 PM
The Design of Pressure Safety Systems in the Alumina Industry: *Brady Haneman*[1]; [1]HATCH Associates

The alumina refinery presents the designer with multiple challenges. For a given process flowsheet, the mechanical equipment installed must be routinely inspected and maintained. Piping systems must also be inspected routinely for signs of erosion and/or corrosion. Rapid deposits of chemical species such as lime, silica, and alumina on equipment and piping need special consideration in the mechanical design of the facilities such that fluid flows are not unduly interrupted. Above and beyond all else, the process plant must be a safe place of work for refinery personnel. This paper outlines some of the pressure safety considerations to be incorporated into the mechanical design of the digestion facilities for some alternate process flow sheets. Armed with these considerations at the process flowsheet definition stage, optimisation of the process and/or

equipment selection is possible preserving the delicate balance of process facility performance, plant operability and maintainability, and personnel safety.

4:55 PM

Mercury Vapor Sensor for Alumina Refinery Processes: Ylias M. Sabri[1]; Samuel J. Ippolito[1]; *Suresh Bhargava*[1]; [1]RMIT University

The sustainability of bauxite mining and refinery practices is reliant on attempts to reduce the environmental impacts of traditional processes. Mercury reduction targets set by industry and regulators has spurred attempts to develop technologies for evaluating the efficiency of mercury removal processes. The development of a mercury sensor suited to alumina refineries will be a significant breakthrough in controlling mercury emissions, as well as having many other applications. Gold coated Quartz Crystal Microbalance (QCM) based sensors employing enhanced nano-structured surfaces have been developed which show a substantial increase in response magnitude of at least 67% over non-modified QCMs. Additionally, the Hg-Au sticking probability calculated from the QCM data of the modified and non-modified sensors showed increased Hg affinity for the modified sensor. Furthermore, the modified QCM sensor was found to have better repeatability and stability while having 47 fold lower drift at the higher operating temperature, when compared to its non-modified counterpart.

5:20 PM Concluding Comments

Aluminum Alloys: Fabrication, Characterization and Applications: Development and Application

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee

Program Organizers: Weimin Yin, Williams Advanced Materials; Subodh Das, Phinix LLC; Zhengdong Long, Kaiser Aluminum Company

Monday PM — Room: 2004
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Shridas Ningileri, Secat Inc

2:00 PM

Development of Low-Cost, High-Performance AlZn4.5Mg1 Alloy 7020: *John Chinella*[1]; [1]U. S. Army Research Laboratory

This paper reviews properties, processing, and performance of Cu-free Al-Zn-Mg alloy 7020. Comparisons are made with alternative military aluminum alloys' chemistry, material costs, properties, levels of strength and ductility, and resistance to stress and exfoliation corrosion. The approach and experimental results for development and optimization of alloy 7020 for vehicle armor or welded structures are identified and described either for the mill, solution treated quenched and aged, or high-temperature aged conditions. Advantages for military and commercial use include: (1) low thermal sensitivity of the microstructure and mechanical properties to deleterious effects either from reheat or solution-treatment quench, (2) low material cost, (3) medium strength, and (4) high levels of weld strength and ductility in the natural or artificial aged condition.

2:20 PM

Aluminum Sheet Applications and Manufacturing Challenges in the Automotive Industry: *Susan Hartfield-Wunsch*[1]; Jody Hall[1]; [1]General Motors Corp

Application of aluminum for mass reduction in automobiles has been a topic of discussion for several decades. Aluminum casting and extrusion applications are pervasive in powertrain and chassis components. In contrast, aluminum sheet metal has only limited application in automotive bodies, and is currently found predominantly on higher-end vehicles. New CAFÉ requirements and higher fuel costs have increased pressure on the automotive industry to improve fuel economy and reduce emissions. This paper will explore the reasons for the 'limited and slow' implementation of sheet aluminum into automotive bodies and make a prediction of how this scenario will change. It will also cover the major challenges in manufacturing aluminum sheet metal components and outline where additional research and development projects can help accelerate production applications.

2:40 PM

High Strength Aluminum Sheet for Automotive Applications: *Dirk Uffelmann*[1]; [1]AMAG Rolling GmbH

The most common aluminum alloys for automotive sheet applications are work-hardening 5000-series and heat-treatable 6000-series alloys. They are used in a wide range of structural parts, components and hang-on-parts. From a general view, the usage of these alloys results in a reasonable ratio of cost per weight saving and a good compability with existing production methods in terms of forming and joining. Superior yield strength and tensile strength can be achieved by heat-treatable 2000- and 7000-series alloys, commonly used for aircraft applications. For certain automotive applications, there is additional weight saving potential by use of these high-strength aluminum alloys. The purpose of this paper is to show the possibilities and limitations of weight saving by usage of high-strength aluminum alloys with respect to production (forming, joining, heat treatment) and performance (corrosion, fatigue, crash performance).

3:00 PM

Development of Twin-Belt Cast AA5XXX Series Aluminum Alloy Materials for Automotive Sheet Applications: *Pizhi Zhao*[1]; Toshiya Anami[1]; Ichrio Okamoto[1]; Kazumitsu Mizushima[1]; Kevin Gatenby[2]; Mark Gallerneault[2]; Simon Barker[2]; Kunihiro Yasunaga[3]; Akira Goto[3]; Hitoshi Kazama[3]; Noboru Hayashi[3]; [1]Nippon Light Metal Company, Ltd.; [2]Novelis Global Technology Center; [3]Honda R&D Co., Ltd

Process routes for AA5XXX series aluminum alloy sheet produced via a twin belt caster (FLEXCASTER)have been successfully trialed. The FLEXCAST AA5XXX sheet has a fine intermetallic and grain structure compared to conventional DC processed AA5XXX aluminum alloy sheet as a consequence of the high cooling rate during solidification. Optimization of composition and refinement of microstructure results in superior dome stretchability and lower susceptibility to SCC than DC AA5182 sheets. Moreover, the FLEXCAST AA5XXX aluminum alloy sheet shows good performance in coating and adhesive bonding tests, which are critical for automotive structure parts.

3:20 PM

Microstructure-Property Correlation of Aluminum Alloy 2219 Produced by Electron Beam Freeform Fabrication: *Ravi Shenoy*[1]; Marcia Domack[2]; [1]Lockheed Martin Mission Support; [2]Advanced Materials and Processing Branch, NASA Langley Research Center

Electron beam freeform fabrication (EBF3) is a layer-additive manufacturing process wherein a metal wire of required alloy composition is fed at a controlled speed into a molten pool created on a metal substrate surface, using a focused electron beam. The component geometry is achieved through the ensuing solidification, by translating the substrate with respect to the beam.In the present study, cast and precipitate microstructures unique to complex thermal histories experienced during successive layered EBF3 depositions in aluminum alloy 2219 were characterized using electron microscopy, microtexture, and thermal analysis in order to investigate the metallurgical mechanisms contributing to the observed properties. The strength level of as-deposited 2219 was between O and T4 temper wrought products and within 2% of T6 temper products after heat treatment. The results of the investigation are presented in relation to process parameters employed such as the translation speed, wire feed rate, and beam power.

3:40 PM

A Novel Thermomechanical Processing Method to Achieve Fine-Grained AA6xxx Sheet: *Shahrzad Esmaeili*[1]; David Lloyd[2]; Haiou Jin[2]; [1]University of Waterloo; [2]Novelis Global Technology Centre

A novel thermomechanical processing method to produce fine-grained sheets of heat treatable aluminium alloys has been developed. The method, which includes a continuous cold rolling and annealing process, has been applied to an AA6xxx alloy plate and a fine-grained sheet with desirable microstructural characteristics has been achieved. The fabricated sheet has shown significantly enhanced ductility in wide ranges of temperatures and strain rates and therefore provides a potential solution to the formability issue in automotive applications of AA6xxx sheets. The present work will outline the processing route and the characteristics of the fine-grained AA6xxx alloy in comparison with the conventionally-produced coarse-grained version of the alloy.

4:00 PM Break

4:15 PM
Re-Use of Aluminum Turning Chips by Hot Extrusion: *Klaus Pantke*[1]; Dirk Biermann[1]; [1]University of Dortmund

Aluminum is the most widely used metal after steel in the manufacturing industry. Due to the convenient material properties, this material can be used in several products. The energy requirement for producing and melting aluminum is one of the major disadvantages of this material. Although the re-melting of scrap aluminum can reduce the energy requirements, the needed energy even for the melting process is still high. This article presents a process chain of direct conversion technology of aluminum chips by cutting, compaction of the chips to billets, hot extrusion to a rectangular square profile, and finally characterization of the profile properties. It is shown that, by direct conversion, a melting of the chips for secondary use can become unnecessary. Due to the fact, that this process chain doesn´t need a melting process, there will be great advantage over the conventionally process chain to save ecological and economical resources.

4:35 PM
Establishing Foil Stock Production through Continuous Casting Route - Our Experience in BALCO: *P.K.N. Raghavan*[1]; Mousumi Kar[1]; Diwakar Singh[1]; [1]Bharat Aluminium Co. Ltd., (A Unit of Vedanta Resources Plc.)

Coils for the production of thin foils can also be done through continuous strip casting process which is directly cold rolled. The alloys processed in a continuous strip casting process result in foil stock which has a higher supersaturation of solute elements, and therefore has undesirable hardening and softening properties, causing difficulties in rolling the foil stock to the final gauge thickness. The various difficulties faced in production of AA 8011 foil stock through twin roll continuous strip casting process and the corrective measures taken at Balco to overcome the difficulties for producing good quality foil stock have been elaborated in this paper. Microstructural examination and structure-property correlation of foil stocks produced through DC casting and Continuous strip casting routes have also been discussed in detail. The advantages of this process viz the Direct Chill Casting route will also be discussed.

4:55 PM
On the Distortion and Warpage of 7249 Aluminum Alloy after Quenching and Machining: *Omar Es-Said*[1]; Eui Lee[2]; [1]Loyola Marymount University; [2]Naval Air Systems Command

From large extrusion plates of 7249 Aluminum Alloy with fins, T sections of length 25.4 cms (10 inches) and width 4.6 cms (1.8 inches) were cut. Three solution temperatures, two quenching media, two aging treatments, and three machine cuts were used. The objective was to determine the degree of warpage as a function of solution temperature, quenching media, and machining sequence. Two machining cuts removed the fin; one left it on. The flatness was measured on the surfaces orthogonal to the z-axis. They were then averaged together to represent the overall warpage of each sample.

Aluminum Cold Rolling and Strip Processing: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
Program Organizer: Kai Karhausen, Hydro Aluminium

Monday PM — Room: 2010
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Kai Karhausen, Hydro Aluminium

2:00 PM Introductory Comments

2:05 PM Keynote
Cold Rolling Processes to Functionalize Semi-Finished Products: Gerhard Hirt[1]; *Koos van Putten*[1]; Reiner Kopp[1]; Mario Thome[1]; [1]RWTH Aachen

Sheets, strips and profiles are semi finished products, which are widely used for structural light weight components in transport systems, civil engineering and machine building. During the last years various rolling processes have been investigated and partly been introduced to industrial applications, which enable to manufacture geometrically tailored products: The so called “flexible rolling” process is used to roll sheets with product specific thickness changes in rolling direction to produce sheet metal parts with load optimized thickness distribution. Thickness changes in width direction of thin strips can be produced by strip profile rolling using modified roll forming equipment. Riblet surface structures similar to shark skin can be rolled directly into aluminium sheet using a new roll structuring technique. The actual status of these processes and their application is presented including consequences for further processing.

2:45 PM
Analytical Stress Field Modelling of Rolled Aluminium Strips under Tensile Loading: *Holger Aretz*[1]; Stefan Neumann[1]; Kai Karhausen[1]; [1]Hydro Aluminium Deutschland GmbH

During downstream processing aluminium strips are often subjected to tensile loading. In particular, during cold rolling upstream and downstream tensile loads are imposed by the de-coiler and the coiler, respectively. The knowledge of the resulting stress field is essential for the rolling and the downstream winding process. The present contribution consists of three major parts: (1) An analytical stress field model is developed resting on the construction of an admissible stress function according to Airy’s theory within the framework of plane stress linear elasticity. Application examples are provided. (2) Based on an elementary analysis the incorporation of off-flatness effects in form of residual plastic strains in the aforementioned stress field model is described. (3) A new approach based on elementary equilibrium conditions is presented that aims at calculating the across-width stress distribution in rolled strips possessing an arbitrary transversal thickness profile which are loaded by a constant remote tensile stress.

3:05 PM
Recrystallization Texture Development under Various Thermo-Mechanical Conditions in Aluminum Alloys: *Jurij Sidor*[1]; Alexis Miroux[1]; Roumen Petrov[2]; Leo Kestens[2]; [1]Materials Innovation Institute; [2]TU DELFT

The texture development during recrystallization annealing is affected by the thermo-mechanical history. A variety of hot and cold rolling parameters account for various recrystallization textures both qualitatively and quantitatively. Asymmetric rolling by a differential circumferential velocity of the top and bottom rolls is applied to the investigated aluminum alloy. The resulting shear deformation gives rise to a non-conventional texture evolution in the hot band. Introduction of intermediate annealing during cold rolling affects both volume fraction of the cube orientation as well as the total strength of the produced texture. The influence of both rolling parameters and initial textures on the development of the deformation and recrystallization textures is discussed based on experimental data and results of texture simulation with a wide variety of crystal plasticity models.

3:25 PM
Experimental Procedures for Characterization of Static Recovery in Cold Rolling Processes of AlFeSi Alloys: *Christoph Heering*[1]; Xiaoli Li[1]; Gerhard Hirt[1]; Markus Bambach[1]; [1]RWTH Aachen

In this paper, experiments regarding the influence of static recovery on the flow stress of AlFeSi alloys are presented. Double compression tests at different temperatures were carried out. From these tests, stress-time curves were generated that describe the static recovery. The stress time curves were used for an empirical recovery. Additionally, two series of cold rolling experiments with AlFeSi alloys were performed. One series of cold rolling tests was performed with a heat treatment at 230°C and one series of rolling experiments was performed under ideal cold rolling conditions. Subsequent to every process step the flow stress of the sheet was measured by tensile tests. Thus, the influence of static recovery on the flow stress in cold rolling processes can be compared to the flow stress development in ideal cold rolling. Finally, the rolling experiments were simulated using a physical flow stress model with an implemented empirical recovery model.

3:45 PM Question and Answer Period

3:55 PM Break

4:10 PM
Innovations in Surface Quality Inspection as a Cornerstone for Production Optimization: *Uwe Knaak*[1]; Elisa Jannasch[1]; [1]Isra Vision Parsytec Ag

Surface defects impair the quality of the manufactured aluminum strip; they may lead to strip breaks or to equipment damage: less ability to deliver usable quality to customers, less throughput, and higher costs are the consequences. Isra Vision Parsytec offers leading-edge solutions for surface quality inspection:

Surface Inspection Systems deliver defect information to be turned into quality data for a most efficient production optimization. The benefits for customers include highest detection sensitivity and accelerated access to relevant quality data combined with highest availability and easiest handling and maintainability of the systems. Furthermore, so-called "production decision intelligence" solutions transforming inspection data to production benefit in selected applications are available. Production optimization can be achieved by combining surface quality data with all available production and process data, as well as with customer and order information. Surface inspection thus serves two aims: increasing product quality, and turning surface quality information into production excellence.

4:30 PM
Modeling of Cold Surface Rolling Process of Al 2014 T6 Alloy, Residual Stress Calculation: *Behzad Majidi*[1]; [1]Amirkabir University of Technology

Cold surface rolling is a very important process which is performed on different parts to enhance surface quality and to generate near- surface residual compressive stresses. In the present investigation cold surface rolling of 2014 T6 aluminum alloy has been modeled by the mean of finite element method using ABAQUS/Explicit software. The effects of rolling load and speed and also number of rolling steps have been studied. Results showed that the most favorable compressive stress gradient in depth of the part corresponds to the load of 100Kgf in one step rolling. It was also found that increasing rotation speed of part during rolling has a positive effect on the residual compressive stress magnitude at surface.

4:50 PM
Effects of Annealing Process on Intermetallic Compound of Carbon Steel/Al Cladding Strip: *Guoyin Zu*[1]; Wei Wang[1]; Jiuming Yu[1]; [1]School of Materials and Metallurgy

The annealing process for carbon steel/Al cladding strip was investigated systematically, to discuss the effects of annealing temperature/time on the growth of intermetallic compound by OM, SEM, XRD measurements, the tensile strength and elongation of cladding strip was tested using electronic universal testing machine. The results show that the deformation energy during rolling process go against growth control of intermetallic compound. The critical temperature spot of intermetallic compound forming is 420°, the intermetallic compound is FeAl3 phase. The tensile strength and elongation of cladding strip tended to raise first and then declined with increasing annealing duration. Based on comprehensive consideration of growth condition of intermetallic compound and the mechanical properties of cladding strip, the optimum annealing process should be at 420° for 40min. In addition, short time high temperature and single-side annealing were beneficial to inhibit the growth of intermetallic compound.

5:10 PM Question and Answer Period

Aluminum Reduction Technology: Environment

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Gilles Dufour, Alcoa Canada, Primary Metals; Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Alcan; Jayson Tessier, Alcoa Deschambault

Monday PM — Room: 2001
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Nancy Holt, Hydro Aluminium

2:00 PM Introductory Comments and Presentation of 2008 Best Paper Award 2008 Light Metals Paper Awards Presentation: Aluminium Reduction

2:05 PM
Global Anode Effect Performance: 2010 PFC Emissions Reduction Objective Met: *Jerry Marks*[1]; [1]International Aluminium Institute

One of the first objectives set by the Directors of the International Aluminium Institute (IAI) as part of the global industry's Aluminium for Future Generations Sustainability Initiative was to reduce PFC emissions per metric ton aluminum produced by 80% from the 1990 baseline by 2010. To monitor progress toward this objective the IAI conducts an annual global industry survey of anode effect performance. The 2006 survey data showed that the 2010 objective has been achieved four years early. Now that the objective has been met the Directors are considering a new PFC emissions objective. This paper discusses the details of the analysis of the anode effect survey data, the progress made to date in PFC emissions reduction, and considers what the potential might be for future PFC emissions reductions.

2:25 PM
An Innovative Method for Sampling and Analysis of Tetrafluoromethane and Hexafluoroethane Emitted from Aluminium Smelter Using Sorbent Tubes: *Josette Ross*[1]; Véronique Bouchard[2]; Michel Gagnon[2]; Jean-Nicolas Maltais[1]; [1]Rio Tinto Alcan; [2]UQAC

Rio Tinto Alcan has aggressive objectives regarding the reduction of CF4 and C2F6 (two greenhouse gases) emitted during the anode effect. Consequently, the PFC concentrations decrease during the sampling measurement campaigns. Presently, the most frequently used method to evaluate the PFC emissions in an aluminium smelter is the Fourier Transform Infra Red analysis. It is a costly method with a probably detection limit not low enough for high performing plants. The Arvida Research and Development Centre has developed a new innovative method using a thermal desorption system coupled with a gas chromatograph and a mass spectrometer for the sampling and analysis of CF4 and C2F6. The sampling is easy and inexpensive to perform, the sample could easily be sent by post, and the analysis is fast and very sensitive. For a 24-hour sampling period, the detection limits were determined as being 120 and 260 pptv for CF4 and C2F6 respectively.

2:45 PM
Initiatives to Reduce Anode Effect Frequency at Dubal: Arvind Kumar[1]; *Ali Al Zaroni*[1]; Maryam Al Jallaf[1]; [1]Dubai Aluminium Co. Ltd.

Extensive studies have been carried out in smelters around the world to understand the fundamental cause of an anode effect. The exact nature of the onset of an anode effect is still shrouded in mystry. However, the consensus is that anode effects are detrimental to pot operation; they result in reduced energy consumption and cause emission of CF4 and C2F6 gases. With the intention of reducing carbon footprint, there is an excellent opportunity to reduce anode effects and the resulting PFC emissions. Occurrence of an anode effect was studied in relation to different aspects; alumina feed rate, work schedule, cathode type, operating parameters, mechanical issues, etc. Onset of an anode effect was primarily due to inability of the response strategy to deal with it efficiently. Alumina fines and pencilling of crust breaker tip were the other reasons. The paper covers strategies pursued at Dubal to reduce anode effect frequency.

3:05 PM
Handling Co2EQ from an Aluminum Electrolysis Cell: *Odd-Arne Lorentsen*[1]; Are Dyroy[1]; Morten Karlsen[1]; [1]Hydro Aluminium

The current focus on reduction of energy consumption and preserving our environment will affect a lot of industries in the coming years, also the aluminum industry. Hydro believes aluminum is a part of a sustainable future, and wants to take an active part in developing an even more environmentally friendly production process. Most of Hydro's electricity used for aluminum production is based on water power, but the plants in Kurri Kurri and Neuss are based on coal and our new smelter in Qatar will be based on gas. This paper gives an insight in Hydro's plans for reduction of their carbon footprint from their primary productions around the world by keeping their focus aiming for elimination of AE and production of CF-gases. Hydro also have developed a gas suction technology enabling CO2 capture from their electrolysis pots, as well as reduction of the net gas suction volume, with promising results.

3:25 PM Break

3:45 PM
Comparison of PFC Emission for Operating and Newly Started Pots at the Alcoa Fjardaal Point Fed Prebake Smelter: *Neal Dando*[1]; Weizong Xu[1]; Jerry Marks[2]; [1]Alcoa Inc; [2]J Marks and Assoc.

Under a jointly sponsored program by the USEPA and Alcoa, PFC monitoring campaigns were performed at Alcoa's Fjardaal smelter to 1) determine Tier 3 PFC emissions rates from previously started (months earlier) operating cells, 2) determine Tier 3 PFC emissions from recently started (days earlier) operating cells and 3) determine the PFC emissions from a population of newly started cells during the initial "bathup" period and subsequent operation. The measured PFC slope terms from these three pot populations indicate that no significant difference exists between initial startup and "normal" pot Tier 3 PFC emission

coefficients when the pots are well heated prior to bath up (gas-bake pre-heating). This data also suggests that anode effect data from the initial "bath-up" period can be included in the plant's PFC reporting inventory using the same emission coefficients determined during normal pot operation, assuming that "soft" (well pre-heated cathode) pot starts are performed.

4:05 PM

Dry Scrubbing for Modern Pre-Bake Cells: *Stephen Lindsay*[1]; Neal Dando[1]; [1]Alcoa Inc

The two fundamental "raw materials" for pot room gas treatment systems are alumina and the process off-gases. Modern dry scrubbing technology offers very efficient removal technology. However, increases in the amount of fluoride evolved from reduction cells and increases in fume evacuation rates can challenge the abilities of dry scrubbers. This is especially so if the goal is to provide; state of the art removal efficiency, and alumina at the pot that is low in fines content. In this paper the author discusses trends in our industry and proposes solutions that include more efficient utilization of alumina and process gas flow to meet emerging needs.

4:25 PM

Pot Gas Heat Recovery and Emission Control: Geir Wedde[1]; *Anders Sorhuus*[1]; [1]ALSTOM Norway AS

Substantial quantities of heat is released to the ambient through pot exhaust and present pot gas temperature of 150-180°C also affect the operation of the Gas Treatment Centres (GTC). Standard polyester filter bags used in the GTC can only sustain gas temperatures of 135°C. A sharp rise in fluoride emissions (HF)is seen as pot gas temperatures exceed 100°C. Dilution of the pot gas with ambient air is used to achieve acceptable GTC gas temperatures (110-115°C) and emission levels. This results in a need for substantial increase in the filtration capacity of the GTC. A heat exchanger has been developed to combine heat recovery and cost efficient cooling of pot gas. The technology has been tested on pot gas in a pilot plant. Promising stable heat exchange and pressure drop over longer test periods have encouraged Alstom to continue the development into a commercial product.

4:45 PM

Development of a Jet Induced Boosted Suction System to Reduce Fluoride Emissions: *Michel Meyer*[1]; Guillaume Girault[2]; Jean-Marc Bertolo[1]; [1]Rio Tinto Alcan; [2]Tomago Aluminium Company Ltd

For many aluminium smelters, reducing fluoride specific emissions is the sine qua non condition for production growth. Since they represent nearly 40% of the overall roof vent results, anode change operations are particularly targeted for improvements. A preliminary review of the available options concluded that significantly increasing the pot exhaust flow during anode change was the more promising technological option. An Alcan patented solution, the Jet Induced Boosted Suction System has been trialled successfully on AP22 pots in Tomago. The performance of the system, in terms of emission reduction, costs, operating and maintenance requirements was evaluated during the trial with the objective of extending the system to larger trial groups and AP30 pots.

5:05 PM

The Impact of Ambient Wind on the Vertical Component of Smelter Roofvent Flow Velocity: *Michael Gershenzon*[1]; Neal Dando[1]; [1]Alcoa Inc

This work details the correlation between ambient wind (direction and speed) and the vertical component of smelter rooftop air flow velocity. This study shows that when ambient winds are co-aligned with the long axis of a smelter building, the average vertical airflow velocity leaving the potroom roofline is reduced. This effect is especially pronounced when the building's side ventilators (basement panels) are shut during the colder months of the year. This study emphasizes the importance of examining prevailing wind patterns for assessing fugitive emissions from both existing and projected smelters, especially for geographic locations with distinct wind roses (e.g., marine regions).

Biological Materials Science: Biomimetic Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee

Program Organizers: Ryan Roeder, University of Notre Dame; John Nychka, University of Alberta; Paul Calvert, University of Massachusetts Dartmouth; Marc Meyers, University of California

Monday PM — Room: 3014
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Roger Narayan, University of North Carolina

2:00 PM Keynote

Chemical Tailoring of Biologically-Derived 3-D Nanostructured Inorganic Assemblies: *Kenneth Sandhage*[1]; Zhihao Bao[1]; Eric Ernst[1]; Sehoon Yoo[2]; Yunnan Fang[1]; Michael Weatherspoon[3]; Samuel Shian[1]; Ye Cai[1]; Qingzhong Wu[1]; Matthew Dickerson[4]; Emily Malcolm[1]; Nicole Poulsen[1]; Nils Kroger[1]; [1]Georgia Institute of Technology; [2]Korea Institute of Industrial Technology; [3]Harris Corporation; [4]Air Force Research Laboratory

The low-cost fabrication of chemically-tailored inorganic structures with selectable morphologies, controlled over the micro-to-nanometer scales and over three dimensions, remains a significant technological challenge. Hierarchical 3-D inorganic assembly is, however, accomplished under ambient conditions by diatoms (single-celled microalgae). Each of the tens of thousands of diatom species forms a nanostructured silica microshell (frustule) with a unique, genetically-determined 3-D morphology. Sustained diatom reproduction can yield enormous numbers of frustules of similar morphology. Such genetically-precise, massively-parallel, 3-D assembly is without analog in synthetic nanofabrication. However, the silica-based frustule chemistry severely limits the range of potential applications. With the patented* BaSIC (Bioclastic and Shape-preserving Inorganic Conversion) process, reaction-based and/or coating-based methods can be used to convert such bioclastic templates into non-natural functional chemistries, while preserving the 3-D hierarchical morphology. Recent work on conversion of diatom frustules into functional metallic and oxide chemistries will be described. U.S. Patents 7,204,971 (4/17/07) and 7,067,104 (6/27/06).

2:40 PM

Writing Fiber-Reinforced Gels for Soft Tissue Replacement: The Robospider: *Paul Calvert*[1]; Animesh Agrawal[1]; Tesfay Meressi[1]; Bharat Mahajan[1]; [1]University of Massachusetts Dartmouth

While large quantities of textile fiber can be readily melt-spun or solution-spun, there is no simple way to form small quantities of fiber from specialty polymers or biopolymer solution. A pultrusion system is being used to spin micron-diameter polymer fibers from solution in analogy to the spinning of a spider web. Extrusion of fibers from a moving syringe needle coupled to a sensitive force transducer was used to measure and model the process. This system is now being used to deposit fibers onto surfaces or into gels in order to build webs and fiber-reinforced hydrogels. This paper will discuss the constraints on using this approach to mimic soft tissues such as cartilage.

3:00 PM

Molecular Biomimetics - A New Paradigm in Functional Materialization: *Mehmet Saikaya*[1]; [1]University of Washington

Properties of engineered materials are structure sensitive and their synthesis, formation and organization take place with the control of heat that allows manipulation of atoms and molecules, providing the energy for compound formation. The TTT diagrams in Fe-C systems are ideal examples where transformation is controlled by temperature and time. In biological systems, such as hard tissues, all these processes, however, take place at ambient conditions in aqueous solutions, without the effect of heat flow. Functions of biological materials are also structure-sensitive, and are the results of evolution. In biological systems, synthesis, formation, and structuring are controlled by peptides and proteins that are evolved to bind to these solids and, in turn, manipulate their behavior. This presentation will highlight examples of materialization in biology and offer ways of how it may be possible to genetically engineer peptides for inorganics as molecular building blocks for next generation materials systems.

3:20 PM
Feasibility of Fabricating Root-Form Implants by Electron Beam Melting: *Marie Koike*[1]; Gilbert Chahine[2]; Radovan Kovacevic[2]; Toru Okabe[1]; [1]Baylor College of Dentistry; [2]Southern Methodist University

Currently, endosteal dental implants are made of titanium alloys which consist of the root-form fixture and transmucosal abutment. Traditional implants are placed in the jawbone after a socket hole is drilled to accept endosteal implants. With the advent of electron beam melting (EBM), we developed the method for micro-CT scanning of teeth in need of extraction and rapid prototyping of one-component biomimetic implants that fit within the existing root socket. These implants are expected to decrease the risk of implant failure at the root-form fixture/transmucosal abutment interface and improve osseointegration to encourage bone in-growth, leading to shorter healing time. Using EBM equipment (ARCAM A2, Sweden), molar teeth of Ti-6Al-4V (ELI) were fabricated from computerized X-ray tomography images of extracted maxillary teeth. The shape and dimension of the EBM teeth were similar to the extracted teeth. In the presentation, the mechanical properties of EBM-fabricated titanium will be reported.

3:40 PM Break

3:50 PM Student Poster Contest Short Oral Talks chaired by Ryan K Roeder, University of Notre Dame

Bulk Metallic Glasses VI: Alloy Development and Glass Forming Ability II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter Liaw, The University of Tennessee; Hahn Choo, The University of Tennessee; Yanfei Gao, The University of Tennessee; Gongyao Wang, University of Tennessee

Monday PM Room: 3007
February 16, 2009 Location: Moscone West Convention Center

Session Chairs: Akihisa Inoue, Tohoku University; Marios Demetriou, California Institute of Technology

2:00 PM Invited
Metallic Glassy Nanowire: *Koji Nakayama*[1]; Y. Yokoyama[1]; G. Xie[1]; Q.S. Zhang[1]; M. W. Chen[1]; T. Sakurai[1]; A. Inoue[1]; [1]Tohoku University

Metallic glassy nanowires were spontaneously created on the fracture surfaces that were produced by a conventional mechanical test. The presence of the nanowires is directly related to the one-dimensional meniscus configuration with a small viscosity at high temperatures and to the wide supercooled liquid region of the metallic glass. The electron microscopic observations demonstrate the diameters, the lengths, and the amorphous structural states, and the energy dispersive X-ray reveals the chemical components. In addition, we found that round ridges are constructed from nanotubes. The finding of amorphous nanostructures provides not only fundamental understanding of fracture processes but also give a new insight into nano-engineering constructions.

2:15 PM
The Role of Friction in Measurements of the Formability of Bulk Metallic Glasses: *Sven Bossuyt*[1]; Jan Schroers[2]; [1]Vrije Universiteit Brussel; [2]Yale University

Recently, a simple experiment was proposed to characterise the formability of bulk metallic glasses. It measures the total deformation of a specimen that occurred, under constant load, while heating from below the glass transition, to above the crystallisation temperature. A priori, it is not clear whether the higher deformation rate achievable with more fragile glass-formers or the larger time-temperature processing window before crystallization of stronger glass-formers is most beneficial for formability of bulk metallic glasses reheated above their glass transition temperature. The proposed measurement takes these contradictory effects into account, and directly measures this formability. We demonstrate the role of friction in these experiments, using analytical results for limiting cases and mixed numerical experimental methods for finite friction coefficients.

2:25 PM Invited
Preparation and Characterizations of Bulk Metallic Glasses Using Spark Plasma Sintering: *Jinn Chu*[1]; Ying Chen Tai[1]; Robert Aalund[2]; Tom C. Clappier[2]; Matt W. Mede[2]; Shian-Ching Jang[3]; [1]National Taiwan University of Science and Technology; [2]Thermal Technology, LLC; [3]I-Shou Univ

Due to many unique properties, bulk metallic glasses (BMG's) have recently generated enormous interest. Yet, some BMG's in large sizes are not readily obtained because their glass-forming ability (GFA) is not high enough to use the conventional casting technique to prepare BMG's. Spark plasma Sintering (SPS) has been reported to sinter nano-sized or amorphous powder materials into bulk forms with no or minimal crystallization and grain growth through a short and effective sintering process. In addition, SPS'ed samples with nearly 100% theoretical density and properties close to those of bulk parts make SPS an attracted technique for BMG's with low GFA. In this presentation, many BMG systems prepared using SPS are characterized by various analytical techniques including differential scanning calorimeter, X-ray diffractometer, scanning and transmission electron microscopes. This study is directed toward to establishing better understanding of SPS-prepared BMG properties, thus utilizing SPS for the large-sized BMG's with desirable properties.

2:40 PM
High Glass Formability for Cu-Hf-Ti Alloys with Small Additions of Y and Si: *Ignacio Figueroa*[1]; Hywel Davies[1]; Iain Todd[1]; [1]University of Sheffield

The effect of small substitutions of Si and Y on the glass forming ability (GFA) of the Cu55Hf25Ti20 glassy alloy is reported and discussed. Fully glassy rods with diameters up to 7 mm and 6.5 mm, were produced for Cu54.5Hf25Ti20Si0.5 and Cu55-xHf25Ti20Y0.3 alloys, respectively. The addition of Si enlarged the Tx considerably from 30 to 53 K for the Cu54Hf25Ti20Si1 alloy. The results showed that the parameters obtained from thermal analysis, such as Trg and ΔTx are not reliably correlated with GFA, at least for these bulk glass forming alloys. The scavenging effect of the Y and Si, in particular the possibility of Y reducing the oxides, could be responsible for enhancing the GFA. The effectiveness of small additions of Si on the GFA was considered that might be enhanced by the large negative heat of mixing and the possible formation of HfSiO4 as a strong network former.

2:50 PM
New Processing Potential for Bulk Metallic Glass Matrix Composites with Tensile Ductility: *Douglas Hofmann*[1]; Jin-Yoo Suh[1]; Aaron Wiest[1]; William Johnson[1]; [1]California Institute of Technology

Recently, metallic glass matrix composites have been demonstrated with high toughness and extensive tensile ductility. A composite with ~60% crystalline phase is demonstrated to have bending ductility in large dimensions and crack arrest in large thickness samples. For the composite, plastic forming above the glass transition temperature is demonstrated, room temperature cold rolling to strains over 100% is achieved with high reduction rates per rolling pass, and tension tests are performed on previously rolled samples. We note that yield strength and stiffness increase as a result of rolling but tensile ductility is preserved. The tests demonstrate that the new toughened glassy composites combine the best properties from metallic glasses (high strength, high elastic limit, plastic forming ability, low melting point, net shape forming ability, high hardness, etc.) with the toughest of crystalline metals. The alloys therefore represent a new paradigm in metallurgy

3:00 PM
Influence of Bond Enthalpy on Metallic Glass Stability: *James Dahlman*[1]; Daniel Miracle[1]; [1]Materials and Manufacturing Directorate

Metallic glasses form structures with a high degree of short range order, indicating an underlying thermodynamic criterion, as interatomic bond strength largely governs nearest neighbor interaction. While earlier empirical guidelines suggest that the enthalpy of mixing exerts a strong influence on glass stability, previous work has failed to show a correlation. This work seeks to establish a connection between nearest neighbor bond energy and glass-forming ability through evaluation of interatomic bond enthalpies. An approach to determine bond enthalpies from available thermodynamic data will be described and resulting bond enthalpies will be presented. The number and type of atom bonds that are present in a metallic glass structure are estimated as a function of metallic glass constitution using the efficient cluster packing structural model. By combining these two analyses, we estimate the enthalpy associated with glass formation, and explore correlations with experimental measurements of glass-forming ability.

3:10 PM Break

3:20 PM Invited
Nanoglasses Synthesized by Extreme Plastic Deformation of BMG: *Hans Fecht*[1]; Yulia Ivanisenko[2]; [1]Ulm University; [2]Forschungszentrum Karlsruhe

We present recent results on the fundamentals of extreme plastic deformation of a range of fully dense metallic glasses. By high pressure torsion it becomes possible to change the free volume of an amorphous material considerably. This method can be used to (i) fully densify a collection of metallic glass nanoparticles or (ii) tune the atomic structure of a metallic glass by the formation of a high density of primary and secondary shear bands. On this basis, the changes in structural, thermodynamic and mechanical properties will be discussed.

3:35 PM
Strong Effects of Alloying Elements on the Structure, Dynamics and Glass Forming Ability of Metallic Supercooled Liquids: *Yongqiang Cheng*[1]; Evan Ma[1]; [1]Johns Hopkins University

The addition of a relatively small amount of alloying element(s) can induce major changes in the viscosity, fragility and glass forming ability of supercooled liquids. A microscopic understanding of this behavior from the structural perspective has been elusive. Through comparisons between Cu-Zr-Al and Cu-Zr supercooled liquids, here we demonstrate the strong effects of Al alloying on the atomic-scale structure, in particular the evolution of icosahedral local motifs, as well as the resulting dramatic slowing down of relaxation dynamics. The composition-structure-dynamics relationship uncovered for realistic bulk metallic glass forming liquids is important for understanding the subsequent glass transition and their high glass forming ability.

3:45 PM
The Prediction of Glass-Forming Compositions in Metallic Systems: *Kevin Laws*[1]; [1]University of New South Wales

A new methodology of predicting specific compositions for glass forming ability based on elemental cluster selection, atomic packing efficiency, ab initio calculations and liquidus lines will be presented and discussed. The proposed composition selection model has lead to the discovery of a number of soon to be reported transition metal-based bulk metallic glasses, some with critical casting thicknesses in excess of 7 mm and high thermal stability. The proposed model may also be used to explain high glass forming ability of known BMG compositions and to pin-point new or superior BMG compositions in existing glass forming systems. Further, the aforementioned model shows strong correlations between proposed elemental clusters, glass forming ability and BMG density, mechanical strength and ductility. This model has also shown applicable adaptation to known ceramic oxide glass forming systems.

3:55 PM
Periodic Amorphous Metallic Cellular Structures: *Joseph Schramm*[1]; Marios Demetriou[1]; William Johnson[1]; [1]California Institute of Technology

The high yield strength and ability to deform plastically at sub-millimeter sizes make amorphous metal an attractive material for strong metallic cellular structures. Additionally, softening at the glass transition allows amorphous metals to be "thermoplastically" formed. Recent work has shown that stochastic amorphous metallic cellular structures (amorphous metallic foams) deform plastically under compression at plateau stresses that correlate consistently with the yield strength of the monolithic amorphous metal. Periodic cellular structures (e.g. honeycombs) are able to inherit a significantly larger fraction of the monolithic yield strength than foams, making them substantially stronger at a given relative density. In this presentation, recent progress on honeycomb-type amorphous metallic structures will be presented. Structures with porosites in the range of 75-90% have been assembled from thermoplastsically-formed corrugated sheets. Compression tests on single cores of the periodic structure reveal their ability to deform in the same manner as foams while maintaining higher plateau stress.

4:05 PM Invited
Formation and Electrochemical Behavior of Mechanically Alloyed Cu-Zr-Ti-Ta Bulk Metallic Glass Composites: *Pee-Yew Lee*[1]; Chien-Yie Tsay[2]; Chin-Yi Chen[2]; Hong-Ming Lin[3]; [1]National Taiwan Ocean University; [2]Feng-Chia University; [3]Tatung University

The preparation of (Cu60Zr30Ti10)90Ta10 BMG composites through a powder metallurgy route was investigated. The metallic glass composite powders were found to exhibit a super-cooled liquid region before crystallization. (Cu60Zr30Ti10)90Ta10 BMG composites were synthesized by vacuum hot pressing the as-milled (Cu60Zr30Ti10)90Ta10 composite powders It was observed that pressure enhanced the thermal stability and suppressed the formation of nanocrystalline phases in (Cu60Zr30Ti10)90Ta10 BMG composites. The corrosion behavior of (Cu60Zr30Ti10)90Ta10 BMG composites in four different corrosive media was studied using the potentiodynamic method. The resultant polarization curves indicated lower corrosion rates, and current densities were obtained for composites measured in 1N H2SO4, NaOH, and HNO3 solutions. The XPS results revealed that the formation of Zr-, Ta-, and Ti-rich passive oxide layers provided a high corrosion resistance in 1N H2SO4 and HNO3 solutions, while the breakdown of the protective film by Cl- attack was responsible for pitting corrosion in the 3 wt% NaCl solution.

4:20 PM
Characterization of Complex Geometry Amorphous Metal Structures Created by Micromolding: *Gerald Bourne*[1]; Jeffrey Bardt[1]; Tony Schmitz[1]; Daniel Zeenberg[1]; Nickolas Ptschelinzew[1]; W Sawyer[1]; Michael Kaufman[1]; [1]University of Florida

Casting and molding are attractive options for low cost mass production. However crystallization during cooling induces shrinkage that can be on the order of several percent, resulting in poor tolerances. Additionally, molds must be fabricated from materials capable of withstanding the high temperatures associated with melting of metals. To enable molding of micro-scale devices, metallic glasses offer the potential to avoid many of the problems associated with molding of metals. The amorphous structure in metallic glasses leads to properties that may include high yield strength, hardness, strength-to-weight ratio, elastic limit, and wear resistance. In this study, we have produced complex geometries using a multilayer sacrificial Si wafer micromolding process from $Zr_{41.2}Ti_{13.8}C$ $u_{12.5}Ni_{10}Be_{22.5}$. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are used to characterize the net shape forming ability, surface quality, and microstructure of the molded products. Results are presented.

4:30 PM Invited
Cu-Hf-Al Bulk Metallic Glasses: Compositional Dependence of Glass-Forming Ability and Compressive Plasticity: Peng Jia[1]; *Jian Xu*[1]; [1]Institute of Metal Research, Chinese Academy of Sciences

In this talk, we will report recent progress regarding the compositional dependence of glass-forming ability (GFA) and compressive properties for Cu-Hf-Al ternary bulk metallic glasses (BMGs). Firstly, the Cu-Hf-Al BMG-forming composition region is identified to correlate with the (L-$Cu_{10}Hf_7$+$CuHf_2$+CuHfAl) eutectic reaction. The fragility parameter D^* of the $Cu_{55}Hf_{45}$ binary and $Cu_{49}Hf_{42}Al_9$ (C1) ternary supercooled liquid was determined from relaxation time measurements, indicating that Al incorporation also leads to a "stronger" liquid. Secondly, Weibull statistics was used to study the distribution of compressive yield strength (σ_y) of the $Cu_{49}Hf_{42}Al_9$ and $Cu_{45}Hf_{46}Al_9$ (C2) BMGs. The σ_y of both BMGs exhibits high uniformity. The C2 BMG with a higher Weibull modulus (m=40) is less brittle than C1 (m=53). Comparison of the elastic constants and fragility between C1 and C2 indicates that the less brittle C2 shows a slightly higher Poisson's ratio and fragile feature with respect to the C1.

Cast Shop for Aluminum Production: Engineering and Industrial Developments

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Pierre Le Brun, Alcan CRV; Hussain Alali, Aluminium Bahrain

Monday PM — Room: 2005
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Ravi Tilak, Almex USA Inc

2:00 PM Introductory Comments by Pierre Le Brun

2:05 PM TMS 2008 - Best Paper Award 2008 Best Paper Award Presentation

2:10 PM
Hindalco Almex Aerospace Limited – A New Greenfield Aerospace Alloy Casthouse: *Shaun Hamer*[1]; Lorraine Fortier[1]; [1]Almex USA Inc

Hindalco Almex Aerospace Limted, an Indian incorporated company started a new Greenfield casthouse in the third quarter of 2008. The casthouse is designed

to supply the aerospace and specialist alloy downstream operations with a billet supply option for 2000 and 7000 series alloys in billet sizes ranging from 7" to 41½ ". The concept for the casthouse, a joint venture between Hindalco and Almex USA has developed out of the understanding of the aerospace downstream industries and identifying the niche markets where billet supply can boost the industry's global growth. This paper describes the implementation of this casthouse from concept through to start-up and the growth steps for the coming years to realize the plant's maximum operating capacity. It further investigates the production philosophies and design criteria incorporated to ensure success of this new venture and world class metal supply to the extrusion, forging, sheet and plate industries.

2:30 PM

Qatalum Cast House: *Andrew Home*[1]; A. Troppeano[2]; [1]K Home International Ltd; [2]FATA EPC

The new smelter being built by joint venture partners Quatalum Petroleum and Hydro will set a new standard for the size of green field smelter projects. An important part of the project is the cast house producing 625 thousand tonnes per annum of value added product, making it the largest smelter cast house to be built in a single phase. This paper will discuss the concept for the project, a novel approach to the contract methodology and report the progress of the project to date.

2:50 PM

CVG Venalum- Design of a 55 t Tilting Melting Furnace: *Santiago Barry*[1]; Fidias Rodriguez[1]; Orlando Gil[1]; [1]CVG Venalum

In this paper, the engineering design of a 55 t tilting melting furnace is described. This includes all the simulations that apply for the efficient design of this furnace, that is: Metallic structure; Refractory body; Tilting system; Foundations system; Combustion system; Control and Power system. The aim of this new tilting furnace design is to replace the current static furnaces which are coupled to a vertical DC casting machine at CVG Venalum; with an outlook of increasing the production levels, the quality of the products, as well as improving the security, ergonomic and environmental conditions of the workplace. This engineering has been developed by CVG Venalum Research and Development Center.

3:10 PM

Advanced Control of a Rotary Drum Furnace in a Secondary Smelter: *Detlef Maiwald*[1]; [1]Innovatherm GmbH

In the secondary smelter industry rotary drum furnaces are used for the remelting of various types of aluminium scrap, especially UCB's and coated scrap. The control of the rotary drum furnace is difficult due to the following circumstances: - temperature of rotary drum refractory or the aluminium bath can't be measured, only the exhaust gas temperature is measurable; - production cycle is interrupted by several repeating phases of charging, decoating and melting; - operation of this equipment is still on a high degree of manual impact. A new technology determine the process parameters indirectly by Fuzzy Logics. Additionally a new measurement is introduced, using the actual electrical current consumed by the rotary drum main motor during the entire melting cycle. The target is the optimization of the melting cycle and minimization of primary fuel usage. Also peak temperatures should be avoided to extend lifetime of refractory.

3:30 PM

Fluid Modeling of the Flow and Free Surface Parameters in the Metaullics LOTUSS System: *Mark Bright*[1]; Florin Illinca[2]; Jean-Francois Hetu[2]; Frank Ajersch[3]; Charbel Saliba[1]; Chris Vild[1]; [1]Pyrotek Inc.; [2]National Research Council of Canada; [3]Fabmatek Services Inc.

The growth of aluminum product consumption has placed an emphasis on improving the efficiency of processing internally generated scrap. In the Metaullics LOTUSS (LOw TUrbulence Scrap Submergence) System, aluminum machining chips can be melted at a rate in excess of 15 tons per hour with very high recovery efficiencies. A computational fluid dynamics (CFD) model has been implemented to optimize the LOTUSS System to further enhance efficiency and maximize melting performance. Preliminary studies of the CFD modeling will be presented outlining the three-dimensional numerical algorithm for solving the turbulent and free-surface flow inside the LOTUSS system. CFD simulations were carried out for melting system conditions and verified against previous experimental studies. The results indicate that the free surface CFD model is an accurate representation of real-world conditions and the predictions for the position and size of the vortex cone compare very well with the measured experimental values.

3:50 PM Break

4:10 PM

Electromagnetic Stirring in Aluminum Ladles: *Robert Stål*[1]; Patrick Hanley[1]; [1]ABB

Electromagnetic stirring (AL-EMS) in aluminum furnaces is now a well-established technology to enhance chemical and thermal homogeneity and to reduce cycle time, energy consumption and dross formation. This paper will discuss the benefits of using AL-EMS for ladle stirring of liquid aluminum. To become more cost efficient some foundries are purchasing liquid aluminum. The molten aluminum is stored in ladles which can be put on hold for as long as 24 hours before being delivered to the foundry. Heating and stirring will be needed to assure correct temperature during this waiting time. Experiences from industrial plant trials have shown that electromagnetic stirring can generate total thermal homogenization in aluminum ladles. Reduction of melt surface temperature by AL-EMS and how this can suppress surface oxidation and improve heat transfer to the melt is discussed in this paper. This further demonstrates the potential of Al-EMS to reduce burner runnihng cost.

4:30 PM

Optifine - A Grain Refiner with Maximized Nucleation Efficiency: *Rein Vainik*[1]; John Courtenay[2]; [1]Opticast Aluminium AB; [2]MQP Ltd

A new grain refiner with a strong nucleation efficiency, Optifine, is presented. By optimizing the growth restriction, i.e. mainly by adding low amounts of titanium, extremely low additions of this gives the same grain size as normal additions of standard grain refiners. The efficiency is explained by a narrow range of boride particle sizes, which allows simultaneous nucleation on a large number of aluminium crystals. Furthermore, the growth restriction determined by the alloy composition and/or aided by minute additions of titanium, will allow a substantial proportion of these crystals to grow resulting in a very fine grain structure. Apart from cost savings due to much reduced application rates, a high efficiency master alloy will have a large impact on the billet and ingot quality, since only a very small amount of the hard boride particles are needed in order to produce a cast grain size resistant to ingot cracking.

4:50 PM

New Grain Refiner Containing Ternary Carbide Nucleant Particles: Marta Suarez[1]; Mauro Martin[1]; *Abinash Banerji*[2]; [1]Aleastur; [2]Microalloy

A new grain refiner, AlTiX (patent pending), has been invented for aluminum and its alloys. It is essentially an aluminum based quaternary master alloy containing ternary carbide nucleant particles. Both short and long time TP1 tests were carried out on commercially pure aluminum (99.7%) to find out the grain refining efficacy of the new refiner under different test parameters and the results have been compared with those obtained with commercially available conventional AlTiB and AlTiC master alloys respectively under similar test conditions. The paper deals with a maiden report on the newly invented grain refiner containing ternary carbide particles which are believed to nucleate aluminum grains during solidification of the inoculated melt.

5:10 PM

New SiC-Graphite Castable for Molten-Metal Transfer Units: *Claude Allaire*[1]; [1]CIR Laboratory Inc

In aluminum molten-metal transfer units, silicon carbide containing refractories should be used where maximum thermomechanical abuse resistance is required. These materials are most often used either in the form of castables or as preformed carbon or clay-bonded shapes using techniques such as ribforming, rollerforming and isopressing. Where applicable, the later materials are advantageous since they may contain graphite that contributes to increase their thermal shock and corrosion resistance due to its higher thermal conductivity and chemical inertia, respectively. To beneficiate of the graphite properties as well as the simple and low expensive forming method available with none shaped refractories, a new silicon carbide ultra-low cement castable containing up to 15 vol. % graphite has been developed. The formulation principles and the properties of this new SiCGraphite castable are presented in this paper.

Characterization of Minerals, Metals and Materials: Characterization of Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee, TMS/ASM: Composite Materials Committee

Program Organizers: Toru Okabe, University of Tokyo; Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF

Monday PM — Room: 3009
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Tzong Chen, CANMET-MMSL; Kazuki Morita, University of Tokyo

2:00 PM

Characterization of Manganese Oxide Scales on Rolled Lead Anodes from a Commercial Zinc Electrowinning Operation: *Tzong Chen*[1]; John Dutrizac[1]; [1]CANMET-MMSL

Rolled Pb-0.7% Ag anodes are porous. The manganese oxide scales, consisting mainly of MnO2, characteristically occur in a banded colloform structure which adheres to an irregular layer of PbSO4 and PbO2 that oxidized from the Pb anode. Hydrous Mn oxides, Mn3O4 and amorphous Mn oxides are believed to be also present. Tiny particles of gypsum, PbSO4, SrSO4 and AgCl are entrapped in the colloform mass. Silver originated from the anodes; strontium carbonate was added to the Zn solution to control Pb, and gypsum originated from the Zn electrolyte. Some colloform bands contain minor amounts of Pb, implying minor dissolution of Pb during electrolysis. Other colloform bands incorporate trace amounts of Zn and Ca. Colloform structures permeated with silica gel were presumably caused by the high silica content of the electrolyte.

2:15 PM

Titanium Production/Coating Process by Disproportionation of Titanium Dichloride in Molten Magnesium Chloride: *Taiji Oi*[1]; Toru Okabe[1]; [1]Institute of Industrial Science, the University of Tokyo, c/o Okabe Laboratory (Fw-301)

In order to establish a new titanium production/coating process, the synthesis and disproportionation of $TiCl_2$ in molten $MgCl_2$ were investigated. $TiCl_2$ was synthesized by reacting $TiCl_4$ with titanium metal in $MgCl_2$ molten salt at 1200-1273 K and titanium metal was produced by the disproportionation of $TiCl_2$ in molten $MgCl_2$ at 1273 K. The results revealed that $TiCl_2$ was successfully obtained in the synthesis experiment and that the efficiency of $TiCl_2$ formation was drastically improved by using molten $MgCl_2$ as a reaction medium. In the titanium production experiment, titanium powder of over 99% purity was produced by disproportionation of $TiCl_2$ in molten $MgCl_2$. From these results, it was shown that the methods investigated in this study can be applied to a new titanium production process. In addition, the feasibility of a new method for titanium coating utilizing dispropotionation of $TiCl_2$ is discussed from some results of preliminary experiments.

2:30 PM

Fundamental Study on Recovery of Nd and Dy from Rare Earth Magnet Scrap Using Molten Salt: *Sakae Shirayama*[1]; Toru Okabe[1]; [1]Institute of Industrial Science, the University of Tokyo, c/o Okabe Laboratory (Fw-301)

In order to develop a new process for the recovery of neodymium (Nd) and dysprosium (Dy) from rare earth magnet scrap, selective extraction of Nd and Dy was investigated by using metal halides. For preliminary experiments, magnesium chloride ($MgCl_2$) was selected as an extracting agent, and molten $MgCl_2$ was reacted with Dy-containing Nd–Fe–B magnet alloys. Experimental results revealed that the rare earth elements in the magnet alloys were successfully extracted into $MgCl_2$ in high yields. After the removal of $MgCl_2$ by vacuum distillation, Nd and Dy could be separately recovered by a wet or dry process. The effectiveness of $MgCl_2$ and other metal halides as extracting agents and the feasibility of effective recycling of Nd–Fe–B magnet scrap are discussed in this paper.

2:45 PM

Characterization of CO_2 Laser-Assisted Deposition of Diamond Thin Films by Combustion-Flame Method: *Travis McKindra*[1]; Matthew O'Keefe[1]; [1]Missouri University of Science and Technology

The effect of the CO_2 laser irradiation and the combustion gas composition on the microstructure of diamond thin films was investigated. A continuous wave CO_2 laser operated at 600 W was used to irradiate the flame tip during $C_2H_2/C_2H_4/O_2$ and C_3H_6/O_2 combustion-flame deposition. The film morphology, chemical bonding, and crystal structure were characterized by scanning electron microscopy (SEM), x-ray photoelectron spectroscopy (XPS), x-ray diffraction (XRD) and Raman spectroscopy. The films were continuous with faceted diamond grains 1-2 μm in size. The CO_2 laser irradiation increased the diamond (111) diffraction peak intensity. These results were in agreement with the film morphology results from the SEM as the film deposited with the laser had a larger grain size which resulted in a sharper, more intense peak. The films deposited with the C_3H_6/O_2 combustion-flame contained W_xO_y, $Co_xW_yO_z$ and Co_xW_yC phases. The XPS results confirmed that the films had a significant amount of diamond.

3:00 PM

Production of V and V-Ti Alloys from Oxide Preforms: *Akihiko Miyauchi*[1]; Toru Okabe[1]; [1]Institute of Industrial Science, the University of Tokyo, c/o Okabe laboratory (Fw-301)

In order to develop a new process for effective production of vanadium metal (V) and V-Ti alloys from V_2O_5, a fundamental study was conducted on a preform reduction process (PRP) based on metallothermic reduction. Feed preforms with good mechanical strength even at elevated temperatures were prepared by adding either CaO or MgO to V_2O_5 feed powder; thus, complex oxides ($Ca_xV_yO_z$, $Mg_xV_yO_z$) were obtained. Reduction experiments were conducted by using either Ca or Mg vapor at 1273 K for 6 h. Vanadium metal with a purity of 99.7% was successfully obtained when Mg was used as a reductant. The feasibility of producing V or V-Ti alloys by the PRP will be discussed on the basis of fundamental experiments.

3:15 PM

Characterization of β-$FeSi_2$ Film Synthesized by Exchange Reaction between Si and Molten Salts: *Motohiro Sakamoto*[1]; Kazuki Morita[1]; [1]University of Tokyo

β-$FeSi_2$ is a candidate material for Si-based optical and photovoltaic devices. In order to fabricate β-$FeSi_2$ film on Si wafer, various kinds of processes have been developed. Since ultra-high vacuum atmosphere is essential for these processes, vacuum-free process is required for mass production of photovoltaic cell at low cost. In this study, β-$FeSi_2$ film was prepared on Si wafer by the cation exchange reaction between Si wafer itself and molten NaCl-KCl-$FeCl_2$ salts. Two phases of FeSi and β-$FeSi_2$ were formed by the reaction between single crystal (100) wafer and 0.1mol% $FeCl_2$ molten salts at 1173K for 1h in He atmosphere. After the sample was annealed at 1173K for 24h, the flat β-$FeSi_2$ layer was confirmed to be formed on Si wafer by X-ray diffraction and scanning electron microscopy. The band gap of this β-$FeSi_2$ was determined to be 0.85eV by infrared spectroscopy, showing good agreement with other reported values.

3:30 PM Break

3:50 PM

FeCr2O4 Spinel Formation: Relationship between Color and Magnetics Properties: *Oscar Restrepo*[1]; Juan Montoya[1]; Ernesto Baena Murillo[1]; [1]Univ Nacional De Colombia

The compounds type spinels are oxides whose ideal formula is AB2O4, where A is a divalent cation and B is a trivalent cation. They have been the subject of scientific interest because their properties allow its use in different applications. They are used as pigments; they are usually obtained by sinterization of various oxides, where cations comply with certain characteristics of atomic ratio and oxidation number. This paper shows the results obtained in the study using iron and chromium oxides in fixed proportions, used as precursors of the compound with spinel type structure FeCr2O4. For different temperatures synthesis, a characterization for the obtained material was performed using X-ray diffraction (XRD), UV-VIS-NIR spectroscopy and Mössbauer spectroscopy. The changes in the reflectance spectra are related with the identified phases, crystallinity degree, spatial ordering of cations and magnetic behavior.

4:05 PM
Synthesis and Production of Ni-Mo Alloys for Hydrogen Production via Mechanical Alloying: *Maria Valero Rocha*[1]; Roberto Martinez Sanchez[1]; Jose Cruz Rivera[2]; Israel Rodriguez Torres[2]; [1]Centro de Investigacion en Materiales Avanzados, S.C.; [2]Instituto de Metalurgia

The present work studied the structural and microstructural evolution of Ni-Mo alloys (10%, 20% and 30%Wt) produced by Mechanical Alloying. Alloys production was performed in a commercial Fritsch planetary mill, the ball-to-powder weight ratio was 6:1, and the process control agent used was hexanos. Nominal compositions of original powders were 99.99% purity, the particle sizes was 2.23 µm and 67.93 µm, respectively. Mixtures of powders of Ni-10%Mo, Ni-20%Mo and Ni-30%Mo were milled by different times. After Milling, powders were cold compact. The sintering compacts were carried out in a resistance tubular furnace. Structural and morphological characterization of the powders and the sintering materials were performance by XRD, SEM, TEM and EDS. From characterization results it is possible to observe an increment in the parameter which suggests that during the process a solid solution and nanometric phase were formed. The results has been analyzing in cathodes for hydrogen production.

4:20 PM
Shape-Controlled Synthesis of Porous Fibrous Cobalt Powders: *Zhan Jing*[1]; Dong Chengyong[1]; Zhang Chuanfu[1]; Wu Jianhui[1]; Fan Youqi[1]; [1]Central South University

The fibrous precursor can be obtained by coordination precipitation process. The composition and morphology of fibrous precursor were characterized by XRD, IR, DTA/TGA and SEM analysis. The results show that XRD pattern and composition of the precursor with fibrous morphology precipitated at pH=9.0 are different from that of ß-CoC2O4•2H2O precipitated at pH=1.0. The mechanism on the thermal decomposition of fibrous precursor was addressed The influences of various conditions in pyrolysis, including the temperature, time, atmosphere, and the morphology of the precursor, on the morphology, average size and specific surface area of the Co powders were investigated in detailed. At last, the final product-fibrous cobalt powders with about 0.3~0.5µm in size and 40~60 in aspect ratio were produced by thermal decomposition at 400~500° in the weak reducing atmosphere. The structure of pores in cobalt powders is capillary tube with open ports and the majority is mesoporous.

4:35 PM
Synthesis of Nanometer Core-shelled Titanium Dioxide/Tungsten Oxide Powder: *Daoxin Wu*[1]; [1]Changsha University of Science and Technology

With the controllability in composition,structure and property,the complex core-shelled nanoparticles have attracted both domestic and international interests in recent years. Originated from $(C_4\ H_9O)_4Ti$,nano-rutile TiO_2 was synthesized by low temperature hydrolytic process in this paper.After being prepared by decomposing ammonium tungstate and covering on the surface of TiO_2, TiO_2/WO_3 were characterized by means of thermogravimetric and differential scanning calorimeter (TG-DSC), X-ray diffratction (XRD), UV-vis diffuse reflectance (DRS), fluorescence spectrum (FS). Results showed that with the increase of the supported concentration of WO_3 the reflectance of DRS and the fluorescence spectrum intensity of TiO_2/WO_3 decreased accordingly which indicated the powder's increase in light absorbance and the decrease in luminescence respectively.

4:50 PM
Vibration Damping of High-Chromium Ferromagnetic Steel: *Satish Bhujang Mutt*[1]; Mahesh Kumbeshwara[1]; Girish Bhujang Mutt[1]; [1]East Point College of Engineering and Technology

The present work aims to study the effect of annealing on the vibration damping capacity of high-chromium (16%) ferromagnetic steel. The alloys were prepared from raw materials of 99.9% purity melted in a high frequency induction furnace under vacuum. The samples were heat-treated in vacuum temperatures (800 to 1200ºC) for 1 hour followed by slow cooling (120ºC/h). The inverted torsional pendulum method was used to evaluate the vibration damping capacity. A water-based magneto-fluid was used to analyze magnetic domain morphology of the alloy using optical microscopy. The results indicated that the vibration damping capacity of the alloys is influenced by annealing and there exists a critical annealing temperature after 1000ºC. The damping capacity increases quickly below the critical temperature since the magnetic domains move more easily. Above the critical temperature the damping capacity decreases due to the larger size of the magnetic domains leading to decrease in domain wall area.

5:05 PM
Corrosion Mechanism of A3 Steel Induced by Chloride Ions in the Purified Water: Liyuan Chai[1]; Haijuan Xiao[1]; *Yunyan Wang*[1]; Yude Shu[1]; Fei Pei[1]; Jinlong Zhang[1]; [1]Central South University

In the nonferrous metallurgical industry it is of great significance to solve the problem of resource waste and environment pollution due to the discharge of heavy metal-containing wastewater. However, there arises a question whether the water impurities with chloride ions and fluoride ions will result in the corrosion of the pipeline during the whole recycling process. In this study, the corrosion mechanism of A3 steel induced by chloride ions in the purified water had been investigated with the A C Impedance technique. The results showed that there were another two factors which determined its electrochemical corrosive rate: the electrode potential and coverage ratio of chloride ions on the surface of A3 Steel. The corrosion mechanism of A3 steel in the solution with chloride ions emendation was two steps of electrode process with the rate-determining step of FeCl Fe2+ +Cl- +e-.

Computational Thermodynamics and Kinetics: Thin Films

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, ASM Materials Science Critical Technology Sector, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Long Qing Chen, Pennsylvania State University; Yunzhi Wang, Ohio State University; Pascal Bellon, University of Illinois at Urbana-Champaign; Yongmei Jin, Texas A&M

Monday PM — Room: 3002
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Pascal Bellon, University of Illinois

2:00 PM Introductory Comments

2:05 PM Invited
Stress-Driven Surface Evolution during Whisker and Hillock Formation: *W. J. Boettinger*[1]; T. Frolov[2]; V. A. Ivanov[2]; Y. Mishin[2]; [1]National Institute of Standards and Technology; [2]George Mason University

In stressed solids, surface evolution is often driven by grain boundary diffusion and can result in growth of hillocks and whiskers. Examples are whisker growth in compressively stressed Sn deposits on Cu and hillock formation in Cu conductor lines during electromigration. The mechanisms of hillock and whisker growth remain largely unknown. We present molecular dynamics simulations aimed at understanding the conditions (stress, temperature, grain boundary diffusion, surface diffusion) at which the hillock/whisker growth processes can be initiated. The simulated geometries include a single boundary normal to the surface and a tri-crystal with a wedge-shape surface grain, both under an applied stress parallel to the surface. We have also studied extrusion of materials through a nano-hole simulating a crack in an oxide layer covering a stressed film. The early stage of hillock/whisker growth is observed at high homologous temperatures when the boundary diffusion flux exceeds the lateral fluxes of surface diffusion.

2:35 PM Invited
Effects of Substrate Symmetry and Prepatterning on the Stability of Compositional Patterns in Ultrathin Alloy Films: Bo Yang[1]; Tejodher Mupidi[1]; Vidvuds Ozolins[1]; *Mark Asta*[1]; [1]University of California

First-principles-based computer simulations are employed to elucidate the effects of substrate symmetry and externally applied prepatterned "potentials" on directing self-assembly of ordered nanoscale compositional patterns in ultrathin films. This work focuses on alloy films as a specific example where the energetics underlying composition modulation can be accurately quantified within the framework of a hybrid model that incorporates an atomistic calculation of interatomic bonding with continuum theories of long-range substrate-mediated elastic interactions. Employing Monte-Carlo simulations based on this hybrid model for alloy energetics, we demonstrate that even relatively weak external potentials, with periodicities considerably larger than the intrinsic composition-modulation wavelengths, can be highly effective in stabilizing ordered compositional patterns at the nanoscale.

3:05 PM

Compositional Domain Formation in Ultrathin Films: A Phase-Field Crystal Study: *Srevatsan Muralidharan*[1]; Mikko Haataja[1]; [1]Princeton Univ

It is well-known that materials confined in one or more dimensions may display properties which are strikingly different from those of their bulk counterparts. An illustrative example of this phenomenon is provided by a binary alloy, which is immiscible in the bulk and yet forms miscible phases when deposited on a surface as a (sub)monolayer aggregate. In this case, the mixing of the components is brought upon by the epitaxial nature of the growth processes. In addition to alloying, surface dislocations provide a mechanism for strain relaxation. In this talk we describe a phase-field crystal (PFC) model we have recently developed to study this technologically relevant process. The PFC model incorporates alloy thermodynamics, the presence and motion of free surfaces and/or grain boundaries, the presence of long-ranged elastic strains, and the nucleation and dynamics of dislocations, thus providing a physically-based picture of the domain formation kinetics at the nanoscale.

3:25 PM

Coarsening of 3D Thin Films under the Influence of Strong Surface Anisotropy, Elastic Stresses: *Peng Zhou*[1]; Steven Wise[2]; John Lowengrub[1]; [1]University of California Irvine; [2]University of Tennessee Knoxville

We develop a diffuse interface model to investigate the three dimensional coarsening in thin films. In this model, both strong surface anisotropy with Willmore regularization, elastic stresses and deposition are included. The governing equation for the phase field parameter is a sixth order Cahn-Hilliard Equation due to the presence of surface anisotropy and the Willmore regularization. The simulated system is assumed to be in mechanical equilibrium with misfit in the film generated by lattice mismatch in the substrate. Thus, the Cauchy-Navier equations are solved for elastic displacements which lead to the elastic energy. Both the Cahn-Hilliard equation and the Cauchy-Navier equations are solved with an non-stiff, adaptive nonlinear multigrid method. Simulation results of coarsening in three dimensions with different strengths of the surface anisotropy, misfit strain, and deposition rates will be shown. Comparison and analyses of these results will help to explain their influence on coarsening processes in thin films.

3:45 PM Break

4:10 PM

Sintering and Microstructure Evolution in Columnar Thermal Barrier Coatings: *Ramanathan Krishnamurthy*[1]; David Srolovitz[2]; [1]Caterpillar Inc; [2]Yeshiva University

Sintering of thermal barrier coatings changes their key properties, thus adversely impacting their reliability. We present a hierarchical modeling approach to study sintering-induced evolution of topcoat microstructure, wherein the sintering of individual topcoat column pairs is modeled using a thermodynamic principle, and column center-to-center approach rates calculated thence are incorporated into a discrete dynamics model of the temporal evolution of hundreds of columns. Surface, grain boundary and strain energy effects are naturally included in this framework. Varied late-time microstructures, with small clusters and random in-plane porosity, or with 50-100 columns-wide clusters separated by elongated inter-cluster channels,are observed, corresponding to small/large extents of contact among 'feathery' protrusions from columns. Statistical measures extracted from predicted microstructures reveal that cluster formation is strongly favored for large column densities and extents of the 'feathery' protrusions from columns. We compare predicted microstructures with recent experimental observations and discuss their import for thermal barrier coating processing/reliability.

4:30 PM

Characterizing Adsorption on Metallic Surfaces: Effect of Composition: *Baskar Ganapathysubramanian*[1]; Nicholas Zabaras[2]; [1]Iowa State University; [2]Cornell University

The enhancement of adsorption of (hydrogen) molecules on metallic surfaces is a key challenge for producing feasible fuel storage technologies. The chemistry of the surface under consideration plays an essential part in the adsorption phenomena. A reliable computational framework requires very accurate first-principle calculations of the energy of the system. We utilize the recently developed weighted multi-body expansion to accurately represent the energy of a cluster of atoms. An adaptive sparse grid collocation strategy provides the ability to effectively tessellate high dimensional surfaces. This allows us to naturally incorporate higher order interactions (up to 5 body terms). We utilize this strategy to construct multibody potentials representing the interaction of hydrogen with various transition metals. We investigate the effect of composition variation on the absorption coefficient.

4:50 PM

Thermodynamics of Nanoscale Binary Systems: *Muralidharan Ramachandran*[1]; Ramana Reddy[1]; [1]The University of Alabama

Nanoscale materials have been considered for and been in use in a variety of industrial engineering applications. It is shown that the melting temperature decreases and the phase diagram of binary systems change with the decrease in particle size. Non-ideal or real solution characteristics were introduced into the binary system using the activity data obtained from the literature. The phase diagrams of selected nanoscale binary systems were constructed considering the non-ideality of the system, the surface effects and the variation in the particle size and shape. The availability of data on surface and interfacial tensions has limited the number of systems considered. The calculated results were compared with that of the experimental results from the literature.

5:10 PM

Simulation of Thickness Effect on Grain Growth in Thin Films and Experimental Verification: Zhinan An[1]; *Yonghua Rong*[1]; [1]Shanghai Jiao Tong University

Various stagnation effects of grain growth in nano materials have been found, but thickness effect has not yet been researched. This paper presents an anisotropic Monte Carlo (MC) algorism to stimulate grain growth in thin films in annealing process. The simulation results reveal that thickness effect begins to work only when the average grain size reaches 0.8 to 1.2 times of the thickness of the film, not in the whole process of grain growth. Experimental data of grain growth in pure Co films with different thicknesses confirm the simulation results. Based on the stimulation and experiments, a modified grain growth kinetic equation is suggested to better describe the whole process of grain growth in nano-films.

5:30 PM

Effect of Partical Failure on the Yield Strength of SiCp/Al Matrix Composites: *Dai-hong Xiao*[1]; [1]Central South University

The SiC particles stress in aluminum matrix composites was examined according to the Eshelby's equivalent inclusion approach. A model was established to examine the influence of SiC particles failure on the yield strength of SiCp/Al composites after assumption that the SiC particles failure follows Weibull statistics. The values of tensile strength of SiCp/Al composites predicted by the model are well agreed with the experimental values. Moreover, the interface debond is the main failure way when the particle diameter is small in yielding condition, the percentage of particle fracture was found to increases with the increase of particles volume fraction and play the more important role in particle failure.

Dislocations: 75 Years of Deformation Mechanisms: Dislocation Ensembles and Structures

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: David Bahr, Washington State University; Erica Lilleodden, GKSS Research Center; Judy Schneider, Mississippi State University; Neville Moody, Sandia National Laboratories

Monday PM — Room: 3022
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: David Bahr, Washington State University; Lyle Levine, National Institute of Standards and Technology

2:00 PM Invited
Measured Elastic Strains within Dislocation Cell Structures: Local Behavior and Statistical Distributions: *Lyle Levine*[1]; Ben Larson[2]; Jon Tischler[2]; Peter Geantil[3]; Francesca Tavazza[1]; Mike Kassner[3]; Wenjun Liu[4]; [1]National Institute of Standards and Technology; [2]Oak Ridge National Laboratory; [3]University of Southern California; [4]Argonne National Laboratory

The existence and magnitude of long range elastic strains (and thus stresses) in dislocation cell interiors and walls in deformed metals have been the subject of extensive investigation for more than 20 years. We have used depth-resolved, submicrometer X-ray beams to directly measure the axial elastic strains within numerous *individual* deeply buried dislocation cell interiors and cell walls in plastically deformed copper single crystals. As previously reported, the average cell interior strains are tensile in unloaded compression specimens and compressive in unloaded tensile specimens. Recent measurements from individual, buried cell walls show that these have the reverse average strains. All of these cell interior and cell wall strains exhibit large cell-to-cell variations with magnitudes up to 50% of the flow stress. The experimentally determined distribution functions describing these strain fluctuations will be presented along with new theoretical models that explain their origin.

2:30 PM
Determination of Geometrically Necessary Dislocation Distributions Using Electron Backscatter Diffraction: *Angus Wilkinson*[1]; [1]University of Oxford

Cross-correlation based analysis of EBSD patterns has been shown to give significant improvements in angular resolution so that strains and lattice rotations can be measured at $\pm 10^{-4}$. This sensitivity coupled with the high spatial resolution makes the technique very attractive. Measurement of lattice curvature allows the geometrically necessary dislocation (GND) content to be assessed using Nye's dislocation tensor analysis (Nye 1953). The technique has been applied to various systems including: crack tip deformation during tensile loading in BCC metals (W and V), fatigue crack tips in Ni-based superalloy, flow fields around nanoindents in Fe, Cu and Ti, low strain deformation of HSLA steel, martensite induced GNDs in dual phase steel, and tilt and twist mosaics in GaN layers grown on sapphire. The presentation will use some of these applications to illustrate the technique.

2:50 PM
Resolving the Geometrically Necessary Dislocation Content by Conventional Electron Backscattering Diffraction: *Wolfgang Pantleon*[1]; [1]Risoe DTU

From local orientation measurements on planar surfaces by means of electron backscattering diffraction, six components of the lattice curvature tensor can be identified. They allow determination of five components of the dislocation density tensor (thus two more than hitherto reported) and, additionally, one difference between two other components. When determining the geometrically necessary dislocation content, all available information should be utilized, i.e. all six independent components of the curvature tensor and not only the three or five components of the dislocation density tensor. With the increased number of available components, more accurate, increased lower bounds for the total dislocation density are obtained by linear optimization. The method is illustrated on deformed metals and rocks.

3:10 PM
An Experimental Investigation of the Plastic Strain Evolution in Commercial Purity Ti Deformed in Bending: *Yiyi Yang*[1]; Leyun Wang[1]; Thomas Bieler[1]; Gene Ice[2]; Wenjun Liu[3]; Philip Eisenlohr[4]; Martin Crimp[1]; [1]Michigan State University; [2]Oak Ridge National Laboratory; [3]Argonne National Laboratory; [4]Max-Planck-Institut für Eisenforschung GmbH

Slip system interaction with grain boundaries leading to heterogeneous deformation and damage nucleation has been studied in commercial purity Ti. In-situ SEM four-point bending was performed to develop a tensile stress state on the observable surface. Orientation-imaging microscopy (OIM) was used to characterize the orientation distributions in the microstructures and to identify deformation twinning during plastic deformation, while electron channeling contrast imaging (ECCI) was used to identify activated slip and twinning systems. Grain boundary inclinations and orientation gradients within particular grains were examined using synchrotron 3-D X-ray analysis and revealed large strain gradients developed near some grain boundaries. From these observations, conditions that facilitate or prevent slip transfer across a grain boundary are identified. This work was supported by National Science Foundation (NSF) grant DMR-0710570 and German Science Foundation (DFG) grant EL 681/2-1. Use of the Advanced Photon Source was supported by the US DOE-BES Contract No. W-31-109-Eng-38.

3:30 PM
Contrasts in Hot Worked Al-Alloy Substructures from EOM, POM, XRD, TEM, STEM, SEM-EBSI and OIM: *Hugh McQueen*[1]; [1]Concordia University

Substructure characteristics in Al alloys are important for mechanical modeling in hot forming and its products. In contrast to simple grain shape in etched-optical microscopy (EOM), polarized optical microscopy (POM) significantly confirmed subgrain presence in better detail than x-ray diffraction (XRD). Transmission electron microscopy (TEM) revealed the dislocations forming subgrain boundaries (SGB) and dispersed between them; scanning mode (STEM) provided microtextures substantiating XRD. Scanning electron microscopy with back-scattered image (SEM-EBSI) exhibited substructures more accurately than POM but much less detailed than TEM. Orientation imaging microscopy (OIM) provided microstructures as in SEM-EBSI along with detailed misorientations; however, omission of very-low angle SGB seen in TEM estimated larger subgrain sizes and misorientations. The field of view is very limited in TEM, but fairly similar in POM, SEM-EBSI and OIM with higher magnifications possible in the last two. They are affected differently by substructure scale, solute and particle distributions (partly through specimen preparation).

3:50 PM Break

4:10 PM Invited
Void Growth by Dislocation Loop Emission: *Marc Meyers*[1]; David Benson[1]; Sirirat Traiviratana[1]; Eduardo Bringa[1]; [1]UC San Diego

Analytical calculations and molecular dynamics simulations show that the initiation of void formation, thought to occur by the convergent vacancy migration, takes place by the emission of shear loops from the void surface. The configurations of these loops are analysed and it is shown that reactions take place, leading to biplanar and triplanar configurations. These configurations are dependent on the tensile direction. The shear loop mechanism operates for voids as small as 0.3 nm radius, containing 13 vacancies. The density of geometrically necessary dislocations is calculated from the expansion of shear loops and is found to be consistent with observed values. The MD calculations are applied to polycrystals and it is shown that voids nucleate at grain boundaries, particularly at triple points.

4:40 PM
Temporal Statistics and Coarse Graining of Dislocation Ensembles: *Jie Deng*[1]; Mamdouh Mohamed[1]; Anter El-Azab[1]; [1]Florida State University

The theoretical modeling and numerical simulation of temporal statistics of dislocation ensembles is presented. A kinetic-equation hierarchy is established to describe the evolution of dislocation density, in which the source terms are governed by the rates of dislocation cross slip, annihilation and junction reactions. The stochastic point process and time series theories are used to model the spatial and temporal dependence of these processes and to model the source terms in the kinetic equation. The statistical properties of these processes, in both time and frequency domain, are analyzed in conjunction with dislocation

dynamics simulations. The moving average is applied to remove the trend and keep all the processes stationary. The numerical simulation of autocorrelation function and spectrum provides the preferred frequencies of different types of processes, which, together with their dependence of dislocation density, provide the better understanding of the temporal nature of those processes.

5:00 PM
Dislocation Structure and Slip Activity of PSBs in Cyclically Deformed Polycrystalline Nickel: *Anja Weidner*[1]; Werner Skrotzki[1]; [1]TU Dresden

Cyclic deformation of metals leads to strain concentrations in so-called persistent slip bands (PSBs) which produce a characteristic extrusion/intrusion profile at the surface. The dislocation structure of these bands can be described as a ladder-like structure of dislocation dense walls and dislocation poor channels embedded in a vein-matrix structure. Beside transmission electron microscopy (TEM), electron channelling contrast (ECC) in a FEG scanning electron microscope is a powerful method to image such dislocation structures. The resolution of ECC images is comparable to that of TEM micrographs. As the ECC method is non-destructive, it is possible to investigate the development of dislocation structures during different stages of fatigue life on the same sample. Moreover, the focused ion beam technology allows visualizing the dislocation structure of individual grains in 3D. An overview on the dislocation structure and the slip activity of PSBs in cyclically deformed polycrystalline nickel will be given.

5:20 PM
Experimental and Microstructurally-Based Finite-Element Investigation of the Dynamic Compressive Behavior of High Strength Alloys: K. Elkhodary[1]; W. Lee[1]; Bryan Cheeseman[2]; *Mohammed Zikry*[1]; [1]North Carolina State University; [2]Army Research Laboratory

The objective of this study is to identify the dominant microstructural and dislocation mechanisms related to the high strength and ductile behavior of 2139-Al, and how high strain-rate loading conditions would affect the overall behavior. Characterization techniques and specialized microstructurally-based finite-element (FE) analyses based on a dislocation-density based multiple-slip formulation that accounts for an explicit crystallographic and morphological representation of O and θ' precipitates and their rational orientation relations was conducted. The predictions from the microstructural finite element model indicated that the precipitates continue to harden, and also act as physical barriers that impede the matrix from forming large connected zones of intense plastic strain. As the microstructural FE predictions have indicated, and consistent with the experimental observations, the combined effects of θ' and O, acting on different crystallographic orientations, enhance the strength, the ductility, and reduce the susceptibility of 2139-Al to shear strain localization due to dynamic compressive loads

5:40 PM
Modelling Inhomogeneous Deformation Using a Dislocation Density Based Crystal Plasticity Finite Element Model: *Alankar Alankar*[1]; David Field[1]; Ioannis Mastorakos[1]; [1]Washington State Univ

A dislocation density based crystal plasticity finite element model (CPFEM) has been developed in which different dislocation densities evolve. Based upon the kinematics of crystal deformation and dislocation interaction laws, dislocation generation, annihilation and flux have been modeled. Stress evolution and inhomogeneous deformation based on dislocation density evolution have been predicted. Texture evolution in plane strain deformation of polycrystalline aluminum was used to validate the crystal plasticity modeling. The framework has been implemented in ABAQUS with user interface UMAT subroutine. Dislocation strength interaction, dislocation segment length interaction, dislocation-solute interaction and dislocation velocity laws have been used as studied using dislocation dynamics (DD) simulations.

Electrode Technology for Aluminum Production: Environmental Issues and Raw Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Barry Sadler, Net Carbon Consulting Pty Ltd; John Johnson, RUSAL Engineering and Technological Center LLC

Monday PM — Room: 2003
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Frank Cannova, BP Coke

2:00 PM Introductory Comments 2:05 PM Presentation of 2008 Best Paper Award by Barry Sadler

2:10 PM
Carbon Products: A Major Concern to Aluminum Smelters: *Ulrich Mannweiler*[1]; Werner Fischer[2]; Raymond Perruchoud[2]; [1]Mannweiler Consulting; [2]R&D Carbon Ltd.

Since decades the world wide primary aluminum production growths continuously with a rate of five percent per year. From thirty eight million tons aluminum today over sixty eight million tons are expected to be produced in 2020. With the aluminum growth the requirement for carbon products – petroleum coke, coal tar pitch, anodes and cathodes - will grow simultaneously. For each carbon product an outlook is given regarding availability, quality, price and production facilities. China's role as important supplier and consumer for coke, pitch, anodes and cathodes will be reviewed. The impact of production technologies and product properties on the emission of green house gases will be quantified for all process steps from raw materials to aluminum production.

2:35 PM
Anode Reactivity: Effect of Coke Calcination Level: *Marie-Josée Chollier*[1]; Alexandre Gagnon[1]; Claude Boulanger[1]; Dany Lepage[1]; Gaby Savard[1]; Ghislain Bouchard[1]; Charles Lagacé[2]; André Charette[2]; [1]Rio Tinto Alcan; [2]Université du Québec à Chicoutimi

Coke properties and anode performance are affected by the coke calcination level. Predictions of anode performance have traditionally been based on evaluation of coke reactivity. Using these methods, the use of a higher calcination temperature, up to the point where desulphurisation occurs, is preferred. Under-calcined cokes have, however, been shown to be beneficial to reduce anode consumption in industry. Changes in coke quality, such as increased sulphur, may affect the optimum calcination level for an individual coke. Improved laboratory techniques are required to define the industrial calcination level that will minimise anode reactivity. In this work, different methods for coke calcination, bench scale anode baking and anode reactivity testing have been compared. A procedure for the evaluation of anodes, which reproduces industrial results, has been developed. The study has also confirmed that, for the coke used, calcination at a lower level will reduce anode reactivity.

3:00 PM
Evaluation of the Necessary Amount of Quinoline Insolubles in Binder Pitch: John Baron[1]; *Stacey McKinney*[1]; Robert Wombles[1]; [1]Koppers Industries Inc

The role of quinoline insolubles in a binder pitch has long been the topic of discussion and disagreement. Throughout the world, especially in Asia, QI levels in crude coke oven tar are decreasing. What was once thought to be the lowest acceptable amount of QI in a binder pitch will have to be adjusted. This study was conducted to determine the effect of the quinoline insoluble content of the binder pitch on the physical properties of aluminum anodes. This study used coal tar pitches having QI levels of 1 wt.%, 2 wt.%, 4 wt.% and 6 wt.%. This paper will present the results of this laboratory anode study by comparing the physical properties of the resulting anodes.

3:25 PM Break

3:35 PM

Environmental and Operating Benefits of a New Fume Treatment System at a Paste Mixing Plant: *Matthias Hagen*[1]; Ralf Forster[2]; [1]LTB; [2]SGL Carbon GmbH

Increasing energy prices and high labour costs made a plant manager think of possibilities to save costs. A green production plant should be optimised in order to reduce production costs, increase the output and fulfil newest emission regulations. The solution was a completely new designed system. Heated suction hoods at the mixers and a ductwork system with preheating avoided condensations. The effect was zero cleaning, which saves time and increases the availability of the whole plant. A central fume treatment was installed with a thermal oxidiser as main part. The resulting energy is used for preheating of the fumes and the production of heat for the process, which reduces the energy consumption dramatically. The paper shows the technical solution and the numbers of savings in detail.

4:00 PM

From the "Low Caustic Leaching and Liming" Process Development to the Jonquiere Spent Potlining Treatment Pilot Plant Start-up, 5 Years of Process up-Scaling, Engineering and Commissioning: *Ghislain Hamel*[1]; Raymond Breault[1]; Gaston Charest[1]; Stéphane Poirier[1]; Bruno Boutin[1]; [1]Rio Tinto Alcan

The LCL&L process is a hydrometallurgical route developed by researchers of Rio Tinto Alcan (RTA) to treat, both environmentally and economically, spent potlining (SPL) generated by aluminium cells. Considering local characteristics, including the low capacity of cement producers to recycle SPL, there is a high SPL inventory that has accumulated over the last 25 years. LCL&L was selected as the preferred solution for the treatment of RTA's SPL in Quebec, and as a sustainable solution for other Quebec aluminium producers. This paper describes LCL&L process characteristics, including valorization alternatives for the by-products and special chemical analysis methods developed for process control. Some technological challenges faced and managed during scale-up from laboratory and mini-pilot process development, design and construction of the industrial scale pilot plant will be discussed. Finally, preparations for the plant commissioning and start-up in April 2008 as well as some early operational highlights will be discussed.

Emerging Applications of Neutron Scattering in Materials Science and Engineering: Residual Stress Mapping and Neutron Imaging

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Xun-li Wang, Oak Ridge National Laboratory; Brent Fultz, California Institute of Technology; Hahn Choo, University of Tennessee

Monday PM Room: 3012
February 16, 2009 Location: Moscone West Convention Center

Session Chairs: Werner Wagner, Paul Scherrer Institute; Philip Withers, Manchester University

2:00 PM Invited

Engineering the Residual State with Mechanical Surface Treatments: *Philip Withers*[1]; Suzanne Clitheroe[1]; Mark Turski[2]; Christopher Rodopoulos[3]; [1]The University of Manchester; [2]Magnesium Elektron; [3]University of Patras

Emerging mechanical treatments, such as laser peening, low plasticity burnishing and ultrasonic impact treatment can introduce residual stresses to a depth of many millimetres. In this paper we will report a study of laser peening and ultrasonic impact treatment as compared to performances of shot peened and unpeened benchmarks. In particular the capacity to overwrite previous residual stresses, as might be introduced by bending or weld stresses, will be assessed. In addition the stability of the resulting residual stresses to thermal and mechanical tests will be established, as well as the effect of the residual stresses on fatigue performance. Results for both stainless steels and titanium alloys will be reported being important for the nuclear and aerospace industries respectively. This work paves the way for the intelligent engineering of the residual stress state in terms of depth and intensity for optimum performance for a given set of in-service conditions.

2:30 PM

Neutron Residual Stress Mapping of Spur Gears under Applied Load: Robert LeMaster[1]; Jeffrey Bunn[2]; Brian Boggs[1]; Jon Kolwyck[1]; William Bailey[3]; *Camden Hubbard*[3]; [1]University of Tennessee-Martin; [2]University of Tennessee-Knoxville; [3]Oak Ridge National Laboratory

Stresses in operating gears arise from the externally induced stresses associated with the transmission of power and from the residual stresses associated with the heat treatment and machining of the tooth profiles. Residual stresses at the surface are typically measured ex situ using x-ray diffraction. Neutrons can non-destructively measure stress within a component and can pass through associated hardware. This paper presents the special capabilities of neutron strain mapping to map stresses in situ. Total stress was measured as a function of externally applied load and at depths below the meshing gear tooth surface. Changes in d-spacing between stressed and unstressed states allow determination of strains as a function of location and load. The measurements were made using the second generation Neutron Residual Stress Mapping Facility (NRSF2) at HFIR. A Static Load Application Device (SLAD) was developed to load the gear pair while mounted on the NRSF2 instrument.

2:50 PM Invited

In-Situ Neutron Diffraction Study of Materials Behavior under Severe Thermal-Mechanical Deformation: Wan Chuck Woo[1]; *Zhili Feng*[1]; Xun-li Wang[1]; Bjorn Clausen[2]; Donald Brown[2]; Thomas Sisneros[2]; Camden Hubbard[1]; Stan David[1]; [1]Oak Ridge National Laboratory; [2]Los Alamos National Lab

The materials behavior under rapid and severe thermo-mechanical deformation is one of the most important yet least understood research areas. We have successfully demonstrated a new measurement method for the direct observation and determination of the material behaviors as they evolve rapidly under complex thermo-mechanical material synthesis environment. For this purpose, a special portable friction-stir processing system was installed inside the beam room of the Spectrometer for MAterials Research at Temperature and Stress (SMARTS) at Los Alamos Neutron Science Center and the in-situ neutron-diffraction experiments were performed during the thermo-mechanical processing of 6.5-mm thick 6061-T6 Al alloy plate. Significant improvement of the temporal resolution of neutron scattering measurements has been achieved using the quasi-steady-state phenomenon. The new measurement methodology and data analysis approach enable us to determine the transient and dynamic variations of temperature, thermal stresses, dislocation density, and subgrain size during severe thermo-mechanical process of materials.

3:20 PM

Analysis of Residual Stress inside Complex Engineering Components Using Neutron Diffraction: *Supriyo Ganguly*[1]; Jon James[1]; Michael Fitzpatrick[1]; [1]The Open University

Residual stress measurement using neutron diffraction is an important tool for structural engineers. A new generation of dedicated engineering strain instruments have been built, offering considerable improvements in counting time and spatial resolution. Alongside these improvements, measurements in complex geometry prototype components are increasingly in demand. Therefore, there is a strong driver towards an integrated sample positioning systems for simplified set-up and operating of experiments on such prototype components. The present study was carried out at the ENGIN-X instrument at the ISIS pulsed neutron source, on measurements in a prototype metal matrix composite aircraft wheel, forged from a billet produced through powder metallurgy route. The measurement was designed to obtain macro and misfit stresses developed in matrix and reinforcement phase during fabrication. The use of the SScanSS software for experimental design and implementation, and for precise spatial location of the measuring gauge volume inside such complex components was also demonstrated.

3:40 PM Break

4:00 PM
Macroscopic Stress Relaxation in Complex High Performance Alloys: *Julia Repper*[1]; Michael Hofmann[1]; Christian Krempaszky[2]; Ewald Werner[3]; Winfried Petry[1]; [1]Forschungsneutronenquelle FRM II; [2]Christian-Doppler-Laboratory of Material Mechanics of High Performance Alloys; [3]Institute for Materials Science and Mechanics of Materials

Because of its excellent thermo-mechanical properties, the multiphase nickel based superalloy IN718 is widely used in industrial components. Macroscopic and microscopic residual stresses are induced during the production process of such components. While the effects leading to macroscopic stresses are well understood, the microscopic mechanisms of stress accumulation are less known. Neutron diffraction is a powerful technique to determine induced macro stresses by a comparison of diffraction angles of strained samples and unstrained reference samples. The identity of the micro stress state in component and reference sample is the basic assumption for macro stress analysis using neutron diffraction. For high performance alloys, like IN718, changes in the micro stress state are conceivable during macroscopic stress relaxation while cutting out reference samples. In this contribution we present the changes in phase specific strains determined by neutron diffraction resulting from a stepwise relaxation of macroscopic residual stresses measured on an IN718 pancake.

4:20 PM
Comparison of Intergranular Residual Strains in Hollow Cylinder Steel Specimens Subjected to Torsion and Tension: *Jeffery Bunn*[1]; Dayakar Penumadu[1]; Camden Hubbard[2]; [1]University of Tennessee; [2]UT-Battelle/ORNL

Torsion provides a unique opportunity to probe mechanical behavior of materials under pure shear stress. Two hollow cylinder 12L14 steel specimens had been subjected to two levels of torsion exceeding yield, and two other specimens were subject to two levels of tension using a combined axial-torsional testing system. Pairs of torsion-tension samples were subjected to the same magnitude of equivalent octahedral shear strain. In this study the samples were characterized with 0.5 x 0.5 mm spatial resolution. Residual strains for the Fe (110), (200) and (211) reflections were recorded for the hoop, radial and axial directions as a function of location through the hollow cylinder wall. Intergranular residual stresses for both torsion samples and for both tension samples were similar. However, major differences exist between the torsion set and the tension set. The largest differences are in the Fe (200) while the smallest differences was in the Fe (211).

4:40 PM Invited
Neutron Imaging – A Promising Tool in Material Science and Technology: *Werner Wagner*[1]; Eberhard Lehmann[1]; [1]Paul Scherrer Institut

At the Swiss Spallation Neutron Source SINQ, two facilities for neutron imaging are operating: NEUTRA, a radiography station at a thermal beam port, and ICON at a beam port viewing the cold moderator. Both facilities make use of different state-of-the-art imaging devices, based on imaging plates, semi-conductor arrays or CCD-cameras, and specifically tailored for various types of applications. Among those are: radiography and tomography with particularly high spatial resolution (down to the 10-µm range), real-time imaging up to 30 frames per sec, stroboscopic imaging with µs resolution, phase contrast imaging and energy selective imaging. One further option is imaging of highly radioactive samples for which NEUTRA is specifically equipped. The presentation will highlight the potential of these options for various applications in materials science and technology by means of selected examples.

5:10 PM
A New Option for Material Characterisation by Means of Energy Selective Neutron Imaging: Eberhard Lehman[1]; Gabriel Frei[1]; *Axel Steuwer*[2]; Winfried Kockelmann[3]; [1]SINQ PSI; [2]ESS Scandinavia; [3]STFC

In recent years neutron imaging techniques have developed significantly driven by digital imaging capabilities which enables a more efficient use of the applied neutrons and by the exploitation of scattering effects. Sophisticated techniques such as tomography and phase contrast imaging have now became available on a routine basis. A new approach based on energy selective neutron imaging was recently developed using different experimental set-ups. This option is particularly important for cold neutrons, where common engineering materials have Bragg edges due to the micro-structural behaviour of the crystals, which provides enhanced contrast.Using two different approaches, a double-crystal mono-chromatizers and the time-of-flight option at a pulsed spallation source, scattering artefacts were identified in transmission mode of imaging for welds and otherwise treated structural materials. In this manuscript, we present and discuss first results with a view to dedicated and optimized installations are foreseen at the upcoming new spallation sources.

5:30 PM
Neutron Transmission Strain Tomography: *Shu Yan Zhang*[1]; Ed Oliver[2]; Alexander Korsunsky[3]; [1]Science and Technology Facilities Council ; [2]Science and Technology Facilities Council; [3]University of Oxford

In many respects, strain mapping by neutron and synchrotron X-ray diffraction can be regarded as imaging techniques in 2D or 3D, i.e. the spatially resolved determination of a material property within the interior of an object. The aim of the study here is to present the concept of strain tomography using Bragg edge neutron transmission measurements. The principle of this novel approach is to analyze residual strain fields by de-convolution of unknown distributions of residual elastic strains from redundant sets of data collected from gauge volumes representing sections through the region of interest. Four representative samples were studied. They have demonstrated spatial resolution and shown the ability to discriminate strains in multiple phases. The strains present within the samples were successfully resolved and were showed very good agreement with the known strain field within the samples.

Energy Conservation in Metals Extraction and Materials Processing II: Energy Conservation and Technology

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Energy Committee
Program Organizers: Edgar Vidal, Brush Wellman, Inc.; Cynthia Belt, Aleris International Inc; Marie Kistler, Air Products and Chemicals, Inc; Mark Cooksey, CSIRO; Rob Hardin, Burner Dynamics, Inc.

Monday PM — Room: 2012
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Cynthia Belt, Aleris International Inc; Marie Kistler, Air Products and Chemicals, Inc

2:00 PM Introductory Comments

2:05 PM
Catalytic Combustion of Coal and Its Application in Blast Furnace Ironmaking: *Zhan-cheng Guo*[1]; [1]University of Science and Technology, Beijing

Catalytic combustion of coal is an energy saving technology. However, due to the low activity or high cost of catalyst or its negative impact on device and applied process, there are a few applications in industry. The present paper will introduce how to improve the activity of catalyst for pulverized coal combustion. Our experimental results show that size and surface electric property of catalyst are key factors to affect the catalytic activity, the less the volatile of the coal, the more effect of catalytic combustion. A kind of Ca(OH)2 based catalysts with nana-micro size was produced, and it was applied to pulverized coal injection combustion in Blast Furnace ironmaking process. When adding the catalyst about 0.5% of coal and coal injection about 150 kg per ton iron, saving coke over 10 kg per ton iron was achieved.

2:25 PM
Improving Energy Efficiency in a Modern Aluminum Casting Operation: *C. Eckert*[1]; Mark Osbourne[2]; Ray Peterson[3]; [1]Apogee Technology, Inc.; [2]General Motors Powertrain; [3]Aleris International Inc

The theoretical melting energy requirement for a typical hypoeutectic aluminum-silicon alloy is approximately 520 BTU/lb. It has been demonstrated, however, that even a state of the art secondary processing-automated lost foam casting operation can exceed this value by at least an order of magnitude when the actual thermal energy input from melting to solidification is monitored. Metal transfer and holding operations constitutes over 65% of this expenditure. The authors present relative benchmark energy expenditure information by unit operation for an off- site melting/lost foam casting line with a daily throughput in excess of 100,000 lbs. Efficiency improvements through optimization of the current process, and anticipated energy values at the culmination of a U.S.

Department of Energy sponsored project to develop, integrate and demonstrate an advanced melting, transportation, and dispensation system will be cited.

2:45 PM
Overview of the Department of Energy's Industrial Technologies Program: *Bob Gemmer*[1]; [1]US Department of Energy

An overview of the Industrial Technologies Program (ITP) will be provided. The presentation will focus both on near-term efforts to reduce the energy intensity of American industry as well as longer-term research activities needed to maintain progress once all the "low hanging fruit" are addressed. Specific examples will be given on the approach ITP takes for helping plants through Save Energy Now assessments. In additional, recent successful development of advanced, energy efficient industrial technologies will be described. Future directions for ITP will be discussed.

3:05 PM
Oxyfuel – Energy Efficient Melting: *Thomas Niehoff*[1]; David Stoffel[2]; [1]Linde Gas; [2]Linde North America, Inc.

Energy in form of natural gas, oil and electricity is expensive and will continue to be a rare resource in the future. Recycling of metals instead of primary production is a logic and crucial step towards energy conservation. Greenhouse emissions and energy consumption are impacted by using advanced combustion systems for metals recycling. Airfuel combustion has been the conventional way to melt and recycle metals. Competitive pressure from global companies as well as high energy prices force melt shop operations to reduce and optimize energy usage and cost. Oxyfuel combustion and process technology can be applied in most cases. Linde Gas has converted several hundred furnaces from airfuel to oxyfuel and has extensive experience to avoid start up issues. Oxyfuel process technology does require optimization and experience in the field. This paper will describe the benefits and potential issues for conversion to oxyfuel.

3:25 PM
Energy Savings and Productivity Increases at an Aluminium Slug Plant Due to Bottom Gas Purging: *Klaus Gamweger*[1]; Peter Bauer[2]; [1]RHI AG; [2]NEUMAN Aluminium Austria GmbH

Gas purging systems are well established in nonferrous metallurgy at multiple steps during metal production, including melting, converting, alloying, and metal cleaning. Since the kinetics of all of these stages are positively influenced by using gas purging systems the overall process benefits are substantial. In the aluminum industry, inert or reaction gases are blown into the melt using porous plugs to achieve improved metal grades, higher productivity, and more efficient energy utilization. To enable the most effective application of the process gases a complete package, termed AL KIN, was developed by RHI that includes porous plugs, refractory expertise, gas supply technology, and gas control equipment. This paper discusses the technological and economic advantages of this system and the specific benefits of fuel reduction, production time savings, decreased process gas consumption, and improved refractory service life and maintenance are illustrated using an aluminum slug plant in Austria.

3:45 PM Break

3:55 PM
Energy Conservation and Productivity Improvement Measures in Electric Arc Furnaces: *Ajit Jaiswal*[1]; [1]Steel Authority of India Limited

Electric Arc Furnace (EAF) being a power-intensive equipment, EAF based industries are highly dependent on scarce electric energy. This is the reason Government of India is highly concerned about this and laid emphasis on the conservation of electric energy. This paper highlights factors contributing to reduction in specific electric energy consumption and suggests measures not only for power saving, but also for productivity improvement. To name a few, these are proper sealing of furnace, introduction of oxy-fuel burners, foamy slag practice, increased usage of hot metal/DRI, post-combustion of CO gas for preheating of scrap, improved selection & design criteria of electrics, power demand management, process automation, etc. Few cases also have been cited, where the above measures have helped in reaping significant benefits.

4:15 PM
Evaluating Aluminum Melting Furnace Transient Energy Efficiency: *Edward Williams*[1]; Donald Stewart[1]; Ken Overfield[1]; [1]Alcoa

Recent increases in energy cost have led to a renewed focus on energy efficiency during aluminum melting operations to reduce fuel usage. The goals of a batch melting operation are to melt the charge using the minimum required energy, to melt the metal to the required temperature within the required timeframe, and to avoid generating unwanted contaminants by overheating the charge. The heat transfer efficiency of a conventional hydrocarbon fired aluminum melting furnace varies over the course of the melt cycle, depending on the conditions in the furnace. The purpose of this work was to develop a method of determining the transient heat transfer efficiency throughout the furnace cycle and to take advantage of this knowledge to optimize the melting process through furnace controls and proper production operations. This furnace survey methodology has been developed and used to evaluate a number of furnaces.

4:35 PM
Billions of Dollars Could be Saved with Reliability Excellence: Darrin Wikoff[1]; Scottie Williams[1]; *Tom Dabbs*[1]; [1]Life Cycle Engineering

The data, compiled by the Department of Energy indicates that the average industrial plant could reduce its total energy cost by 14.8% by implementing effective Life Cycle Asset Management processes. The actual savings varies by industrial classification, but in all cases the potential is substantial and could have a marked impact on the operating profit of the company. Improvements geared towards improving equipment reliability have distinctive linkages to environmental performance, such as reducing the amount of product and raw material waste through routine monitoring of system parameters through predictive technologies, and preventing interruptions to production cycles with a focus on Overall Equipment Effectiveness. Alcoa successfully reduced solvent disposal costs by more than 40% and GE reduced greenhouse gas emissions by more than 250,000 metric tons. This presentation will show you how companies have successfully reduced energy and disposal costs through a focused effort on manufacturing process reliability.

4:55 PM
Elements of an Energy Management Program: *Ray Peterson*[1]; Cynthia Belt[1]; [1]Aleris International Inc

World energy prices have increased significantly in the last several years. These increasing costs impact the overall manufacturing costs of industrial operations and their ability to be profitable. At the same time, there is societal pressure to reduce greenhouse gas emissions. To address these forces, every company should have an energy management program. In this paper, the key elements of an energy management program will be addressed using examples from the Aluminum metals industry. In particular, the elements of Data Collection, Data Analysis, Project Selection, Implementation, and Communications will be reviewed. The size and degree of sophistication of such a program will be dependent upon the magnitude of the energy costs and the resources available to address the issues.

5:15 PM
Understanding and Evaluating Energy Saving Options: *William Choate*[1]; Robert D. Naranjo[1]; [1]BCS Inc

Simply put - energy efficient heating, melting, holding, transporting, and refining operations do not let energy or metal (oxidation) escape. Numerous operating practices and technologies are available that conserve energy, lower costs, and lower GHG emissions (insulation; molten metal pumps; oxy-fuel firing; preheaters; recuperators; refractories, …). New technologies are emerging that promise even greater benefits (thermoelectric energy recovery; electron, infrared, microwave, plasma heating and melting; solar furnaces…). The challenge for manufacturers is how to evaluate a multitude of opportunities considering that each change brings new learning curves and combinations of changes have diminishing returns (i.e., benefits do not simply add together). This paper explores old technologies and practices that have new life given the price of energy today, emerging technologies, and experimental technologies. It examines lessons learned from changes and upgrades, and provides guidelines to understanding how incorporating the old, emerging or experimental technologies will impact productivity and profitability.

5:35 PM Concluding Comments

Fatigue: Mechanisms, Theory, Experiments and Industry Practice: Theory and Simulation

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee

Program Organizers: Koenraad Janssens, Paul Scherrer Institute; Corbett Battaile, Sandia National Laboratories; Brad Boyce, Sandia National Laboratories; Luke Brewer, Sandia National Laboratories

Monday PM Room: 3008
February 16, 2009 Location: Moscone West Convention Center

Session Chairs: Koenraad Janssens, Paul Scherrer Institute; Corbett Battaile, Sandia National Laboratories

2:00 PM Invited
A Geometric, Multiscale Approach to Stochastically Modeling Microstructurally Small Fatigue Crack Formation: *Anthony Ingraffea*[1]; Jacob Hochhalter[1]; Jeffrey Bozek[1]; Michael Veilleux[1]; Paul Wawrzynek[1]; [1]Cornell University

Recent advances in computational and experimental capabilities have provided the opportunity to model accurately fatigue damage in its entirety, from incubation to structural failure. The main thrust of the work presented here is toward the creation of a computational framework that geometrically models microstructurally small fatigue crack (MSFC) formation for a proof-test material: aluminum alloy 7075-T651. Methods are presented that generate and discretize statistically accurate microstructure geometry models, and explicitly simulate the MSFC formation stages: incubation, nucleation, and microstructurally small propagation. A multiscale approach is taken to couple a microstructural domain with the local deformation fields in a structural domain experiencing variable amplitude spectrum loading. The physics-based crack formation criteria are validated through direct comparisons to experimental observations. Thousands of simulations are computed, each for a unique statistical realization of the microstructure, to generate high fidelity, probabilistic predictions of MSFC growth rates.

2:30 PM
Microstructure-Based Approach for Predicting Microplastic Ratcheting in Metals: *Remi Dingreville*[1]; Corbett Battaile[1]; Luke Brewer[1]; Elisabeth Holm[1]; [1]Sandia National Laboratories

This study examines the elasto-plastic response of nickel microstructures under microplastic ratcheting conditions using a crystal plasticity model combined with experimental characterization of microstructures. The morphology and deformation behavior of polycrystals are characterized using Electron Back Scatter Diffraction (EBSD), while a non-local crystal plasticity framework with augmented kinematics is used in a computational context. The predicted cyclic behavior is compared against experimental results both at the macroscopic level and microstructural level. The examination of the macroscopic and microscopic material behavior suggests fundamental mechanisms of microplastic ratcheting at the microstructural scale, while the discrepancies between the experimental and computational observations underline the limitations of the current theoretical framework.

2:50 PM
Application of Field Dislocation Mechanics to Cyclic Plasticity: *Armand Beaudoin*[1]; Koenraad Janssens[2]; Amit Acharya[3]; [1]University of Illinois; [2]Paul Scherrer Institute; [3]Carnegie-Mellon University

In the study of cyclic plasticity, interplay between mechanisms of elasticity, anelasticity and (micro-) plasticity must be addressed. There exists a varying landscape of internal stress — due to dislocation interactions with boundaries, precipitates and solute atoms, for example. Some obstacles may be relatively soft, giving rise to a component of area swept by dislocations recoverable upon unloading. Development of local constitutive equations presents a challenge, with the variety of mechanisms at odds with the desire to maintain a tractable description. We adopt a field description, "Mesoscale Field Dislocation Mechanics" (MFDM), wherein long range stresses are developed through incompatibility and transient elastic response follows from the motion of the polar dislocation density. MFDM leverages existing models of continuum plasticity within a non-local framework. Cyclic deformation of a polycrystal is examined, with attention given to the development of mobile polar density, internal stress and effect on the averaged stress-strain response.

3:10 PM
Fatigue Mechanism and Multistage Fatigue Modeling for Wrought Mg-3Al-1Zn: *Yibin Xue*[1]; Adrian Pasco[2]; Mark Horstemeyer[2]; [1]Utah State University; [2]Mississippi State University

The microstructure-fatigue properties relation is developed based on multiscale fatigue experiments and micromechanical simulations. The large intermetallic particles in coarse grains at or near the surface are identified as the fatigue damage incubation sites. The morphology of the inclusion particles, as well as the bonding strength between the particle and alloy matrix, affects the fatigue incubation life as observed in micromechanical simulations in conjunction with the modified microscale Coffin-Manson law. The microstructurally and physically small crack growths were observed using in-situ SEM fatigue testing. The crack growth rate was directly quantified as a function of applied stress amplitude weighted by the applied stress ratio. The fatigue long crack growth was modeled combining a generalized Paris law with the application of a strip-yield model at the crack tip. Finally, the multistage fatigue model was implemented to evaluate the fatigue life of a simple component in an automobile Mg-front end application.

3:30 PM Invited
Microstructure-Sensitive Modeling of Rolling Contact Fatigue: Erick Alley[1]; *Richard Neu*[1]; [1]Georgia Inst of Technology

Crack nucleation, first spall generation and spall growth in rolling contact fatigue (RCF) are known to be highly sensitive to the heterogeneity of the microstructure. Yet the current state-of-the-art in the design of high performance bearing materials and microstructures is highly empirical requiring substantial lengthy experimental testing to validate the reliability and performance of these new materials and processes. We have laid the groundwork necessary to determine the influence of microstructure in RCF and related very high cycle fatigue problems. Crystal plasticity material models provide more realistic accumulations of localized plastic strains with cycling compare to homogenized J2 plasticity. With J2 plasticity, the bearing must be overloaded to capture significant plasticity near inclusions; with crystal plasticity, realistic bench test loads can be applied with plastic strain accumulation observed near inclusions in cases where RCF failure is anticipated.

4:00 PM Break

4:30 PM Invited
Microstructure-Sensitive Modeling of High Cycle Fatigue: Craig Przybyla[1]; Rajesh Prasannavenkatesan[1]; Nima Salageheh[1]; *David McDowell*[1]; [1]Georgia Tech

We explore microstructure-sensitive computational methods for predicting variability of low cycle fatigue (LCF) and high cycle fatigue (HCF) processes in metallic polycrystals to support design of fatigue resistant alloys. We outline a philosophy of establishing relations between remote loading conditions and microstructure-scale plasticity/crack behavior as a function of stress amplitude, stress state and microstructure, featuring calibration of mean experimental responses for known microstructures that bound the range of virtual (digital) microstructures. Effects of process history and resulting residual stresses are considered in certain cases of subsurface crack formation. The need to characterize extreme value correlations of microstructure attributes coupled to the local driving force (i.e., features) for HCF crack formation is outlined, along with a strategy involving a set of Fatigue Indicator Parameters (FIPs) relevant to different mechanisms of crack formation.

5:00 PM
Science-Based Modeling and Simulation of Fatigue Damage: *Elias Anagnostou*[1]; [1]Northrop Grumman Corp

Probabilistic microstructurally-based models for fatigue are being developed as part of a DARPA/Northrop Grumman Structural Integrity Prognosis System (SIPS). The fatigue models are based on a fundamental understanding of the fatigue process, and trace the structural degradation caused by fatigue back to its physical origins in the microstructure of the metallic component. The objectives are to discover and link all the important damage mechanisms leading to a macroscopically observable crack and to allow defects to emerge naturally from statistically meaningful ensembles of material representations, subject to more accurate, scale-specific, damage inducing fields. An example is modeling the

interplay between the grain crystallography and crack incubation at a constituent particle. A microstructurally-based model allows estimation of the total fatigue life from incubation and nucleation at a constituent second phase particle to propagation of micro-cracks to emergence of macro-cracks all in a statistical sense to permit accurate estimation of reliability indices.

5:20 PM
Small-Crack Growth Based Prediction of the Effect of Temperature on Fatigue Lifetime Distribution and Probabilistic Lifetime Limit in Ti-6Al-2Sn-4Zr-6Mo: *Sushant Jha*[1]; James Larsen[2]; [1]Universal Technology Corp; [2]US Air Force Research Laboratory

Recently, it has been shown that the fatigue lifetime distribution can be modeled as a superposition of the crack-growth probability density and a mean-lifetime-dominating density. It has also been demonstrated, that the effect of microstructural and extrinsic factors on the lifetime distribution can be understood in terms of the different rates of response of the two behaviors with respect to these variables, thereby producing a separation (or convergence) between them. In this paper, this modeling approach is applied to predict the lifetime probability density and the probabilistic lifetime limit as a function of temperature in Ti-6Al-2Sn-4Zr-6Mo. A range of temperatures including 23°C, 260°C, and 399°C were considered. The effect on the fatigue variability and the probabilistic limit could be analyzed, almost independently of the number of experimental points, via the role of the small-crack growth regime in separation (or convergence), with respect to temperature, of the two aforementioned responses.

MONDAY PM

Friction Stir Welding and Processing-V: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee
Program Organizers: Rajiv Mishra, Missouri University of Science and Technology; Thomas Lienert, Los Alamos National Laboratory; Murray Mahoney, formerly with Rockwell Scientific

Monday PM — Room: 2014
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Murray Mahoney, BYU

2:00 PM Invited
Microstructure and Properties of Friction Stir Welded 1.3wt%N Containing Steel: *Yutaka Sato*[1]; Kei Nakamura[1]; Hiroyuki Kokawa[1]; Shuji Narita[2]; Tetsuya Shimizu[2]; [1]Tohoku University; [2]Daido Steel

In this study, FSW was applied to a high nitrogen steel (HNS) containing 1.3wt% nitrogen using a PCBN tool, and feasibility of FSW for HNS, and microstructure and properties of the weld were examined. FSW produced defect-free welds at several welding parameters in the HNS. The stir zone had roughly the same nitrogen content as the base material, which suggested that both the PCBN tool wear and the nitrogen desorption hardly occurred during FSW. FSW refined the grain structure in the stir zone, which resulted in the higher hardness than the base material. Simultaneously, FSW resulted in rapid formation of Cr2N precipitates on the grain boundaries in the stir zone, which caused reduction of the corrosion resistance. This study showed that FSW is an effective method to produce a defect-free weld with high hardness in the HNS, although the corrosion resistance of the stir zone is reduced.

2:20 PM Invited
Friction Taper Stud Welding of Creep Resistant 10CrMo910: Daniel Hattingh[1]; Mark Newby[2]; *Axel Steuwer*[3]; Ian Widderburn[1]; Philip Doubell[2]; Malcom James[4]; [1]Nelson Mandela Metropolitan University; [2]ESKOM Holdings Ltd; [3]ESS Scandinavia; [4]University of Plymouth

Friction Taper Stud Welding (FTSW) is a novel welding technique that involves forcing a rotating consumable tool into a tapered (conical) cavity of nearly matching shape. The resultant generated heat causes a plasticised layer which bonds to the bottom of the hole and radially to the adjacent hole side. This is similar to other friction welding techniques such as linear and inertia friction welding, but involves a conical interface. Possible applications are repair welds in steel pipes. However, detailed knowledge of the residual stress distributions is essential for structural integrity interactions. This manuscript introduces the main concepts of FTSW and discusses the effects of pre and post weld heat treatment on the triaxial residual stress field (measured by neutron diffraction) generated by FTSW in a creep resistant steel manufactured from 10CrMo910 steel.

2:40 PM Invited
Friction Stir Welding of High Temperature Materials for Power Plant: *Seung Hwan Park*[1]; Kazutaka Okamoto[1]; Satoshi Hirano[1]; Akihiro Sato[1]; [1]Hitachi, Ltd. Materials Research Laboratory

Friction stir welding has been applied to high temperature materials for power plant such as 12 Cr steel, Ni-base oxide dispersion strengthened (ODS) alloy and Zr alloy, to examine the microstructures and properties in the welds. All welds were conducted using polycrystalline cubic boron nitride tool. Hardness remarkably increased in the 12 Cr steel weld, which was attributed to the formation of martensite in the stir zone (SZ). The weld of Ni-base ODS alloy showed the coarsening and aggregation of Y2O3 strengthening oxide particles in the SZ, which resulted in the decrease in the hardness. Remarkable hardness increase, which generally occurs due to oxygen absorption during welding, was not observed in the SZ of Zr alloy weld.

3:00 PM
Exploring Geometry Effects for Convex Scrolled Shoulder, Step Spiral Probe FSW Tools: *Carl Sorensen*[1]; Bryce Nielsen[1]; [1]Brigham Young University

A new tool design for FSW is the convex scrolled shoulder, step spiral probe (CS4) tool. Compared with traditional FSW tools, the CS4 tool has been demonstrated to offer larger process windows, lower operating forces, and the possibility of operating at zero tilt angle. This paper presents a parametric geometric description of the CS4 tool. Based on this description, a series of experiments has been performed to determine the effects of tool geometry on operating forces and weld surface finish. The advantages of convex scrolled shoulder tools in difficult FSW applications are presented.

3:20 PM
An Analytical Investigation of Tool Deformation and Wear in FSW of Hard Metals: Brian Thompson[1]; *Zak Pramann*[1]; Jeff Bernath[1]; Timothy Stotler[1]; [1]EWI

Friction stir welding (FSW) has progressed rapidly from a technology developed for joining of soft metals such as aluminum to a technology capable of joining hard metals such as steel, titanium and nickel based alloys. This advancement in technology has been possible primarily due to advancements in tool materials. There are two widely accepted tool material categories for FSW of hard metals: refractory metal based tools and composite tools. The tool technology has progressed to significantly reduce tool deformation, wear and breakage compared to early designs. However, tool deformation and tool wear and the mechanisms causing this degradation are still two very important topics in FSW. An analytical approach has been developed to study tool deformation and tool wear as two separate issues. Tool deformation will be evaluated using a thermomechanical model and tool wear will be studied using a tribological approach.

3:40 PM
Precipitation Reactions in Friction Stir Welded PH15-5 Steel: *Thomas Weinberger*[1]; Norbert Enzinger[1]; Horst Cerjak[1]; [1]Graz University of Technology

In the present study, the effect of friction stir welding on the precipitation microstructure of a martensitic precipitation hardened steel PH15-5 was investigated. Friction stir welding was performed using a tungsten based tool and different welding parameters. To analyze the temperature - stress profile in the plates, which has a significant influence on the precipitation reaction, a coupled thermo-mechanical model was used. Temperature measurements on the upper and bottom side of the plates were performed to verify the temperature distribution. To study the precipitation mechanism, advanced techniques like transmission electron microscopy and atom probe field ion microscopy were used. Additionally, the local mechanical properties of the joint were analyzed and the relationship between the precipitation and the hardness was studied. With the combination of different methods, it was possible to identify the hardening mechanism and the influence of the thermal cycle on the precipitation process.

4:00 PM Break

4:10 PM

Friction Stir Welding of "T" Joints in HSLA-65 Steel: *Murray Mahoney*[1]; Russell Steel[2]; Tracy Nelson[1]; Scott Packer[3]; Carl Sorensen[1]; [1]BYU; [2]Megadiamond; [3]Advanced Metals Products

Our objective is to demonstrate a practical approach for friction stir welding (FSW) "T" joints in long lengths of HSLA-65 steel. FSW of HSLA-65 steel offers challenges but achieving a defect-free weld in a "T" joint geometry in HSLA-65 steel is even more challenging. In addition to producing a sound weld nugget, the "T" fillet requires additional consideration. An excessive fillet volume can create thinning of the top sheet and an unbonded lap adjacent to the leg of the "T" where metal extrudes into the fillet cavity. Conversely, if the fillet volume is small, there is a risk of the FSW tool contacting the support tooling. Our FSW studies use both different PCBN tool designs and different "T" joint geometries in attempts to circumvent these concerns and create a practical weld approach. Metallographic results, tool designs, different "T" joint geometries, and joint properties will be presented.

4:30 PM

Surface Processing, Tempering and Toughening of 4340 Steel by Friction Stir Processing (FSP) of Melt-Deposited Alloy Layer: Sibasish Mukherjee[1]; *Amit Ghosh*[1]; Harshad Natu[2]; Ashish Dasgupta[3]; [1]University of Michigan; [2]POM Group, Inc; [3]Focus Hope

Direct Melt Deposition (DMD) of pre-alloyed 4340 steel, complemented by FSP, has been examined as a means to repair damaged areas of expensive components used by the Navy. Laser deposition process of steel 4340 steel powder leads to fully martensitic structure and high hardness but with low toughness. Surface processing of DMD 4340 steel by FSP using W-Re tool is found to provide penetration of deformation and thermal effects several millimeters into the material surface encompassing the depth of the DMD layer and converts this layer into a high toughness repaired region. In addition to processing by W-Re tool, flame softening followed by FSP by using H-13 tool steel was also found to be an adequate low-cost approach to develop a tempered and togh microstructure. Microstructural changes and mechanical property after these operations will be reviewed.

4:50 PM

Microstructure and Mechanical Properties of Friction Stir Welded MA956: *Ramprashad Prabhakaran*[1]; Wei Yuan[2]; James Cole[1]; Rajiv Mishra[2]; Indrajit Charit[3]; [1]Idaho National Laboratory; [2]Missouri University of Science and Technology; [3]University of Idaho

Oxide dispersion strengthened (ODS) steels would require good weldability for in-core applications in advanced nuclear reactors. Conventional fusion welding of ODS steels can cause undesirable effects such as coalescence of oxide dispersoids and significant porosity. In this study, friction stir welding was performed in bead-on-plate configuration on an ODS MA956 steel sheet using a cermet tool. Tensile properties of the parent and the stir zone materials were evaluated using mini-tensile testing. Interestingly, the yield and tensile strength of the stir zone showed marked improvement over the parent material with no loss in ductility. Microhardness profile of the processed material was obtained to understand the extent of microstructural gradient. Optical microscopy and transmission electron microscopy were used to evaluate changes in grain size and characteristics of the nanoscale oxide dispersoids (particle size, volume fraction, etc.) across the processed zone. This work is partly supported by the US Department of Energy.

5:10 PM

Correlating Extended Plasticity Mechanisms to Final Microstucture in Friction Stir Welding of 304l Stainless Steel: *Benjamin Nelson*[1]; [1]Brigham Young University Department of Mechanical Engineering

The formation of sigma phase (which assists corrosion) in friction stir welded (FSW) 304L stainless steel is one of the main obstacles keeping FSW 304L from being used in industry. There is evidence that sigma phase formation, in general, is a recrystallization related phenomenon. The proposed research is focused on identifying the mechanisms of extended plasticity active in producing the final microstructure in FSW 304L stainless steel. This research will give further insight into whether recrystallization plays an active role in the formation of sigma phase in FSW 304L. This characterization will be carried out for several regions within the stir zone. The mechanisms will be identified by use of EBSD. Using five FSW process parameters a central composite design will be used to determine a relationship between extended plasticity mechanisms and process parameters. FSW will be performed using a polycrystalline cubic boron nitride convex scrolled shoulder step spiral tool.

5:30 PM

Quantifying Post-Weld Microstructures in FSW HSLA-65: Tracy Nelson[1]; *Lingyun Wei*[1]; Majid Abassi[1]; [1]Brigham Young University

A comprehensive microstructural investigation of friction stir welds in HSLA-65 steel has been undertaken. Friction stir welds were made in 6.4 mm HSLA-65 steel over a range of process parameters using a polycrystalline cubic boron nitride (PCBN) convex scroll-shoulder step-spiral (CS4) tool. The post weld microstructure was investigated by optical microscopy (OM) and Orientation Imaging Microscopy (OIM). OM revealed primarily lath upper bainite microstructures in the stir zone. OIM was used to establish quantitative measures of the prior austenite grain size, bainite packet size, and lath size. Prior austenite grain sizes in the stir zone were as large as 50µm. This new approach to acquiring quantitative microstructural data is presented.

5:50 PM

Friction Stir Welding of Dual Phase Steel: *Wei Yuan*[1]; Jeffrey Rodelas[1]; Rajiv Mishra[1]; [1]Missouri University of Science and Technology

Friction stir welding (FSW) of a dual phase (DP590) steel was evaluated with a cemented carbide tool. Different tool traverse speeds at 1000 rpm tool rotation rate were employed to compare the microstructural changes (phase transformation, grain morphology, and grain size) and mechanical properties in the nugget region. The properties of parent material and nugget regions were characterized by mini-tensile and microhardness tests. The yield strength and ductility increased in the nugget region after FSW. For these traverse speeds, the yield strength and ductility increased with the increase in traverse speed. Microhardness profiles showed that hardness in the nugget was higher than base material and the heat affected zone was the softest region. Detailed microstructure evolution and corresponding thermal history in different regions will be compared and discussed.

Frontiers in Solidification Science III: Dendritic Growth Phenomena

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Phase Transformations Committee, TMS: Solidification Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Ralph Napolitano, Iowa State University; James Morris, Oak Ridge National Laboratory

Monday PM — Room: 2018
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Jeffrey Hoyt, McMaster University

2:00 PM Invited

Measurements of Dendrite Tip Growth in Succinonitrile-Acetone Alloys: *Christoph Beckermann*[1]; Antonio Melendez[1]; [1]University of Iowa

Measurements are performed of dendrite tip growth of succinonitrile-acetone alloys solidifying freely in an undercooled melt. The experiments are conducted using a setup similar to the IDGE of Glicksman and coworkers. The setup allows for precise measurements of the dendrite tip velocity, radius and shape for a range of undercoolings and solute concentrations. The measurements are compared to available theories of free dendritic growth. It is found that for pure succinonitrile, the measured dendrite tip Péclet numbers and selection parameters agree well with previous theories of free dendritic growth, if the effects of melt convection are taken into account. For finite solute concentrations, however, the tip selection parameter is found to deviate significantly from the pure succinonitrile value, especially at higher undercoolings. Furthermore, the three-dimensional dendrite tip shape becomes significantly more anisotropic. In light of this new data, a re-examination of the dendrite tip growth theory for alloys is needed.

2:20 PM Invited

Pattern Formation in Dendritic Directional Solidification of Al-Based Alloys: Investigation of 3D- Dendrite Shape and Dynamical Mechanical Effects by Synchrotron Live X-Ray Imaging: *Bernard Billia*[1]; Henri Nguyen-Thi[1]; Nathalie Mangelinck-Noel[1]; Nathalie Bergeon[1]; Adeline Buffet[2]; Guillaume Reinhart[3]; Thomas Schenk[4]; Jose Baruchel[2]; Hyejin Jung[5]; Jurgen Hartwig[2]; Paul Tafforeau[2]; [1]CNRS - University Paul Cezanne; [2]ESRF; [3]European Space Agency; [4]Ecole des Mines de Nancy; [5]National Fusion Research Institute

Precise characterization of the dynamical formation and selection of the dendritic microstructure in alloy solidification is critical for both the understanding of fundamental aspects and the breaking of technology barriers in materials processing. Owing to a unique experimental set-up combining in situ and real-time X-ray radiography and topography at the European Synchrotron Radiation Facility, detailed investigation of the solidification progress in thin Al-based alloys solidified upwards is enabled. Beyond revealing strains and stresses of various origins (shape-induced solute segregation; gravity causing bending of secondary arms and even dynamical disorientation along the primary trunk; competitive growth with eutectic behaving as a metal-matrix composite…) that have a significant influence on the crystalline quality of dendrites, X-ray topography gives access to the 3D-morphology through the equal-thickness fringes captured in Laue 2D-images. These 3D-shapes can be compared with theoretical predictions for free growth and growth in a channel, and lateral confinement effects discussed.

2:40 PM

Real Time Observation of Dendritic Solidification in Real Alloys by Synchrotron Microradiography: *Bin Li*[1]; [1]Johns Hopkins University

The opacity of real alloys poses a challenge to the study ofdendrite growth during solidification. Conventional experiments have to be performed after solidification is completed or interrupted. We present real time observations of dendrite growth in real alloys (Sn-Bi and Al-Cu) by using synchrotron radiation and the cutting-edge technology at national synchrotron facilities at CHESS and APS. Dendrite growth and coarsening in Sn-13%Bi alloy was studied in real time. Kinetics of coarsening was measured based on the real time observations. Dendrite morphology evolution during directional solidification was also studied, and we found that temperature gradient zone melting (TGZM) had a strong effect on the dendrites. These observations provided unambiguous understanding towards morphological evolution during dendritic solidification in real alloys.

3:00 PM

Spatial Correlations in Directionally Solidified Dendrites: *Amber Genau*[1]; Peter Voorhees[1]; [1]Northwestern Univ

Spatial correlations in directionally solidified Pb-Sn dendrites are analyzed using a recently developed technique for directly measuring the radial distribution function on complex, three-dimensional surfaces. We will discuss changes to the correlation function due to variations in the volume fraction of solid, as well as changes which occur after isothermal coarsening. At very long coarsening times, as the morphology undergoes dramatic changes to become predominantly vertically aligned tubes, long-range periodic order appears. Changes in correlation are also compared between dendrites in a typical mush and those in regions more reminiscent of free-growing dendrites. These types of spatial correlations are critical to understanding the evolution of solidification structures, as the coarsening process is driven both by local mean curvature and by diffusional interactions with surrounding interface.

3:20 PM Invited

Effect of Interface Anisotropy on Spacing Selection in Constrained Dendrite Growth: *Ingo Steinbach*[1]; [1]Ruhr-University

The selection of spacing in directional dendritic solidification is investigated numerically using the phase-field method in 2D and 3D. A criterion for the critical spacing below which no stable array growth can exist is derived from analysis of individual tip shapes. Constricted solute diffusion in the array leads to a deformation of the dendrite tip shape that competes with the deformation due to surface tension anisotropy. At the critical spacing both effects balance and a stable growth solution is destroid. This mechanism is identified to determine the critical spacing of a dendritic array and leads to a dependence of the spacing on the anisotropy of the solid-liquid interface energy in a similar way as for the dendrite tip radius.

3:40 PM Break

4:00 PM Invited

In-situ Observations of Coarsening of Dendritic Solid-Liquid Mixtures: J.L. Fife[1]; L. Aagesen[1]; E.M. Lauridsen[2]; *Peter Voorhees*[1]; M. Stampanoni[3]; [1]Northwestern University; [2]RISO Laboratories; [3]Paul Scherrer Institute

The solid-liquid mixtures produced following dendritic solidification are morphologically complex with spatially varying mean and Gaussian curvature. To understand the manner in which these systems evolve during coarsening, we have employed in-situ three-dimensional x-ray tomography and phase field simulations. Both the experiments and simulations determine the evolution of the interface shape distribution, the probability of finding a patch of curvature with a certain mean and Gaussian curvature. The approach allows the interfacial velocities to be determined experimentally and compared directly to phase field simulations that employ the experimentally measured microstructures as initial conditions. We also determine both experimentally and theoretically the flow of the probability that governs the evolution of the interfacial shape distribution. The experiments show the importance of topological singularities in the coarsening process as well. An analysis of this process will be given.

4:20 PM Invited

Ginzburg-Landau Model of Polycrystalline Solidification: *Alain Karma*[1]; Robert Spatschek[2]; [1]Northeastern University, Boston, Physics Department and Center for Interdisciplinary Research on Complex Systems; [2]Northeastern University, Boston, Physics Department and Center for Interdisciplinary Research on Complex Systems - and - Ruhr-University, Interdisciplinary Centre for Advanced Materials Simulation

This talk will describe a Ginzburg-Landau model of polycrystalline solidification that is formally derived by a multiple scale analysis of the phase-field crystal model. The free-energy of the model is formulated in terms of complex order parameters that describe the slow spatial modulation of both the amplitude and orientation of density waves corresponding to principal reciprocal lattice vectors of the crystal lattice. This model has the advantage that is can be used to simulate efficiently polycrystalline solidification with defects and elastic interactions in the limit of small misorientation between crystal grains where the model is quantitatively valid. Fundamental insights into grain boundary prelmelting in pure metals obtained with this model will be discussed in the light of quantitative comparisons with phase-field crystal model predictions and atomistic simulations.

4:40 PM

Real-Time X-Ray Observations of Hot Tearing in Al-Cu Alloys: *Richard Hamilton*[1]; Devashish Fuloria[1]; Andre Phillion[2]; Peter Lee[1]; [1]Imperial College London; [2]Ecole Polytechnique Fédérale de Lausanne

Hot tearing was directly observed using an in situ, high temperature, tensile/compression tester and x-ray radiography in synchrotron and laboratory sources. This allowed the load to be measured whilst directly observing the deformation of the primary dendrites and flow of Cu-enriched interdendritic fluid. The localisation of load, followed by void formation, coalescence and final fracture was observed whilst monitor the changes in load. The effect of cooling rate and strain rate on the mechanisms of hot tears initiation, growth, and potential healing was studied. At low strains, healing by liquid flow was observed, whereas at higher strains void formation combined with liquid necking between grains was prevalent.

5:00 PM

Modeling on Dendrite Growth during Slab Continuous Casting of Stainless Steels: *Wei Guo*[1]; Lifeng Zhang[1]; Miaoyong Zhu[2]; [1]Missouri University of Science and Technology; [2]Northeastern University

Dendrite growth is an important phenomenon of solidification structure, which is controlled by interfacial atom deposit dynamics, interfacial tension, heat diffusion, mass diffusion, etc. In the current paper, the undercooling for AISI304 type stainless steel in both the mold and the secondary cooling zone of the continuous casting process was simulated using non-traditional methods. The radius of the dendrite tip, growth velocity and the temperature gradient in the front of S/L interface were calculated. The simulation agreed well with the experimental data published in the literature..

5:20 PM
Novel Periodic Diphase Dendrite Structure in Solidified Al-35wt. %La Alloy: *Zidong Wang*[1]; [1]McGill University

By vacuum-melting and casting in metal, graphite and sand molds, Al-35wt. % La alloys with different solidification velocities were fabricated. With the help of X-ray diffraction and SEM, a novel dendrite structure has been determined to be found in Al-35wt. % La alloy. The dendrite is composed of a-Al alternating with Al11La3 and thus called periodic diphase dendrite structure. The dendrite structure, different from common dendrite of single phase, is composed of alternating two phases, so that the chemical compositions along the arms of the diphase dendrite change in discontinuous and periodic oscillatory. The structure is somewhat similar to banded structure and possesses light and dark regions to turn up alternately; but unlike banded structure, the regions, which are of two phases, regularly arrange in dendrite shape to form the periodic diphase dendrite structure with the chemical composition periodic variation along a dendrite growth direction.

Global Innovations in Materials and Technologies for Energy Harvesting: Plenary Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Global Innovations Committee
Program Organizers: Sivaraman Guruswamy, University of Utah; Robert Hyers, University of Massachusetts, Amherst; Joy Forsmark, Ford Motor Co

Monday PM — Room: 3005
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Joy Forsmark, Ford Motor Co; Sivaraman Guruswamy, University of Utah; Robert Hyers, University of Massachusetts

2:00 PM Introductory Comments

2:05 PM Keynote
Harvesting Alternate Energies from our Planet: *Bhakta Rath*[1]; [1]Naval Research Laboratory

Recent price increases at the gas pump have brought our attention to the phenomenal increase of global energy consumption in recent years. It is now evident that we have almost reached a peak in global oil production. Several projections indicate that total world consumption of oil will rise by nearly 60% between 1999 and 2020. In 1999 consumption was equivalent to 86 million barrels of oil per day, extracted from most known oil reserves. These projections, if accurate, will present an unprecedented crisis to the global economy and industry. As an example, in the US, nearly 40% of energy usage is provided by petroleum, of which nearly a third is used in transportation. An aggressive search for alternate energy sources, both renewable and nonrenewable, is vital. The presentation will review national and international perspectives on the exploration of alternate energy with special focus on energy derivable from the ocean.

2:55 PM Plenary
Solar Photovoltaics Technology: The Beginning of the Revolution: *Larry Kazmerski*[1]; [1]National Center for Photovoltaics, National Renewable Energy Laboratory

The prospects of current and coming solar-photovoltaic (PV) technologies are envisioned, arguing this solar-electricity source is at a tipping point in the complex worldwide energy outlook. The co-requirements for policy and technology investments are strongly supported. The emphasis of this presentation is on R&D advances (cell, materials, and module options), with indications of the limitations and strengths of crystalline (Si and GaAs) and thin-film (a-Si:H, Si, Cu(In,Ga)(Se,S)2, CdTe). The contributions and technological pathways for now and near-term technologies (silicon, III-Vs, and thin films) and status and forecasts for next-generation PV (organics, nanotechnologies, non-conventional junction approaches) are evaluated. Recent advances in concentrators with efficiencies headed toward 50%, new directions for thin films (20% and beyond), and materials/device technology issues are discussed in terms of technology progress. Insights into technical and other investments needed to tip photovoltaics to its next level of contribution as a significant clean-energy partner in the world energy portfolio. The need for R&D accelerating the now and imminent (evolutionary) technologies balanced with work in mid-term (disruptive) approaches is highlighted. Moreover, technology progress and ownership for next generation solar PV mandates a balanced investment in research on longer-term (the revolution needs revolutionary approaches to sustain itself) technologies (quantum dots, multi-multijunctions, intermediate-band concepts, nanotubes, bio-inspired, thermophotonics, . . .) having high-risk, but extremely high performance and cost returns for our next generations of energy consumers. Issues relating to manufacturing are explored—especially with the requirements for the next-generation technologies. This presentation provides insights into how this technology has developed—and where we can expect to be by this mid-21st century.

3:40 PM Break

3:50 PM Plenary
New Composite Thermoelectric Materials for Energy Harvesting Applications: *Mildred Dresselhaus*[1]; Gang Chen[1]; Zhifeng Ren[2]; Jean-Pierre Fleurial[3]; [1]MIT; [2]Boston College; [3]Jet Propulsion Laboratory

There have recently been several important advances in both thermoelectrics research and industrial applications that have attracted much attention, increasing incentives for developing advanced materials appropriate for large scale applications of thermoelectric devices. One strategy that seems promising is the development of materials with a dense packing of random nanostructures as a route for the scale-up of thermoelectrics applications. The concepts involved in designing composite materials containing nanostructures for thermoelectric applications will be discussed in general terms. Specific application is made to the Bi_2Te_3 nano-composite system for use in power generation. Also emphasized are the scientific advantages of the nanocomposite approach for the simultaneous increase in the power factor and decrease of the thermal conductivity, along with the practical advantages of having bulk samples for property measurements. A straightforward path is identified for the scale-up of thermoelectric materials synthesis containing nanostructured constituents for use in thermoelectric applications.

4:35 PM Plenary
Lessons from Natural Photosynthesis for Synthetic Photosynthesis: Graham Fleming[1]; [1]University of California, Berkeley, Department of Chemistry - and - Lawrence Berkeley National Laboratory

In this talk, I will briefly outline the design principles responsible for the remarkable efficiency of, and regulation of, natural photosynthetic light harvesting. I will then show how some of these ideas are beginning to be applied in the design of human-constructed light harvesting systems and photoconversion devices.

5:15 PM Plenary
Integration of Manufacturing Limits to Design Methodologies: Stephane Renou[1]; *Shu Ching Quek*[1]; [1]GE Global Research

In 2007, US capacity of wind-powered generators was estimated at a total of 5.2 gigawatts, and worldwide capacity was 94.1 gigawatts. Currently wind turbines produces less than 1% of US electricity, however, in 2007 US saw an increase in wind energy by 45% and the US government plans to supply 20% of electricity with wind power by 2030, according to American Wind Energy Association. There is a clear need to improve robustness and production cycle time in order to meet the demands of the growing market. Innovative technologies in both blade design and manufacturing processes will allow our current platforms (1-2 megawatt systems) to produce larger megawatt class machines. Although larger blades are desirable for higher efficiency, there remain challenges in material development, manufacturing, and design to make larger blades a reality. Novel polymers that are durable and have favorable processing characteristics will need to be developed. Eventually recyclability is also needed for scrapped wind turbine blades due to damage and/or replacement. Combination of material development and process improvements will result in not only more efficient blades but also cost effective, lightweight, reliable wind turbines that would allow expansion of wind sites to lower wind speed locations.

5:55 PM Concluding Comments

Magnesium Technology 2009: Casting

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Monday PM Room: 2006
February 16, 2009 Location: Moscone West Convention Center

Session Chair: Randy Beals, Chrysler LLC

2:00 PM Introductory Comments

2:05 PM
Refinement of microstructure by electromagnetic vibration process in magnesium wrought alloy and cast alloy: *Kenji Miwa*[1]; Mingjun Li[1]; Takuya Tamura[1]; [1]National Institute of Advanced Industrial Science and Technology (AIST)

We have developed the refinement process of the microstructure of metallic materials by imposition of electromagnetic vibration force during solidification. This process is effective for both wrought and cast magnesium alloys. By imposition of a static magnetic field of 10 tesla under an alternative electric current of 60 A, the average grain size of the AZ31B wrought alloy and the AZ91D cast alloy was obtained about 50 micron. The grain size was affected by electric current frequency and decreased the minimum value at the special electric current frequency of 500 to 2000 Hz and 900 Hz for both wrought alloy and cast alloy, respectively. From experimental results, we suggested the mechanism of refinement of microstructure during solidification by imposition of electromagnetic vibration force. It is important the cavitation phenomenon in liquid phase and also the difference of electric conductivity between solid phase and liquid phase.

2:25 PM
Melt Conditioned High Pressure Die Casting (MC-HPDC) of Mg-Alloys: *Spyridon Tzamtzis*[1]; Huawei Zhang[1]; Nadendla Hari Babu[1]; Zhongyun Fan[1]; [1]Brunel University

The high pressure die casting (HPDC) process is characterized by low cost and high efficiency. However, HPDC Mg-alloy components have non-uniform microstructure, chemical segregation, and substantial amount of casting defects, such as porosity and hot tearing. Recently, we have developed a new shape casting process named as melt conditioned high pressure die casting (MC-HPDC) where liquid metal is conditioned under intensive forced convection provided by the MCAST unit (melt conditioning by advanced shear technology), and then transferred to a conventional HPDC machine for shape casting. Melt conditioning can be done at temperatures both above and bellow the liquidus of the alloy. Compared to conventional HPDC, the MC-HPDC process offers cast components with fine and uniform microstructure, much reduced cast defects and substantially improved mechanical properties. In this paper we present the microstructures and mechanical properties of MC-HPDC Mg-alloys processed under different conditions and discuss the solidification behaviour of conditioned melt.

2:45 PM
Microsegregation Study of Mg Alloys and Adaptation of Directional Solidification Technique: *Rainer Schmid-Fetzer*[1]; Djordje Mirkovic[1]; [1]Clausthal University of Technology

This paper copes with new challenges in directional solidification posed by liquid alloys containing both Mg and Al. These liquid alloys are highly reactive and attack standard ceramic as well as metallic container materials. Another novelty is an extension of the well known Scheil method to reflect solute profiles of components in all precipitating phases. It predicts the primary crystallizing Al-Mn intermetallic phase, experimentally detected in the microstructure. This prediction is also confirmed for the first time by application of an advanced processing of quantitative EPMA mapping data. Dendritic microstructures observed in longitudinal sections of the quenched mushy zone, X-ray maps of fully directional solidified cross sections and quantitative solute profiles reveal the impact of cooling rate and alloy type in a comparison of AZ31 and AM50. This work is supported by the German Research Foundation (DFG) in the Priority Programme "DFG-SPP 1168: InnoMagTec".

3:05 PM
Intermetallics Distribution in Two and Three Dimensions in High Pressure Die Cast Mg-Al Alloys: *Venkata Nagasekhar Anumalasetty*[1]; Carlos Caceres[1]; [1]University of Queensland

The strength of Mg-Al alloys is influenced by the solute content, the grain size, and the volume fraction and distribution of intermetallics. An additional factor is that the distribution of intermetallics is a function of the casting wall thickness. Hence, in order to find the contribution of intermetallics to the strength of a given casting, it is necessary to determine in detail the distribution of intermetallics across the cross-section. The distribution of intermetallics across the cross-section has been studied in hpdc Mg-Al alloys of various thicknesses. Scanning Electron Microscope (SEM) and dual beam (FIB [Focussed Ion Beam]-SEM) system have been used for 2D and 3D characterization of the intermetallics, in selected areas near the edge and in the core regions of the castings.

3:25 PM
Investigations on Hot Tearing of Mg-Al Binary Alloys by Using a New Developed Quantitative Method: *Zisheng Zhen*[1]; Norbert Hort[1]; Oliver Utke[1]; Yuanding Huang[1]; Nikolai Petri[1]; Karl Kainer[1]; [1]GKSS Research Center

Hot tearing, also referred as hot cracking, has been widely recognized as one of the most fatal defects in casting processes. Although it has been intensively investigated for decades, most of the contributions are still based on qualitative level. In this work, a quantitative method for investigating hot tearing had been developed. The new method is based on true contraction force measuring principle, and shows very good repeatability. The recorded true contraction force can not only quantitatively evaluate hot tearing susceptibility, but also monitor the hot tear initiation and propagation. With this method, hot tearing behavior of Mg-Al binary alloys has been investigated. The results show that increasing mold temperature decreases hot tearing susceptibility. The recorded true contraction force curves also indicate that increasing mold temperature increases hot tearing initiation temperature, i.e. liquid fraction. Therefore liquid refilling has a chance to heal the initiated hot crack.

3:45 PM Break

4:00 PM
Magnesium Recycling System Prepared by Permanent Mould- and High Pressure Die Casting: *Daniel Fechner*[1]; Norbert Hort[1]; Karl Kainer[1]; [1]GKSS Research Center

Due to changing legislation and an increasing use of magnesium alloys in the automotive industry, magnesium recycling will get more important in future. Treating end-of life vehicles often means shredding. Separating the resulting magnesium scrap according to chemical compositions is complex and expensive. Therefore it would be useful to define alloys made from blended post consumer scrap. For creep resistant alloys the weight per component is usually high and a secondary alloy is reasonable.The scenario of blended post consumer scrap from different heat resistant magnesium alloys was realised by modifying the AM50 system with varying additions of Ca, Si and Sr. After preparing a matrix of potential recycling systems via permanent mould casting, three alloys were selected for further processing via HPDC. The materials properties are compared with regard to the processing techniques.

4:20 PM
Solidification Behavior of Recyclable Mg Alloys - AZ91 and AZC1231: *Adam Gesing*[1]; Jerry Sokolowski[2]; Carsten Blawert[3]; N. Reade[2]; [1]Gesing Consultants Inc; [2]University of Windsor; [3]GKSS

Common Mg alloys come from the Mg-Al-Mn and Mg-Al-Zn-Mn families, AM and AZ respectively. The popular AZ91 die casting alloy has a high concentration of all of the common alloying elements and hence can accommodate new scrap from any alloys coming from these families. Old scrap often contains contaminants, notably copper which cannot be refined out and contributes to corrosion of the AZ91 product alloy. Recently it was determined that the addition of 3% Al and 2% Zn to AZ91 allows the product to accept up to a 1% Cu impurity without increasing the susceptibility of the product alloy to corrosion - leading to the development of the AZC1231 alloy. The solidification behavior of the AZ91 and AZ1231 alloys was tested under various solidification rates using the UMSA Technology Platform to determine the compatibility of the new alloy with various casting technologies.

4:40 PM
Stresses and Cracking during Direct Chill Casting of AZ31 Alloy Billet: *John Grandfield*[1]; Vu Nguyen[2]; Ian Bainbridge[3]; [1]Grandfield Technology Pty Ltd; [2]CSIRO; [3]CAST CRC

The Alsim FEM model which has been applied to aluminium DC casting was applied to the problem of crack formation during vertical direct chill casting of magnesium alloys. The model is a fully coupled thermal stress model. Predictions were compared to crack incidence observed for a variety of cast start speed conditions used on AZ31 alloy, 208 mm diameter billet casting. Crack incidence was related to the principle stresses and other criteria such as the liquid pressure in the mush. The model can be used to develop improved starting head designs and cast start practices.

5:00 PM
Refinement of Solidification Microstructures by the MCAST Process: Z. Fan[1]; *Mingxu Xia*[1]; Z. Bian[1]; I. Bayandorian[1]; L. Cao[1]; H. Li[1]; G.M. Scamans[1]; [1]BCAST

MCAST (melt conditioning by advanced shear technology) is a novel technology developed recently for conditioning liquid metal under intensive forced convection before solidification. It uses twin screw mechanism to impose a high shear rate and a high intensity of turbulence to liquid metal, so that the conditioned liquid metal has uniform temperature, uniform chemical composition and well-dispersed and completely wetted oxide particles with a fine size and a narrow size distribution. The microstructural refinement is achieved through an enhanced heterogeneous nucleation rate and an increased nuclei survival rate during the subsequent solidification. In this paper we present the MCAST process and its applications for microstructural refinement in both shape casting and continuous casting of magnesium alloys. Discussions will be made on the effect of intensive forced convection on the enhanced heterogeneous nucleation. The concept of physical grain refinement will be proposed and discussed in contrast to the conventional grain refinement.

5:20 PM
Preliminary Investigation on the Grain Refinement Behaviour of ZrB_2 Particles in Commercial Mg-Al Alloys: Gerald Klösch[1]; Brian McKay[2]; *Peter Schumacher*[2]; [1]Austrian Foundry Research Institute; [2]University of Leoben

This paper investigates the effect of ZrB_2 particles on the grain refinement of Mg-Al and commercial AZ alloys. Samples were taken in accordance with the TP1 test procedure and the resulting grain size of the primary Mg measured using the linear intercept method. An SEM equipped with EDS was employed to elucidate the effect of the Zr. Results show that the ZrB_2 successfully grain refines the Mg-Al alloy resulting in ultimate grain sizes of 100 and 60 µm for the synthetic ZrB_2 particles respectively. Mg-Al and AZ alloys can be successfully grain refined using ZrB_2 heterogeneous particles and the resultant effect should be beneficial in improving the mechanical properties of the alloy.

Magnesium Technology 2009: Primary Production

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Monday PM — Room: 2007
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Neale R Neelameggham, U.S.Magnesium LLC

2:00 PM Introductory Comments

2:05 PM
Cathode Wetting Studies in Magnesium Electrolysis: *Kevin McLean*[1]; James Pettingill[1]; Boyd Davis[2]; [1]Queens University; [2]Kingston Process Metallurgy Inc.

The effects of cathode materials and electrolyte additives on magnesium wetting were studied with the goal of improving current efficiency in a magnesium electrolysis cell. The study consisted of static wetting and electrolysis tests, both conducted in a visual cell with a molten salt electrolyte of MgCl2-CaCl2-NaCl-KCl-CaF2. The wetting conditions were tested using high resolution photography and contact angle software. The electrolysis tests were completed to qualitatively assess the effect of additives to the melt and were recorded with a digital video camcorder. Results from the static wetting tests showed a significant variation in wetting depending on the material used for the cathode. Mo and a Mo-W alloy, with contact angles of 60° and 52° respectively, demonstrated excellent wetting. The contact angle for steel was 132° and it ranged from142°-154° for graphite depending on the type. Improvements to the cathode wetting were observed with tungsten and molybdenum oxide additives.

2:25 PM
Mechanism and Kinetics of Reduction of Magnesium Oxide with Carbon: *Leon Prentice*[1]; Michael Nagle[1]; [1]CSIRO Minerals

The reaction mechanism of the gas-solid carbothermal reduction of magnesium oxide is not well known, although some kinetic evaluations have been conducted. Previous studies have reported a two- or three-stage reaction process, each with different activation energy, while others have found a catalytic effect of other metal species. The present study, conducted as part of ongoing research into the carbothermal process, found that the reaction mechanism and its kinetics may be usefully described by a phase-boundary-controlled model. The activation energy of the gas-solid reaction was found to be 222 (±20) kJ/mol. It did not exhibit multi-stage complexity, but was otherwise consistent with reported values. The data obtained are at a larger scale than previously investigated, which minimises the errors related to surface area differences. The information is useful for the scale-up and control of the carbothermal reduction process.

2:45 PM
A Study on Influence of Fluxing Additives on Magnesium Refining Process: *Yeliz Demiray*[1]; Bora Derin[1]; Onuralp Yucel[1]; [1]Istanbul Technical University

This study aims to investigate the effect of different flux addition and time on refining of crown magnesium produced via pidgeon process. The different flux compositions (MgCl2, KCl, CaCl2, MgO, CaF2, NaCl, and SiO2) with or without B2O3 additions and reaction durations (15-45 min) were selected in order to lower iron content in Mg ingot samples at 690°C. The chemical compositions of the final ingots were measured by using wet chemical analyses technique. Each final ingot was also subjected to a corrosion test to understand the influence of iron to Mg corrosion. It is found that when the flux composition with B2O3 was used, iron content in the Mg ingot can be reduced from 0.080 to 0.0027 wt%. The corrosion test results showed that corrosion rates decreased with decreasing iron content in Mg ingots. The minimum corrosion rate was obtained as 0,235 mg/cm2/day.

3:05 PM
Study on Ultrasonic Purification of Magnesium Alloy Melt: Qichi Le[1]; Zhiqiang Zhang[1]; Jianzhong Cui[1]; Xue Wang[1]; [1]Northeastern University

The fluxing processing, a traditional purification method for magnesium melt, not only bears the risk of flux inclusions but also is facing more and more environmental pressure today. Therefore, the effective substitutes for fluxing processing are paid more attention recently. The mechanical effect generated by ultrasonic field in the media also called as ultrasonic agglomeration in chemical industry could conglomerate solid particulates in suspending liquid and then realize their separation. In this research, it is used to treat magnesium alloy melt with aim to promote and accelerate the separation of oxidation inclusion from melt. The effects of ultrasonic power, ultrasonic processing temperature and the holding time after ultrasonic treatment on the inclusion distribution in the billet were investigated. The results indicate that the ultrasonic conglomeration produced at low power ultrasonic field could be used to promote and accelerate the separation of oxidation inclusions form magnesium melt.

3:25 PM
Prediction Model of Magnesium Powder Consumption during Hot Metal Pre-Desulfurization: *Dongping Zhan*[1]; Huishu Zhang[1]; Zhouhua Jiang; Zhouhua Jiang[1]; [1]Northeastern University

Based on the productive practice of a steel plant, adopted the back propagation (BP) algorithm with the network configuration of 4-12-1 and the range of normalization from 0 to 1, used Visual Basic 6.0 software, the prediction model of magnesium powder consumption during hot metal pre-desulfurization processing was established. Meanwhile, four parameters, which are the weight and temperature of hot metal, the initial and final sulfur content in hot metal, were selected as input parameters. The data of 210 heats were used as the training samples and the other 46 heats were randomly selected as the test samples. The results show that the prediction errors of magnesium powder consumption less

than ±5 kg and ±10 kg are 54.3 percent and 89.1 percent of the total test heats respectively. Average absolute error is 5.12 kg. Minimum absolute error is 0.02 kg. The model greatly coincides with the actual production operation.

3:45 PM Break

4:00 PM
Study on Electrolysis of Magnesium Oxide on 200A Scale: Shaohua Yang[1]; Fengli Yang[1]; Qingsheng Liu[1]; *Xianwei Hu*[1]; Zhaowen Wang[2]; Zhongning Shi[2]; Bingliang Gao[2]; [1]School of Materials and Chemistry, Jiangxi University of Science and Technology; [2]School of Materials and Metallurgy117#, Northeastern University

Preparation of aluminum-magnesium alloy from magnesium oxide was studied by molten salt electrolysis method. Aluminum liquid as cathode and graphite as anode, the test was carried through on 200A scale in MgF2-LiF-KCl electrolyte. It was proved that the process of electrolysis was stable, rang of variation for voltage cell was narrow, and the value was in 0.4V. Content of magnesium in alloy was not even, the highest and the lowest was 20%, 6%, respectively. Even alloy could be attained by re-melting the alloy, and current efficiency was about 82%. The loss of anode oxidation was not serious. The results attained by this test could provide some technical parameters for further developing test.

4:20 PM
Vacuum Thermal Extract Magnesium from Boron Mud: *Xiaolei Wu*[1]; Naixiang Feng[1]; Jianping Peng[1]; Yaowu Wang[1]; [1]Northeastern University

Boron mud is residue from which the ascharite minerals is produced borax by carbon dioxide-soda process. It still contains a lot of magnesium and silicon. This experiment utilized the vacuum-thermal to extract most of magnesium, and the residual materials which is suitable to manufacture flat glass. The process can come to clean production. The experiment includes two parts. At first, optimum roasting conditions were determined through roasting tests at 650~700° for 0.5~1.0h.The major composition of after calcination boron mud is Mg2SiO4. Then, in the process of vacuum-thermal reduction experiment, calcium carbide was used as reductant. Fortunately the reduction rate of magnesium can reach as high as 99.6%.

4:40 PM
Study on Behavior of Anode Bubble: Shaohua Yang[1]; Fengli Yang[1]; Qingsheng Liu[1]; *Xianwei Hu*[1]; Zhaowen Wang[2]; Zhongning Shi[2]; Bingliang Gao[2]; [1]Jiangxi University of Science and Technology; [2]School of Materials and Metallurgy117#, Northeastern University

Behavior of anode bubble was studied by transparency cell. It was proved that anode bubble was grown up gradually at bottom of anode, and bubble generated on side of anode was smaller than that at bottom of anode. Obvious phenomena were observed that diameter of anode bubble opposite cathode was the smallest in all bubbles, some small bubbles together into big bubble were not observed in whole test, and the bubbles were separated out electrolyte as small shape, this was different with that of anode other side. Behavior of anode bubble was influenced by current density. The diameter of anode bubble at high current density was bigger than that of anode bubble at low current density, and the released velocity of anode bubble at high current density was faster than that of anode bubble at low current density.

Manufacturing Issues in Fuel Cells: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS: Shaping and Forming Committee
Program Organizers: Tsung-Yu Pan, Consultant, Ann Arbor Michigan; John Bradley, General Motors Corp; Michael Miles, Brigham Young University

Monday PM — Room: 3006
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Tsung-Yu Pan,

2:00 PM
A High-Temperature Sealing Technology for Solid Oxide Fuel Cells: *Timothy Lin*[1]; Chunhu Tan[1]; Bob Liu[1]; Jens Darsell[2]; Scott Weil[2]; [1]Aegis Technology Inc.; [2]Pacific Northwest National Laboratory

A reliable, cost-effective high-temperature sealing technology for the joining of ceramic components to metallic structures is critical to the successful development of solid oxide fuel cells (SOFCs). In this presentation, Aegis Technology will present its latest development of a novel reactive air brazing (RAB) technology, which is sponsored through an U.S. Department of Energy (DoE) Small Business Innovative Research (SBIR) project in collaboration with the Pacific Northwest National Laboratory. This RAB technology uses Ag-CuO as base braze compostions with a varity of additional elements. With a proper processing control, the resultant sealing technology is capable of providing a high-temperature sealing with sufficient chemical inertness, thermal reliability, and bonding strength. This presentation will report our latest studies on composition design/synthesis of braze filler material, and the characterizations including microstructure and mechanical properties, and a preliminary numerical simulation addressing the residual stress development in the joining assembly during thermal cycling.

2:25 PM
Aging Effect on the Mechanical Properties of Perfluorosulfonate Polymer for Fuel Cell Proton Exchange Membranes: *Hyun Jee Park*[1]; Takuya Hasegawa[2]; Jiping Ye[3]; Reinhold Dauskardt[1]; [1]Stanford University; [2]Nissan Research Center, Nissan Motor Co., Ltd; [3]Research Department, Nissan Arc Ltd

Perfluorinated sulfonic polymers are widely used as proton exchange membranes in fuel cells. These polymers have high selectivity and permeability to water mediated by their sulfonic groups, and exhibit good thermal and mechanical stability. However, water sorption leads to extensive swelling and degradation of mechanical properties, which can degrade the performance and lead to early breakdown in fuel cells. In this study, we experimentally investigate the change of mechanical properties by aging at different thermal and hydrothermal conditions. Specifically, both micro-tensile tests and constrained and unconstrained swelling tests were conducted under selected environmental and exposure times. The effects of dehydration and hydration/dehydration cycling were also investigated. We shown that the membrane is highly sensitive to water content and mechanical properties significantly degraded by cycling. The work has implications for the thermal management of cells during operation and is intended to provide guidance on the long term reliability of membrane materials.

2:50 PM
Structural Information of α- Alumina Supported Cobalt Nanoparticle Catalysts during Autothermal Reforming of Iso-Octane: Mohammad Shamsuzzoha[1]; Earl Ada[1]; Ramana Reddy[1]; [1]University of Alabama

The microstructure of a nanoparticle Co catalyst supported on a-alumina prior to and after the autothermal reformation were studied using Transmission Electron Microscopy, and X-ray Photoelectron Spectroscopy. The support of the fresh catalyst exhibits a homogenous aggregation of amorphous granules with sizes ranging between 10 to 20 nm. The structure of the fresh catalyst support is of hexagonal alumina phase. Cobalt in the fresh catalyst is highly dispersed and embedded in the matrix in the form of contrasted crystallites with size in the range of 5 - 20 nm. The support of the used catalyst exhibits external coating made of carbon related compound, but show very little grain growth. Co particles in the reformed sample were found to be in the mooted form. Crystallographic information in relation to this autothermal reformation of a-alumina supported Co nanoparticles has been discussed in the light of the efficiency of Co as catalyst.

3:15 PM Break

3:30 PM
Economic Production of Metallic Separator Plates: *Marc Decker*[1]; [1]Graebener Maschinentechnik GmbH & Co. KG

Metallic separator plates are used for building high-quality and efficient fuel cells. To produce highest-quality metallic separator plates, a new production system had to be found since theoretical calculations and practical tests have shown that the machines and systems available on the market nowadays are not able to provide highest forces on small surfaces. In search of such an efficient system Gräbener has developed a special hydraulic press. This so called PowerBoxx®, the tool technology developed by Gräbener and the sheet hydroforming process provide a perfect symbiosis for the efficient production of highest-quality metallic separator plates. The system is especially designed for pressing thin metallic sheets (foils) within shortest cycle times and with greatest evenness. Gräbener will present this symbiosis starting from the idea to its realisation, compare it with other production systems and give an outlook on the future and the developments.

3:55 PM
Preparation and Characterization of Nano-Structured Proton Conductive Electrolytes: *Zhigang Xu*[1]; Jag Sankar[1]; [1]North Carolina A&T State University

The purpose of this study was to acquire a preliminary understanding in portion conductive electrolytes through material preparation and characterizations. 20mol% ytterbium doped Barium cerates which is partially substituted with zirconate was chosen for study. At the first place, nano crystalline powders were prepared using sol-gel technique. Pellets were produced from the powder compact by high-temperature sintering. Electrolyte thin films were also made by spin-coating of the gel. The crystallographic properties of the powders and sintered pellets were determined with X-ray diffraction. The crystallite size was measure by using Scherrer method and confirmed by TEM direct observations. The morphologies of the thin films and pellets were determined on the polished and etched surfaces by TEM. The conductivity of the material was measured using ac-impedance in a temperature range from 300-800ºC in the presence of 4% hydrogen in argon.

4:20 PM
Novel Coating Process to Facilitate Traditional Solder Connection to Graphitic Fabrics for Use in Fuel Cell Assemblies: *Ben Poquette*[1]; [1]Keystone Materials LLC

High conductivity graphite fabrics show much promise for use in future fuel cell assemblies. However, creating low resistance electrical connections with these fabrics generally requires excessive mechanical compression joints or high temperature brazing techniques which can damage other components of the fuel cell stack and require inert or vacuum processing. A nove process, to deposit a uniform coating around the individual fibers, has been developed to allow joining to graphitic fabrics by traditional soldering techniques.

Materials for High Temperature Applications: Next Generation Superalloys and Beyond: Next Generation Superalloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS: Refractory Metals Committee
Program Organizers: Joseph Rigney, GE Aviation; Omer Dogan, National Energy Technology Laboratory; Donna Ballard, Air Force Research Laboratory; Shiela Woodard, Pratt & Whitney

Monday PM — Room: 3010
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Dallis Hardwick, US Air Force; Sammy Tin, Illinois Institute of Technology

2:00 PM Invited
Superalloys: Evolution and Revolution for the Future: *Hiroshi Harada*[1]; [1]NIMS

Superalloys have evolved from wrought to conventionally cast, directionally solidified, and then single crystal (SC) alloys. SC superalloys have also evolved from 1st(0Re) to 2nd(2-3Re), 3rd(5-6Re), 4th(5-6Re and 2-3Ru), and then 5th (5-6Re and 5-6Ru) generation alloys. So far 1st to 3rd generation SC superalloys are used practically, e.g., CMSX-10, a 3rd generation alloy, as turbine blade materials in the latest aeroengines. The highest temperature capability, 1100°C (137MPa, 1000h creep rupture), has been reached by NIMS 5th generation alloys, typically TMS-196. In the 5th generation alloys, an interfacial dislocation network on γ and γ' phase boundary is designed to be finer (20 nm) to prevent dislocations from cutting through the interface and suppress creep deformation. In the present paper, after the introduction of historical evolution in superalloys, possible further evolution and revolution will be discussed in conjunction with advanced aeroengines and ultra-efficient gas turbines being planned to improve specific fuel consumption and reduce CO2 emissions.

2:25 PM
New Fabricable Dispersion Strengthened Cobalt Based Wrought Superalloy: *S. Srivastava*[1]; [1]Haynes International Inc

The problems associated with the fabricabilty, irreproducibility of properties, and high cost of mechanically alloyed ODS alloys provided the motivation for the development of a nitride dispersion strengthened alloy. The program goals for HAYNES® NS-163™ alloy (Nom. Comp: Co-28Cr-9Ni-21Fe-1.25Ti-1Nb) were to take a segmented approach to develop a sheet alloy that would be fabricable in the as-received condition and would achieve its high temperature creep strength as a result of a nitride dispersion strengthening (NDS) heat treatment. Specifically, the aim was to obtain a stress rupture life of > 250h at 982°C/55 MPa (1800°F/8 ksi). Based on the laboratory data, it appears that its 1000h-rupture strength at 982°C was more than twice that of HAYNES 188 and 230® alloys, the two leading solid solution strengthened gas turbine alloys. The paper will present preliminary data derived from the laboratory heats and a production heat, and briefly describe the ongoing work.

2:45 PM
Fatigue Crack Growth Behavior of the Ni-Base Superalloy ME3: *Jeffrey Evans*[1]; Ashok Saxena[1]; Andrew Rosenberger[2]; [1]University of Arkansas; [2]Air Force Research Lab

A set of crack growth tests was performed on the turbine disk alloy ME3 at 704°C (1300°F) in vacuum and in air at 0 and 10 second hold times using two microstructures developed with two different cooling rates. Fatigue crack growth tests were also conducted at 25°C (77°F) with the two microstructures. For the tests conducted in air at elevated temperature, both hold time and microstructural effects were evident while tests conducted in vacuum showed no difference regardless of microstructure or hold time. A coupling effect was also observed between the microstructure and the environment. The slow cooled samples had larger secondary gamma prime particles, slower crack growth rates, and less intergranular fracture in air as compared to the fast cooled samples.

3:05 PM
Dwell Notch LCF Behavior of Advanced Powder Metallurgy Disk Superalloys: Jack Telesman[1]; John Gayda[1]; Timothy Gabb[1]; [1]NASA Glenn Research Center

The lives of powder metallurgy superalloy disks in aerospace turbine engine applications can be limited by fatigue cracking at notches. The most severe limitations can sometimes occur for notched locations exposed to high temperatures and dwells at maximum stress, where cycle-dependent and time-dependent damage can each accumulate. Improvements of performance and durability in future disk applications require an understanding of what drives such damage. Several aspects of this fatigue cracking problem were examined in disk superalloys ME3 and LSHR using notched specimens. Specimens were fatigue tested at high temperatures, with dwells at maximum stress. The effects of applied stress, dwell time, and temperature on fatigue life and failure modes were examined and will be discussed.

3:25 PM
Alloy 10 - An as-HIP Compacted Nickel Based Superalloy for High Pressure Turbine Rotor Applications: *Derek Rice*[1]; Brian Hann[1]; Pete Kantzos[1]; Dan Greving[1]; James Neumann[1]; [1]Honeywell Engines, Systems & Services

As part of the VAATE program Honeywell evaluated the potential of PM Alloy 10 in the as-HIP super solvus heat treated condition for high pressure turbine disk applications. This report presents the high temperature mechanical properties of as-HIP coarse grain PM Alloy 10 relative to cast and wrought fine grain U720Li up to 760C. Properties presented and discussed include tensile, creep, LCF, and crack growth. Operating gas turbine disks above 700C will require sophisticated PM Ni based alloys. Utilizing these materials in the as-HIP compacted form will help mitigate component cost and risk.

3:45 PM Break

3:55 PM Invited
Development of Pt-Modified γ-Ni+γ'-Ni_3Al-Based Alloys Having Strength and Environmental Resistance at High Temperatures: *Brian Gleeson*[1]; Andy Heidloff[2]; Zhihong Tang[2]; Takeshi Izumi[3]; [1]University of Pittsburgh; [2]Iowa State University; [3]Hokkaido University

Heat-treatable γ-Ni+γ'-Ni_3Al-based alloys having excellent resistance to high-temperature oxidation, hot corrosion, and creep are being developed in a systematic manner using multiple alloying additions, including Pt and/or Ir, *i.e.*, platinum group metals (PGMs). Alloys that collectively possess these high-temperature properties are highly attractive for niche applications involving extreme conditions. The results discussed in this presentation stem from a larger-scale project supported by the U.S. Air Force within the Materials for Air-Breathing Propulsion in Support of the Versatile Affordable Advanced Turbine Engine (VAATE) Program. It will be shown that PGM additions reduce the detrimental effects of "strengthening" alloying additions on oxidation and

hot corrosion. Microstructural characterization of the alloys included elemental partitioning and thermal stability, with both being compared to thermodynamic predictions using the software package PANDAT.

4:20 PM

Gamma Prime Dissolution and Grain Growth during Supersolvus Heat Treatment of Advanced Ni-Base Disk Superalloys: *Eric Payton*[1]; Gang Wang[1]; Yunzhi Wang[1]; Dan Wei[2]; David Mourer[2]; Deborah Whitis[2]; Michael Mills[1]; [1]Ohio State University; [2]GE Aviation

Grain size control is critically important for achieving desired mechanical properties in Ni-base superalloys for turbine disk applications. New jet engine designs demand increased operating temperatures for improved efficiency. To improve manufacturing processes and useful life of the turbine disks, physics-based prediction of grain size as a result of thermomechanical processing is desired. The size and volume fraction of particles of the gamma prime phase have a significant effect on the grain size during heat treatment, and can influence the final grain size of the material. Dissolution of gamma prime occurs rapidly during supersolvus heat treatment. Grain growth and gamma prime dissolution during supersolvus heat treatment have been measured experimentally. Gamma prime dissolution observations have been compared to phase field simulation results to develop a model for gamma prime dissolution.

4:40 PM

Elemental Partitioning in Ni-Based Superalloys with PGM Additions: *Jason Van Sluytman*[1]; Tresa Pollock[1]; [1]University of Michigan

Elemental partitioning in Ni-based superalloys containing various PGM additions has been investigated through use of electron probe microanalysis (EPMA). Alloys with a baseline composition of 15Al-5Cr-1Re-2Ta-0.1Hf (at%) containing various amounts of Pt, Ir, Ru, and W, have been heat treated to produce coarse two phase γ-γ′ microstructures. Large γ′ particles approximately 3-4 μm diameter were utilized to acquire EPMA scans for phase composition to determine partitioning of elements between the two phases. Limited TEM energy dispersive spectroscopy as well as local electron atom probe analysis were also utilized to compare partitioning values gathered from EPMA. These analysis indicate that Cr, Re, and W partition preferentially to the matrix γ phase while Al, Pt, and Ta preferentially partition to the γ′ phase. Additions of Ir reduce the partitioning of W, as well as Re, to the matrix.

5:00 PM

Net-Shape, Powder Metal, HIP-Bonded Surface Layers for Environmental Compatibility of Superalloys in Rocket Engine Turbines: *Cliff Bampton*[1]; Victor Samarov[2]; Alex Lobovsky[3]; Daniel Matejczyk[1]; Mohammad Behi[3]; [1]Pratt & Whitney Rocketdyne; [2]Synertech Inc.; [3]United Materials Technologies, LLC

Net shape consolidation of powder metal (PM) by hot isostatic pressing (HIP) provides opportunities for cost, performance and life benefits over conventional fabrication processes for rocket engine structures. The method employs sacrificial metallic tooling (HIP capsule and shaped inserts), which is removed from net-shape surfaces of the consolidated part, by selective acid dissolution. Net-shape PM HIP enables fabrication of complex configurations providing additional functionalities. One example is discussed in detail: a novel HIP-Bonded Surface Layer method which has been demonstrated for provision of both smoother net-shape surfaces and robust surface layers of environmentally compatible alloys integral with a stronger, but less compatible substrate alloy.

5:20 PM

Next Generation Materials Property Profiles for Superalloy and Refractory-Based Panels in Scramjet Combustors: *N. Vermaak*[1]; L. Valdevit[2]; A. Evans[1]; [1]University of California, Santa Barbara; [2]University of California, Irvine

The operating conditions of scramjet engines require lightweight materials that withstand extreme heat fluxes and structural loads. An optimization tool has been introduced to direct the development of next generation materials that outperform existing high temperature alloys and compete with ceramic matrix composites. Performance maps reveal the relative merits of candidate superalloys and refractory-based materials over a broad operating domain. Specific results are presented for scramjet combustor liners applicable to a Mach 7 hypersonic vehicle (albeit the methodology is general). By probing the constraints that limit performance, the respective roles of the material properties and the design variables are unearthed. Based on these insights, notional materials are used to demonstrate how feasible design space can be reclaimed by tailoring critical material properties. These performance benefits are benchmarked and compared for two of the more viable material candidates: the nickel-based superalloy, Inconel X-750 and the niobium-based refractory alloy, Cb-752.

5:40 PM

Hot Working of Platinum Group Metal-Modified Nickel-Base Superalloys: *Donna Ballard*[1]; Lee Semiatin[1]; Patrick Martin[1]; [1]US Air Force

Platinum- and iridium-modified γ-γ' nickel-base superalloys are being evaluated for high-temperature use due to their superior oxidation resistance compared to conventional nickel-base superalloys. These materials also retain excellent strength at temperatures in excess of 1100°C due to a higher γ'solvus. Because of their cost and density, however, specific applications must be chosen carefully. Two product forms of interest are thin gage sheet and foil for thermal-protection-system applications. Initial research to evaluate the conversion of subscale ingots of two PGM alloys to sheet, the latter stages of which utilize conventional pack rolling, will be reviewed. Additional results on extrusion experiments on a third alloy will also be covered.

Materials Issues in Additive Powder-Based Manufacturing Processes: Additive Manufacturing Metals I

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Powder Materials Committee
Program Organizers: David Bourell, University of Texas; James Sears, South Dakota School of Mines and Technology; Pavan Suri, Mississippi State University

Monday PM — Room: 3004
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: John Smugeresky, Sandia National Laboratories

2:00 PM

Direct Digital Manufacturing with Layer-by-Layer Melt Deposition Processes: *Khershed Cooper*[1]; Sam Lambrakos[1]; [1]Naval Research Lab

The essence of direct digital manufacturing (DDM) is developing controllable, incremental, additive processes to generate objects and components without tools, without manual assembly, at point-of-use. DDM allows manufacturing where we can know the microstructure and properties at every moment and at every point during a build. Deposition processes involving metal powder are, among others, direct laser deposition (e.g., LENS, DMD), selected laser melting (SLM) and e-beam melting (EBM). These processes involve either powder feed or powder beds. To understand the manufacturing science of these "melt deposition" processes, we need to model the spatial-temporal dynamics of materials under conditions involving short time durations and intense localized heat. In this paper, we will present examples of ongoing direct "melt deposition" research and our attempts to develop thermal models for such processes using inverse problem methodology.

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Laser-Material Interaction Research in a Metal Deposition Process: *Frank Liou*[1]; Zhiqiang Fan[1]; Hsin-Nan Chou[2]; Kevin Slattery[2]; James Sears[3]; Mary Kinsella[4]; Joseph Newkirk[1]; [1]Missouri University of Science and Technology; [2]Boeing Phantom Works; [3]South Dakota School of Mines and Technology; [4]AFRL/RXLMP

A laser deposition process involves the supply of metallic powders into a laser-heated spot where the powder is melted and forms a melt puddle which quickly solidifies into a bead. The development of an accurate predictive model for laser deposition is extremely complicated due to the multitude of process parameters and materials properties involved. In this work, the metal powder used in the laser deposition process is injected into the system by using a coaxial nozzle. In order to design an effective system, the laser beam, the powder beam, and their interactions need to be fully understood. This presentation summarizes the work to model a powder delivery system using non-spherical particle-wall interactions. The laser-material interaction within the melt pool is also investigated using a multi-scale model: a macroscopic model to model mass, heat and momentum transfer, and a microscopic model to model the evolution of solidification.

2:50 PM
Maintaining Consistent Conditions over a Wide Range of Material Deposition Rates in Beam-Based Additive Manufacturing: *Jack Beuth*[1]; Shane Esola[1]; Raymond Walker[2]; [1]Carnegie Mellon University; [2]Keystone Synergistic Enterprises

Significant advances have been made in the development of laser and electron beam-based freeform fabrication processes using powder injection, powder bed or wire feed systems for material delivery. Electron beam-based deposition is currently receiving serious consideration for additive manufacturing and repair applications in the aerospace industry. To be successful, these processes must work over a wide range of material deposition rates to combine affordability (requiring high deposition rates) with the ability to precisely deposit fine geometries (requiring low deposition rates). The goal of this modeling research is to identify paths through processing space yielding consistent melt pool sizes independent of material deposition rate, ultimately yielding rules of thumb useable by processing engineers. Process variables to be controlled are beam power and translational speed.

3:15 PM
Effect of Process Parameters on Electron Beam Melted (EBM) Additively Manufactured Components in Ti-6Al-4V: *Micheal Blackmore*[1]; Sinan Al-Bermani[1]; Iain Todd[1]; [1]University of Sheffield

Additive layer manufacturing (ALM) in metallic materials has many potential applications and offers many advantages over conventional subtractive machining practices. However, ALM machines at present are yet to be fully utilised in a production environment due to lack of validation and process maturity. An Arcam S12 EBM machine has been used to deposit near net components in titanium 6Al-4V (grade 5) alloy operating directly from CAD data. The effects of changes in various process parameters such as beam speed, power, focus and scan strategy have been investigated and related to material integrity and as deposited surface finish.

3:40 PM Break

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Powder-Cored Tubular Wire Manufacturing for Electron Beam Freeform Fabrication: *Christine Hillier*[1]; Marcia Domack[2]; Robert Hafley[2]; Stephen Liu[1]; [1]Colorado School of Mines; [2]NASA Langley Research Center

Powder-cored tubular wires exhibit great flexibility in terms of final deposit composition when used in conjunction with a heat source, whether arc, laser or electron beam. By modifying the core composition, a wide range of chemical compositions can be easily produced. With known alloy recovery, powder-cored tubular wires can be used to produce deposits of custom chemical composition. In this work, the manufacturing process of titanium-based cored tubular wires is discussed. These tubular wires are manufactured via U-O bending of CP-Ti Grade 2 strip metal, with a core of Ti-Al-V powder. By adjusting the powder composition, the aluminum loss observed in wire-based electron beam processing can be mitigated. Using a button melting technique, custom powder compositions are being developed with enhanced Al and V chemistries to account for alloy losses, as well as sheath metal compositions.

4:30 PM
Structure-Property-Process Optimization in the Rapid-Layer Manufacturing of Ti-6Al-4V Components by Electron Beam Melting: *Sara Gaytan*[1]; Lawrence Murr[1]; Edwin Martinez[1]; Daniel Hernandez[1]; Stella Quinones[1]; Francisco Medina[1]; Ryan Wicker[1]; [1]University of Texas

Rapid prototype (RP) manufacturing using Ti-6Al-4V powder and electron beam melting (EBM) has presented the prospects of microstructure-property control within small volumes and linear dimensions of <1 mm. Utilizing optical and electron microscopy (SEM and TEM), it has been demonstrated that alpha (hcp) acicular platelet dimensions and dislocation substructures within these platelets, composing simple build geometries, can be varied with related variations in hardness, tensile strength, and elongation. These structure-property variations occur by thermal differences as a consequence of beam current, focus, and scan rate or scan sequencing. In addition, during layer building various defects can be created by beam tripping and related phenomena. These include spherical or irregular voids ranging from a few microns to tens of microns in diameter as well as porous zones of even larger dimensions which result from non-melting or local variations in liquid-phase sintering. Examples of these build-related defects will be described.

4:55 PM
Laser Surface Modification of 2024 Al Alloy to Enhance Thermal Conductivity: *Amit Bandyopadhyay*[1]; B. Vamsi Krishna[1]; Susmita Bose[1]; W. M. Keck[1]; [1]Washington State University

With rapid advances in microelectronics particularly in the areas of miniaturization with increased power and greater functionality, innovative heat-removal materials as well as techniques are needed for thermal management of active devices for next generation military and commercial applications. Different approaches have been used to make materials with properties suitable for thermal management applications, but not via surface modification. We have examined the feasibility of enhancing thermal conductivity of 2024 Al by depositing 80Cu-20Mo using a Laser Engineered Net Shaping (LENS\\153;). Coatings of 667 2.5 micron thickness were formed with metallurgically sound interface. Results showed an 87% increase in the thermal conductivity of 2024 Al alloy. The coating approach in combination with LENS\\153; process can also be used to deposit high TC materials in desired locations to reduce 'hot spots'. The presentation will discuss materials and manufacturing issues related to laser surface modification of 2024 Al alloy to enhance thermal conductivity.

5:20 PM
Spheroidisation and Oxide Disruption Phenomena in Direct Selective Laser Melting (SLM) of Pre-Alloyed Al-Mg and Al-Si Powders: *Eyitayo Olakanmi*[1]; Robert Cochrane[1]; Kenneth Dalgarno[2]; [1]University of Leeds; [2]University of Newcastle

Spheroidisation and oxide disruption phenomena in the direct laser melting of pre-alloyed Al-Mg and Al-Si powders had been explored. Spheroidisation is interpreted in terms of Raleigh's instability, Marangoni convection, laser absorptivity, heat conductivity, fluidity of the melt volume and the powder's oxide content. Balling occurred only in the single layer parts due to the lower thermal conductivity of the bed and possibly the difference in wettability of the powder bed and sintered layers. The existence of a high degree of thermal expansion mismatch between the oxide film and the parent metal and a uniform oxide layer thickness were found to favour the disruption of the oxide shell promoting inter-particulate melting across the layers. This was the case for Al-Si parts, whereas SLM of pure aluminium and pre-alloyed Al-Mg powders gave rise to high incidence of randomly distributed pores.

Materials Processing Fundamentals: Process Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Technology and Modeling Committee
Program Organizer: Prince Anyalebechi, Grand Valley State Univ

Monday PM — Room: 2016
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Prince Anyalebechi, Grand Valley State University

2:00 PM
Solidification Stresses in Steel for Continuous Casting Conditions: *Matthew Rowan*[1]; Brian Thomas[1]; Robert Pierer[2]; Christian Bernhard[2]; [1]University of Illinois; [2]University of Leoben

Measuring stress development in solidifying steel is very difficult. The submerged split cell tensile (SSCT) test can measure force developed in a cylindrical shell of steel during solidification under controlled conditions identical to continuous casting. Determining the stress profile is difficult given the nonuniform temperature and strength across the shell. A computational model of thermal-mechanical behavior during solidification is applied to simulate the SSCT test. The 2-D axisymmetric elastic-viscoplastic finite-element model features different mechanical properties for delta-ferrite and austenite that vary with temperature and strain rate. The model successfully matched measurements of 1) temperature history; 2) shell thickness; 3) solidification force; and 4) failure location. The results show the effect of carbon content on critical failure strain, and stress profiles, which depend on the phase fraction history. The SSCT test and validated model together is a powerful analysis tool of hot tear crack formation and other phenomena in continuous casting.

2:15 PM

Numerical Simulation of Continuous Casting Process of Bloom by Finite Point Method: *Seyed Ahmad Jenabali Jahromi*[1]; Mostafa Alizadeh[1]; S. Behrouz Nasihatkon[1]; [1]Shiraz University

In this paper a meshless method called Finite Point Method (FPM) is developed to simulate the solidification process of continuously cast bloom steel in both primary and secondary cooling region. The method is based on the use of a weighted least-square interpolation procedure. A transverse slice of bloom as it moves with casting speed is considered as computational domain. The two dimensional heat transfer equation together with temperature dependent thermophysical properties is solved in the computational domain. The enthalpy method is used to calculate the latent heat. The present work is verified in the mold region by the comparison of the surface temperature simulated by FPM and finite volume method (FVM) and also comparison of solidified shell thickness simulated by FPM and measured on a breakout bloom. For secondary cooling region the validation is done by comparison of the surface temperature simulated by FPM, FVM and thermovision measurements.

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Numerical Simulation of Stress Field in a Wide Slab Mold of Peritectic Steel Continuous Casting: *Min Chen*[1]; Liang Zhao[1]; Yongkuan Yao[1]; [1]Northeastern University

A numerical simulation of the stress field of the solidified shell in the mold during continuous casting of peritectic steel slab was calculated with commercial software. The results showed that the maximal stress on wide face of the solidified shell was located at the corner and near middle of the slab, and it increased with increasing the width of slab. In addition, the stress was also influenced by operating parameters such as super heat degree, drawing speed and cooling intensity of the mold, and the maximal stress value and its position changed with these parameters. For the slab with section size of 3200mm×150mm, the proper drawing speed was around 1.2m/min, with cooling intensity of 5500L/min wile the super heating rate was 15°C to 25°C in order to prevent longitudinal crack happening during peritectic steel continuous casting.

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Modeling the Effects of Processing Variables in Simple Castings of Actinide Metal: *Paula Crawford*[1]; Deniece Korzekwa[1]; [1]Los Alamos National Lab

The application of modern computer modeling and simulation techniques to materials processing can aid in providing valuable insight into the effects of process variables and mold design variations on the final casting product. A computer model was developed to simulate the casting process using a multi-physics computational modeling package. The effects of mold design variations on the solidification rates and final product phase stability are evaluated through modeling. Additional sensitivity analysis of various processing variables provides an indication of the process controlling variables. Combining the results of the simulation with the experimental casting results should provide a better understanding of the effect of process variables on the casting of plutonium metal.

3:00 PM

Simulation of Microstructure Evolution during Solidification of Magnesium Alloys: *Hebi Yin*[1]; Sergio Felicelli[2]; [1]CAVS, Mississippi State University; [2]Department of Mechanical Engineering, Mississippi State University

A coupled cellular automaton(CA) - finite element(FE) model was developed to calculate the growth of dendrites during the solidification of cubic and hexagonal metals. The model solves the conservation equations of mass, energy and solutes in order to calculate the temperature field, solute concentration and the growth morphology of dendrites, including the grain structure and the dendritic microstructure. Validation of the model was performed by comparing the simulation results with experimental data from previously published works, showing qualitatively good agreement in the dendritic morphology. Application to magnesium alloy AZ91 illustrates the difficulty of modeling dendrite growth in hexagonal systems, observed as deviations in growth direction caused by mesh-induced anisotropy. The model was applied to the simulation of small specimens with single- and multiple- equiaxed grain growths and columnar grain growth in directional solidification. The influence of cooling rate and some kinetics parameters on the grain morphology are also discussed.

3:15 PM

Experimental and Numerical Modelling of the Flow Field in an Industrial Bronze Caster – Improving the Numerical Model: *Sven Eck*[1]; James Evans[2]; Abdellah Kharicha[1]; [1]University of Leoben; [2]University of California Berkeley

In previous work the influence of the casting speed on the flow field and the shape of the solidification front in an industrial 0.82x0.25x0.8m³ bronze caster had been investigated. Both numerical and experimental model represented 1:1 the real caster geometry. A comparison of the results of both numerical and experimental models for the flow during the casting process showed a good agreement in the qualitative velocity fields(flow direction and vortex formation). This work represents a parameter study of the numerical model with variations of the turbulence conditions at the inlet in order to clarify their influence on the flow field in the caster. In order to find the best boundary condition at the inlet, the numerical calculations for water have been compared with the measured flow fields in the water model. The new boundary conditions were then applied to the numerical model of the bronze casting process.

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Advances on Multiscale Design of Deformation Processes for the Control of Material Properties: Nicholas Zabaras[1]; *Babak Kouchmeshky*[1]; [1]Cornell Univ

We will review advances on the development of a robust design methodology for achieving desired distribution of macro-scale properties in polycrystal plasticity problems during metal forming processes. The polycrystal is represented by an orientation distribution function using the Rodrigues parameterization. Using this continuum representation of texture the underlying texture is allowed to evolve during the process. An updated Lagrangian framework is used in modeling the finite deformation processes. A multi-scale sensitivity analysis is utilized for calculating the sensitivity of the macro-scale properties with respect to the perturbation in process parameters. The multi-scale sensitivity analysis is used in a gradient optimization framework for achieving the desired distribution of the macro-scale properties. The effectiveness of the methodology is shown through controlling properties such as ductility and hardness of the product in a metal forming process. Process conditions (e.g. forging velocity, die and performs) and initial texture are used as the design parameters.

4:00 PM

Simulation of the Filling of Moulds by the Method FEM/CV in Techniques RTM: Jamal Samir[1]; *Hattabi Mohamed*[1]; [1]ENSEM

In the course of this study, the simulation of the resin flow in the RTM process is developed by the control volume finite element method (CVFEM) coupled with the equation of the free surface location. This location is made by means of the so called "Volume of Fluid" methods or VOF. Thus, the position of the flow front, the time-lapse and the rate of the non saturated zone are calculated at every step. Our results will be compared with the experimental and analytical models in the literature. On the whole, our study is concerned with the simulation of the thermally insulated filling of moulds in RTM process while adopting the CVFEM and VOF method, taking into account the presence of obstacles, coupled with the thickness variation effect and the reinforcement coats.

4:15 PM

A Comparison of Gas and Low Pressure Carburization of 9310 and 8620 Steels - A Numerical Simulation Study: Gang Wang[1]; Mohammed Maniruzzaman[1]; *Richard Sisson*[1]; [1]Worcester Polytechnic Institute

A comparative study of gas carburization and low pressure carburization processes has been performed using CHTE's numerical simulation software – CarbTool. CarbTool is a 1-D carbon diffusion model developed based on the thermodynamics and kinetics of the carburization process. The model is capable of simulating the complex boost-diffuse processes used in the industry. The output of CarbTool is the carbon concentration distribution inside the part. Two steels- 9310 and 8620 are investigated in this study. The quenching process of carburized rods is simulated using DANTE/ABAQUS FEM software. Results are compared in terms of carbon profile, microstructures and residual stresses.

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A Diffusion Model for the Prediction of a-Case Depth during Heat Treatment of Ti-Alloys: Stephen Brown[1]; *Daniel Clark*[2]; Steven Tuppen[2]; [1]Swansea University; [2]Rolls-Royce Plc

A Fickian model of oxygen diffusion in alpha-beta titanium alloys has been developed. It is well known that the diffusion of oxygen into the surface of Ti-alloys gives rise to a potentially harmful outer layer of a-phase. The validated

computer model of diffusion can be used to predict likely penetration depths of alpha-case for different times and temperatures. This work was carried out specifically to provide information on likely a-case depths that might be expected for various heat-treatments. Small amounts of oxygen within the vacuum heat treatment chambers are considered. The code has been compared to existing alpha-case experimental data and presentation of simulated results in convenient design diagram format is described. Efficiency in definition of effect can determine economics and process performance. While established codes have general predictors for given temperatures and cooling rates these do not currently allow discrimination due to the effects of diffusion with microstructural variation.

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Numerical Simulation of Welding Arc and Weld Shape Variations under Helium Shielded GTA Welding: *Dong Wenchao*[1]; Lu Shanping[1]; Li Dianzhong[1]; Li Yiyi[1]; [1]Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences

A numerical modeling of welding arc and weld pool is established for moving helium shielded GTA welding to investigate the effect of surface active element oxygen on the weld shapes. For different oxygen content from 20 to 200ppm, the simulation results showed that the weld shape is decided by the pool flow patterns, which are driven by the surface tension, gas shear force, electromagneric force and the buoyancy force. The surface tension induced Marangoni convection plays an important role on the weld shape. Small addition of oxygen can change the Marangoni convection from outward to inward direction on liquid pool, and make the wide shallow weld shape become narrow deep one. The weld D/W ratio under high oxygen content decreases with increasing welding speed, but it is not sensitive to the welding speed under low oxygen content. The experimental results agree well with the predicted results by numerical simulation.

Mechanical Behavior of Nanostructured Materials: Nanostructures by Severe Plastic Deformation

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Xinghang Zhang, Texas A & M University; Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Monday PM — Room: 3024
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Yuntian Zhu, North Carolina State University; Michael Zehetbauer , University of Vienna

2:00 PM Invited

Using ECAP for the Processing of Magnesium and Other Similar Difficult-to-Work Alloys: Roberto Figueiredo[1]; *Terence Langdon*[1]; [1]University of Southern California

Magnesium and other similar difficult-to-work alloys, such as titanium, often present challenges for successful processing by equal-channel angular pressing (ECAP). This presentation examines these difficulties and discusses possible solutions. Examples are presented showing the exceptional properties that may achieved through the successful pressing of magnesium-based alloys.

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Strategies for Improving the Ductility of Nanostructure/Ultrafine-Grained Metals without Sacrificing Strength: Yonghao Zhao[1]; Enrique Lavernia[1]; *Yuntian Zhu*[2]; [1]University Of California, Davis; [2]North Carolina State Univ

The strength and ductility of structural materials are frequently inversely related. In other words, high strength is often accompanied by low ductility, and vice versa. Inspection of the scientific literature reveals that this is also the case for nanostructured and ultrafine-grained materials, which are usually strong, but with accompanying low ductility levels. Conventional approaches to improve ductility often yield a loss of strength. This talk presents several approaches that can be effectively implemented to increase the ductility of nanostrutured/ultrafine-grained materials while simultaneously improving or at least maintaining their strength. The fundamental mechanisms that underlie the proposed approaches are discussed in this talk.

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Correlations between Texture and Mechanical Properties of Mg and Cu Processed by High-Pressure Torsion: *Bartlomiej Bonarski*[1]; Nariman Enikeev[2]; Erhard Schafler[1]; Bernhard Mingler[1]; Borys Mikulowski[3]; Ruslan Valiev[2]; Michael Zehetbauer[1]; [1]University of Vienna; [2]UFA State Aviation Technical University; [3]AGH - University of Science and Technology, Cracow

Ultrafine grained and nanocrystalline metals processed by Severe Plastic Deformation (SPD) reveal outstanding mechanical properties, especially a considerable ductility at still enhanced strength. While the latter can be quantitatively associated to the grain refinement, the ductility is still under dispute. This work aims at clarification by investigating both the texture evolution and the hardening characteristics during and after HPT processing of Mg and Cu. Deformations by HPT (High Pressure Torsion) have been achieved up to shear strains g = 120 with hydrostatic pressures between 1 and 6 GPa. While HPT processed Cu exhibits marked shear textures with only little influence of hydrostatic pressure, that of Mg shows massive recrystallization which even ceases the further evolution of shear textures and of grain refinement. The extent and the nature of recrystallization depend on the prestrain, the hydrostatic pressure and the alloy content, which has a direct influence to the macroscopic mechanical properties.

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Mechanical Properties of Ultrafine-Grained Cu with Bimodal Grain Size Distribution: *Yonghao Zhao*[1]; Troy Topping[1]; Ying Li[1]; Ruslan Valiev[2]; Yuntian Zhu[3]; Enrique Lavernia[1]; [1]University of California at Davis; [2]Institute of Physics of Advanced Materials, Ufa State Aviation Technical University; [3]Department of Materials Science and Engineering, North Carolina State University

A bimodal grain size distribution was investigated as an effective strategy to enhance the ductility of nanostructured materials. In this work, we studied the influence of a bimodal grain size distribution on the mechanical properties of ultrafine grained (UFG) Cu. The UFG Cu samples were prepared by equal-channel angular pressing, Bc, route by 2 and 16 passes, respectively. The samples were then were annealed at 250 C for different times to attain a bimodal grain size distribution. We found with increasing annealing time (fraction of coarse grains), the yield strength values of both samples decreases, and the ductility increases gradually. The microstructural origin of the observed behavior is discussed in detail in the lecture.

3:05 PM Invited

Ultrahigh Tensile Ductility and High Strength in Nickel via Cryo-Milling and Quasi-Isostatic Forging: Yonghao Zhao[1]; Troy Topping[1]; Ying Li[1]; John Bingert[2]; A.M. Dangelewicz[2]; Peiling Sun[3]; Yuntian Zhu[4]; Yizhang Zhou[1]; *Enrique Lavernia*[1]; [1]University of California at Davis; [2]Los Alamos National Laboratory; [3]Department of Materials Science and Engineering, Feng Chia University; [4]Department of Materials Science and Engineering, North Carolina State University

The limited ductility of nanocrystalline/ultrafine-grained materials has emerged as a singular issue in the study and application of this novel class of materials. Numerous investigators have addressed this topic, with varying degrees of success, via a variety of approaches, most of which can be grouped into two general categories: microstructural design and introduction of alternative deformation mechanisms. In this talk, results are reported obtained with fine-grained Ni and bimodal grained Ni (mixture of fine grains and coarse grains) prepared by cryo-milling and subsequent quasi-isostatic forging (formerly known as Ceracon forging). In tension, the fine-grained Ni shows remarkable strength and ductility, yielding at 470 MPa with 42% elongation to failure. The bimodal Ni shows lower strength and higher ductility, yielding at 310 MPa with 49% elongation to failure. In contrast, the coarse-grained Ni, processed by annealing the fine-grained Ni at 1000 C for 10 hours, has a yield strength of 150 MPa and elongation to failure of 48%. The combination of strength and ductility of our Ni is superior to those of the nanocrystalline/ultrafined-grained Ni prepared by electrodeposition, cryo-rolling, and equal-channel angular pressing methods. The microstructural origins for such combinations of good strength and high ductility will be discussed.

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Microstructure and Mechanical Properties of Cu-Al2O3 Nanocomposites Prepared by High Energy Mechanical Milling and Thermomechanical Powder Consolidation: *Deliang Zhang*[1]; Aamir Mukhtar[1]; Charlie Kong[2]; Paul Munroe[2]; [1]University of Waikato; [2]University of New South Wales

Cu matrix nanocomposite powders with each of the powder particles consisting of a dispersion of nanometer sized (2.5-10)vol.%Al2O3 particles were synthesized by high energy mechanical milling (HEMM) of a mixture of Cu and Al2O3 powders. HEMM also results in reduction of the grain sizes of the Cu matrix to submicrometer or nanometer range. Bulk nanocomposite samples were prepared by powder consolidation of the nanocomposite powders using thermomechanical processes such as hot pressing, upset forging and extrusion. The microstructure and mechanical properties of the samples were studied as a function of the consolidation conditions, with the aim of establishing a correlation between the microstructure and mechanical properties of the materials and determining the effect of consolidation defects on mechanical properties. This paper is to present an overview of the major findings from this study and discuss the effects of various factors on the mechanical properties of Cu matrix nanocomposite.

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3:50 PM Invited

Grain Boundaries Interface Phenomena and Mechanical Properties of Ultrafine-Grained Metals: *Ruslan Valiev*[1]; [1]UFA State Aviation Technical University

In the present report using variations of regimes and routes of severe plastic deformation (SPD) processing we show for several light alloys (Al, Mg and Ti) the ability to produce ultrafine-grained (UFG) materials with different grain boundaries, and this can have a dramatical effect on mechanical behaviour of the processed materials, particular, on their strength and ductility, fatigue or superplasticity. We demonstrate several examples of this approach for attaining superior strength and ductility as well as enhanced superplasticity at low temperatures and high strain rates in various UFG metals and alloys. The origin of these phenomena is discussed on the basis of the results of microstructural studies and observations of deformation mechanisms. Special emphasis is laid on the innovation potential and first applications of SPD-produced nanometals.

4:10 PM

Simulation of Stress-Strain Characteristics and Microstructural Evolution of SPD Processed Nanomaterials: *Michael Zehetbauer*[1]; Nariman Enikeev[2]; Christian Holzleithner[1]; [1]University of Vienna; [2]Ufa State Aviation Technical University

The composite model by Zehetbauer has proven to be successful in describing the hardening characteristics and specific dislocation densities of nanomaterials during and after SPD processing. For both cases, the model could be extended for correct simulation of the grain size and of the grain boundary thickness. Recently, at the example of Cu being processed by High Pressure Torsion (HPT), efforts were made to predict also the mean misorientation as a function of accumulated strain and applied hydrostatic pressure. The results fit best to the experimental data when a linear array of dislocation in the grain boundary is assumed. Differences left between simulation and experiment may be attributed to the failing of X-ray profile analysis which detects too small dislocation densities when the dislocation distance in the grain boundary becomes smaller than 1 nm.

4:25 PM Invited

Development of Shear Drawing Process for the Spheroidization of Medium and High Carbon Steels: Industrial Application of ECAP: *Dong Shin*[1]; Hyun H. Cho[1]; Young G. Ko[2]; Il-Heon Son[3]; KiHo Rhee[3]; Duklak Lee[3]; [1]Dept. of Mater. Sci. & Eng.,Hanyang University; [2]Dept. of Mater. Sci. & Eng.,MIT; [3]Technical Research Laboratory,POSCO

Severe plastic deformation of metals utilizing ECAP has been recognized as one of the attractive methods for tailoring the ultrafine-grained microstructures which exhibit better performance in mechanical properties than those of the traditional metals where the deformation during ECAP is simply shear in nature. Particularly, with regard to obtaining good formability, this feature is also beneficial for metals to break down lamellar structure in steel through the decomposition of pearlitic cementite phase. Therefore, it can be put to use for the industrial applications of steel-wire manufacturing when problems causing from the batch processing will be solved. The aim of this study is to propose the new-typed ECAP termed shear drawing (SD) by modifying the design of the die in order to avoid dimensional inhomogeneity. Moreover, the deformation characteristics and the microstructural changes associated with the spheroidization of carbon steel during SD are discussed and compared to results obtained by ECAP.

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Molecular Dynamics Simulations of Dislocation Activity in Single Crystal and Nanocrystalline Copper Doped with Antimony: Rahul Rajgarhia[1]; *Douglas Spearot*[1]; Ashok Saxena[1]; [1]University of Arkansas

Recently published simulation results have indicated that high temperature grain growth in nanocrystalline copper can be suppressed by introducing dopant atoms at the grain boundaries [Millett et al., 2006]; however, the impact of grain boundary dopants on plastic behavior is still unclear. In this work, molecular dynamics simulations are used to study dislocation activity in single crystal and nanocrystalline copper with low concentrations of antimony (0.0-2.0 at.%Sb). A new interatomic potential for Cu-Sb is developed in this work and used to model the dopant/host interatomic interactions. In single crystal models, it is observed that the strained regions around the Sb atoms act as sources for partial dislocations and that the dislocation nucleation stress decreases with increasing concentration of antimony. In nanocrystalline models, antimony atoms randomly dispersed along the grain boundaries alter the stress-strain response and subsequently the grain diameter at which the maximum strength is observed (modified inverse Hall-Petch response).

5:00 PM

The Influence of Temporary Hydrogenation on ECAP Formability and Low Cycle Fatigue Life of CP Titanium: Andrew Czerwinski[1]; *Rimma Lapovok*[1]; Dacian Tomus[1]; Yuri Estrin[1]; [1]Monash University

Titanium has been successfully competing with other metallic materials due to its high strength-to-density ratio. UFG titanium processed by ECAP can be made 30-120% stronger than conventional structural steel. However, the increase in tensile strength is often accompanied with decrease in the low cycle fatigue (LCF) life. The new opportunities to improve the LCF life come from the use of hydrogen as a temporary alloying element, which has been shown to enhance the properties of titanium. In particular, for some Ti alloys hydrogen induced ductility is in the low temperatures range, which is directly correlated with improved formability by ECAP and increased LCF life. Typically damage initiates from the surface, and, therefore, hydrogenation and the associated enhancement of the surface properties are beneficial. The effect of hydrogenation on the improvement of ductility, formability by ECAP, and enhancement of LCF life of ultrafine grained titanium has been investigated.

5:15 PM

Mechanical Behavior during Tensile Straining of Nano/Ultrafine-Grained Structures Formed by Reversion in Metastable Austenitic Steels: *Sashank Nayak*[1]; Sachin Mali[1]; Devesh Misra[1]; Mahesh Somani[2]; Pentti Karjalainen[2]; [1]University of Louisiana; [2]University of Oulu

The deformation behavior of nano/ultrafine-grained structures during tensile deformation has been examined by transmission electron microscopy in metastable austenitic steels. Special fine-grained structures were obtained by controlled reversion annealing of strain-induced martensite. Proper gradual strain hardening by the formation of ultra-fine martensite results in excellent tensile strength-ductility property combination. Twinning and dislocation glide were identified as the primary deformation mechanisms in nano-/ultrafine-grained structures.

5:30 PM

Grain Refinement and Mechanical Properties in Nanostructured Al and Al–Mg Alloys Subjected to Severe Plastic Deformation: *Hans Roven*[1]; Manping Liu[1]; Maxim Murashkin[2]; Ruslan Valiev[2]; Tamas Ungár[3]; Levente Balogh[3]; [1]Norwegian University of Science and Technology (NTNU); [2]Ufa State Aviation Technical University; [3]Eötvös University

Bulk nanostructured materials can be produced by a variety of severe plastic deformation methods. The present work focuses on commercial purity Al and Al–Mg alloys subjected to high pressure torsion. The grain sizes are in the range 10–200 nm with typical average values ranging from 46 to 120 nm. The hardness and strength values as well as the dislocation densities increased, whereas, the average grain size decreased significantly with increasing Mg contents. The local dislocation densities in grain boundary and triple junction areas are two to three orders of magnitude larger than the average values. Extensive high-resolution

transmission electron microscopy observations reveal that these materials develop new nanostructures such as deformation nano-twins, stacking faults and non-equilibrium grain boundaries. The purpose of this work is to explore the impact of these nanostructures as well as Mg contents on grain refinement mechanism and mechanical behavior in these materials.

5:45 PM

Effect of Microstructure on the Tensile Behavior of Ultrafine-Grained Cu: *Pei-Ling Sun*[1]; Chia-Hao Yang[1]; Chung-Yi Yu[2]; Yun-Jun Wang[3]; Po-We Kao[3]; Chih-Pu Chang[3]; [1]Feng Chia University; [2]China Steel Corporation; [3]National Sun Yat-Sen University

Pure Cu with ultrafine-grained (UFG) structure was produced by equal channel angular extrusion (ECAE) to an equivalent strain of ~8. Different UFG structures were obtained by performing ECAE either at ambient temperature (sample A) or at 373 K (sample B). Additionally, cryo-rolling was also applied to an ECAEed specimen in order to create an UFG structure with higher dislocation density and higher fraction of high angle boundaries (sample C). Low temperature annealing (<423 K) treatments were applied to introduce various amount of microcrystalline grains into these three different UFG matrices via partial recrystallization. The microstructures of these samples were characterized by TEM and SEM. Tensile properties were measured at room temperature. The influence of the microstructure of the UFG matrix as well as of the size and amount of microcrystalline grains on the tensile properties will be presented.

6:00 PM

The Effect of Milling Media and Time on Cryomilled 99.95% Pure Al: *Troy Topping*[1]; Chris San Marchi[2]; Ying Li[1]; Zhihui Zhang[1]; Rustin Vogt[1]; Osman Ertorer[1]; Julie Schoenung[1]; Richard Karnesky[2]; Nancy Yang[2]; Enrique Lavernia[1]; [1]University of California, Davis; [2]Sandia National Laboratories

Aluminum powder (99.95% pure) was cryomilled in separate batches in liquid argon (LAr) and liquid nitrogen (LN_2) to obtain a nanocrystalline structure. Samples were removed from the milling vessel every 4 hours to characterize the powders over a 24 hour period. Transmission electron microscopy (TEM) and x-ray diffraction (XRD) show differences in microstructural evolution during milling for the two different media. After cryomilling, powders removed after 12 and 24 hours for the LAr and 24 hours for the LN_2 were hot vacuum degassed and consolidated via hot isostatic pressing (HIP). After HIPping, bars were extruded for tensile testing. The extrusions were analyzed for interstitial elemental composition to determine the uptake of nitrogen in all cases, since it is believed to contribute to the mechanical behavior of the consolidated aluminum. High resolution TEM is also employed to further investigate the microstructural contributions to the properties of the respective materials.

Microstructural Processes in Irradiated Materials: Radiation Effects II: Advanced Characterization and Fe-Cr Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee

Program Organizers: Christophe Domain, Electricite De France; Gary Was, University of Michigan; Brian Wirth, University of California, Berkeley

Monday PM — Room: 2008
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Pascal Bellon, University of Illinois; Vasily Bulatov, Lawrence Livermore National Laboratory

2:00 PM Invited

State-of-the-Art Positron Annihilation Techniques to Study Embedded Nano-Clusters in Metals: *Takeshi Toyama*[1]; Yasuyoshi Nagai[1]; Koji Inoue[1]; Zheng Tang[2]; Masayuki Hasegawa[1]; Abderrahim Abderrahim[3]; Eric van Walle[3]; Robert Gerard[4]; [1]Tohoku University; [2]East China Normal University; [3]SCK-CEN; [4]Tractebel Engineering

We have developed a "positron quantum-dot" method for embedded nano-clusters in metals using Coincidence Doppler Broadening (CDB) of positron annihilation. It features self-searching and gives exclusive information of atomic and electronic structures of the embedded clusters. It extends its applicability of positron annihilation other than that in the research field of vacancy-type defects and has been successfully applied to studies of Cu nano-clusters in Fe-Cu model alloys and surveillance test specimens of nuclear Reactor Pressure Vessel (RPV) steels. Furthermore recently we developed new experimental techniques for the positron quantum-dots, (i) two dimensional angular correlation of annihilation radiation (2D-ACAR), and (ii) Age MOmentum Correlation (AMOC) between positron lifetime and Doppler broadening. From smearing of 2D-ACAR momentum distributions we can estimate sizes of Cu nano-clusters and from AMOC spectra we can obtain the chemistry and number densities of the nano-clusters. Thus we clarify detailed evolution of the nano-clusters in Fe matrix.

2:30 PM Invited

Imaging and Spectroscopy of Single Atoms and Point Defects through Aberration-Corrected STEM: *Stephen Pennycook*[1]; M. Varela[2]; A. R. Lupini[2]; A. Y. Borisevich[2]; W. Luo[1]; S.-H. Oh[3]; K. Van Benthem[4]; S. Rashkeev[5]; K. Griffin Roberts[6]; K. M. Krishnan[6]; J. García-Barriocanal[7]; C. Leon[7]; J. Santamaría[7]; S. T. Pantelides[1]; [1]Oak Ridge National Laboratory - and - Vanderbilt University; [2]Oak Ridge National Laboratory; [3]Korea Basic Science Institute; [4]University of California, Davis; [5]Idaho National Laboratory; [6]University of Washington; [7]GFMC, Universidad Complutense de Madrid

In recent years the probe size in the scanning transmission electron microscope has decreased more than a factor of two, bringing improved resolution both laterally and vertically. In addition, the smaller, sharper probe provides greatly increased signal to background ratio, enabling the study of single atoms inside materials for the first time. In Si nanowires individual Au interstitials are seen that match well in both atomic position and relative formation energies with density functional calculations. Single atom sensitivity has also been achieved in electron energy loss spectroscopy, which can not only positively identify the element, but also, from the fine structure, provide information on local bonding, band structure and carrier concentration. Several examples will be shown of oxide materials and heterostructures where spectroscopic analysis reveals the origin of surprising bulk properties including ferromagnetism in Co-doped anatase and collosal ionic conductivity in Y-stabilized ZrO2/SrTiO3 heterostructures. This research was sponsored by the Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, US Department of Energy and NSF/ECS No. 0224138.

3:00 PM

JANNUS: A New Multi-Ion Irradiation Facility to Study the Stability of Nano-Oxides in ODS Materials: *Ribis Joël*[1]; de Carlan Yann[1]; Boulanger Loic[1]; Serruys Yves[1]; Trocellier Patrick[1]; [1]CEA

Ferritic ODS alloys are primary candidates as long life cladding materials for the Sodium Fast Reactor (SFR) developed in France in the frame of Generation IV research program. These alloys exhibit a fairly high resistance to swelling under neutron irradiation and the embedded oxide dispersion helps to preserve good mechanical properties at high temperature. Previous experimental studies have shown a partial dissolution of some oxides under neutron irradiation. However, a relative stability of the nano-oxide particles has been reported under ion irradiation experiments even at a damage level up to 200 dpa.[1] Then, the behaviour of ODS alloys under ion irradiation at high damage dose has to be clarified using multi-ion irradiations. The new multi-ion irradiation facility JANNUS which is in completion at CEA Saclay and CNRS Orsay will be described in details and the future irradiation experiments on ODS alloys will be discussed. [1]T.R. Allen et al., J. Nucl. Mater. 375, 2008, pp. 26-37.

3:20 PM

Effect of Grain Size on the Radiation Resistance of Materials: *Steven Zinkle*[1]; [1]Oak Ridge National Laboratory

Creation of a high density of point defect sinks in the form of nanoscale grain boundary interfaces offers a potentially attractive solution to the problem of developing radiation –resistant materials for fusion and Generation-IV fission energy systems, where the structural materials may be exposed to unprecedented high displacement damage levels. This presentation summarizes experimental observations on several materials that demonstrate the effectiveness of small grain sizes on inhibiting defect cluster nucleation and growth during irradiation. Whereas nanoscale grain dimensions may be very effective in improving radiation damage resistance, grain dimensions on the order of 100-5000 nm may under some conditions produce an acceleration of radiation damage. This enhanced retention of radiation damage can occur for example if the grain size is intermediate between the mean diffusion lengths for vacancy and interstitial type defects. Several caveats to using fine-grained materials will be noted, including the potential for radiation-enhanced grain coarsening.

3:40 PM Break

4:00 PM Invited
Density Functional Based Modeling of FeCr Alloys: *Par Olsson*[1]; [1]EDF R&D

FeCr alloys have garnered renewed interest during the last years, due to the foreseen use of Ferritic/Martensitic steels of high Cr content in future nuclear installations. Especially interesting as a model system is the binary FeCr since the Cr content has a major influence on the evolution of the mechanical properties in irradiated Ferritic/Martensitic alloys. Experimentally, these alloys have been studied for over 40 years, but for a long time the understanding of many observed effects was lacking. Already 20 years ago calculations appeared that indicated the relevance of the concentration, in terms of the inversion of short range ordering around 10% Cr. However, these studies were not extensively followed up until recently. Here we will discuss the latest developments in the modeling, based on density functional theory, of thermodynamics and defect properties in FeCr alloys and how the understanding of the microscopic origins of macroscopic effects is evolving.

4:30 PM
Heavy-Ion Irradiation Damage in Fe-Cr Alloys: *Sen Xu*[1]; Zhongwen Yao[1]; Mike Jenkins[1]; [1]University of Oxford

Bulk Fe-Cr specimens (of thickness about 100 µm and with Cr contents ranging from 0-12%) were irradiated with dual-energy iron ions (0.5 and 2 MeV) at Surrey Ion Beam Centre at temperatures of 300°C and 500°C to doses up to 7dpa. TEM experiments showed that the radiation damage took the form of dislocation loops of interstitial nature with sizes up to 100nm. The average loop size was about four times larger at the higher irradiation temperature. In Fe-Cr alloys, both b = <100> and b = ½<111> loops were present in roughly equal proportions at both temperatures. However, in the pure Fe, b = ½<111> loops predominated at 300°C whilst b = <100> loops predominated at 500°C. Comparisons of this work with parallel in-situ experiments in which ion irradiations were performed on TEM specimens in the form of thin foils will also be reported.

4:50 PM
Cluster Dynamics Modeling of Microstructural Evolution of Ferritic Martensitic Steels under High Energy Ion Irradiation: *Donghua Xu*[1]; Francois Gallet[1]; Brian Wirth[1]; [1]University of California, Berkeley

The design goals of higher temperatures, higher radiation doses and higher energy neutron spectrum for future fission and fusion reactors demand new advanced materials with superior resistance to irradiation damages. Typical irradiation damages such as hardening, embrittlement, swelling, creep, precipitation etc., are all closely related to the generation and clustering of point and small fragmented defects, the fundamental reason for microstructural changes and subsequent mechanical property degradation under irradiation. Here we present our rate theory based modeling of cluster evolution in both compositional and geometric spaces under high energy ion irradiation. Particularly, we calculate the concentrations of interstitial loops and voids as a function of multiple variables including time, number of interstitials/vacancies, spatial position, dislocation densities, temperature, dose and dose rate, impurities and so on. Our calculations are compared with coordinated in-situ transmission electron microscopy irradiation studies performed by others.

5:10 PM
Microstructure Evolution of Fe-9Cr-0.1C and Fe-12Cr-0.1C Model Martensitic Steels Ion-Irradiated In-Situ in a TEM: *Djamel Kaoumi*[1]; Arthur Motta[1]; Mark Kirk[2]; [1]The Pennsylvania State University; [2]Argonne National Laboratory

Two model martensitic steels of compositions Fe9Cr0.1C and Fe12Cr0.1C are irradiated with 1 MeV Kr ions at 400°C to doses of 10 dpa in-situ in a TEM. These model materials have been processed to exhibit lath microstructures similar to those found in commercial steels. The microstructure evolution under irradiation –black dot density, nature and number density of defect clusters present,and stability of as-fabricated microstructure – is followed and characterized using weak-beam dark-field imaging and g.b analysis. The goal of the irradiations is to perform a direct determination of the spatial correlation of the time evolution of the irradiation-induced defect structures with the pre-existing alloy microstructure including lath boundaries, network dislocations and carbides, for comparison with computations. These results will be summarized in this paper.

5:30 PM
In-Situ Ion Irradiations of Iron-Chromium Alloys: *Carolyn Tomchik*[1]; Mark Kirk[2]; Maria Okuniewski[3]; James Stubbins[1]; Stuart Maloy[4]; [1]University of Illinois at Urbana-Champaign; [2]Argonne National Laboratory; [3]Idaho National Laboratory; [4]Los Alamos National Laboratory

Iron-chromium alloys are used as a model to study the microstructural evolution of defects in irradiated structural steel components of a nuclear reactor. We examine the effects of temperature and chromium concentration on the defect evolution, segregation behaviour, and second phase precipitation in the early stages of damage. In-situ irradiations are conducted at the IVEM-Tandem facility at Argonne National Laboratory at 300°C, 450°C, and 550°C with 150keV Fe ions in single crystal Fe14Cr and Fe19Cr bicrystal to doses of 2E15 ions/cm^2. The microstructures of the irradiated iron-chromium alloys are characterized by analysis of TEM micrographs and compared with those of pure iron.

Nanocomposite Materials: Polymer Nanocomposites

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee, TMS: Nanomaterials Committee

Program Organizers: Jonathan Spowart, US Air Force; Judy Schneider, Mississippi State University; Bhaskar Majumdar, New Mexico Tech; Benji Maruyama, Air Force Research Laboratory

Monday PM — Room: 3020
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Karen Winey, University of Pennsylvania; Judy Schneider, Mississippi State Univ

2:00 PM Introductory Comments

2:05 PM Invited
Inside Polymer Nanocomposites – Interphases, Gradients and Percolation: *Catherine Brinson*[1]; Rui Qiao[1]; Supinda Watcharotone[1]; [1]Northwestern University

Polymers with small loadings of nanoparticles exhibit dramatic changes in thermomechanical properties, in large part due to the development of interphase zones of altered polymer properties near the interfaces. Given the enormous surface-to-volume ratio for nanoparticles, the interphase volume fraction can dwarf that of the inclusions and percolate through the composite. Experimental evidence of the existence of this interphase region is presented for several nanofiller types via local and global glass transition changes and microscopy. By properly controlled functionalization of the nanoscale inclusions, we can impact the properties of the interphase region and the nanocomposites. The nature of the properties of this interphase region and the gradient in properties away from the nanofiller is probed by novel nano-DMA experiments on model nanocomposite systems. In conjunction with the experimental results, the concept of percolated interphase is investigated by a finite element approach which accounts for an interphase region of gradient properties.

2:30 PM
In Situ FTIR Studies on Double Wall Carbon Nanotube Composites of High Volume Percent: *Scott Brownlow*[1]; Alexander Morvasky[2]; Nikolai Kalugin[1]; Bhaskar Majumdar[1]; [1]New Mexico Tech; [2]MER Corporation

While carbon nanotube (CNT) based polymer composites have drawn attention in recent years, the potential has been limited by the low volume fraction and poor load transfer to the nanotubes. Here, we have utilized functionalized mats of double walled nanotubes (DWNT) to develop strong epoxy based composites with 10 - 13 weight percent DWNT. In addition, we utilized the FTIR technique using in situ loaded specimens to monitor the changes in vibrations, with the goal of understanding load transfer behavior. Tests with neat epoxy and composite samples show that the stretching of epoxy bonds is greatly reduced in the composite at equal stress. We will attempt to show that in situ FTIR using current instruments and analysis procedures provides a means to tap this understanding and improve CNT composites. Our work is complemented with in situ Raman studies of the embedded DWNT that also shed insight on load sharing behavior.

2:50 PM
Magnet-Polymer Nanocomposites for Shape Changing Structures: *Raju Ramanujan*[1]; V Nguyen[1]; [1]Nanyang Technological University

There is an urgent need for intelligent shape changing structures for energy, biomedical and structural health monitoring applications. However, current materials either have low strains or are too slow for many practical applications. MAGPOL, a composite containing MAGnetic nanoparticles in a soft POLymer matrix is capable of large deformation and fast response, hence is very attractive for novel shape changing structures. An external magnetic field can be used to apply forces on the particles, leading to fast shape change of the composite in contraction, elongation, deflection and torsion modes. The extent of shape deformation as a function of magnetic field strength was found to be highly nonlinear, large stress and strain was achieved with MAGPOL. MAGPOL can also act as a sensor, the resistance of composites at percolation concentration is a strong function of the actuation strain. Novel nanocomposite systems can be built by exploiting this unique combination of properties.

3:10 PM
Nucleation and Growth of Hierarchical Structures and Phases in Pressure-Induced Crystallization of Polymer Nanocomposites: *Qiang Yuan*[1]; Devesh Misra[1]; [1]University of Louisiana

The objective of the presentation is to elucidate the basic physical mechanisms underlying the evolution of hierarchical structures and phases during pressure-induced crystallization of polymers containing dispersion of nanoparticles. The phase selection in the polymers is normally dictated by pressure and temperature, however, the introduction of nanoparticles can dramatically alter the kinetics of the formation of the phases via nanoparticle interface driven nucleation. Thus, by controlling pressure and crystallization temperature, a high degree of phase selection and structural control may be achievable, which has profound effect on mechanical properties.

3:30 PM Break

3:45 PM Invited
Electrical Conductivity and Polymer Diffusion in Polymer Nanocomposites: *Karen Winey*[1]; [1]University of Pennsylvania

Both electrical conductivity and polymer diffusion in polymer nanocomposites are dominated by the presence of a nanotube network. This talk will present three-dimensional simulations of electrical conductivity above the filler percolation threshold for composites containing conductive, finite-sized rods with various orientations. The random resistor network model was used to calculate the electrical conductivity of these simulated composites. The observed trends compare favorably with our earlier experimental results. In addition, polymer tracer diffusion in single wall carbon nanotube / polymer nanocomposites is reported. Polymer diffusion is suppressed at low SWCNT loadings to a surprising extent and recovers at higher loadings. A new phenomenological trap model is used to discuss these results. This fundamental study provides insight to polymer dynamics in nanostructured environments and furthers the understanding of melt processing in polymers with nanoscale, high-aspect ratio fillers that form networks.

4:10 PM
Solvent Studies on Submicron-Sized Aluminum/Epoxy Composites: *S. Johnson*[1]; Jared Boock[1]; Christopher Crouse[2]; C. Michael Lindsay[1]; Jennifer Jordan[1]; Jonathan Spowart[2]; [1]Air Force Research Laboratory, Munitions Directorate, Eglin Air Force Base; [2]Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson Air Force Base

Composite materials processing often employs binders of polymeric epoxy systems that are highly viscous at lower temperatures. One processing technique utilized to reduce the viscosity of the polymer matrix is that of using solvents to facilitate incorporation of higher percentages of solids into the epoxy binder. This processing technique has been widely employed in the preparation of many types of composite samples without the benefit of comparing the properties of the composite materials processed both with and without solvents. Epoxy/Al composite materials obtained from commercially available epoxy resins and micron-/ nanometer-sized aluminum powders were prepared by decreasing mix viscosity with solvents of varying functional groups and volatilities. This study evaluates the impact of various solvents on the properties of the resultant composite materials. The chemical and mechanical properties of the resultant composites were evaluated and are reported.

Neutron and X-Ray Studies of Advanced Materials: Diffuse Scattering

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Titanium Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter Liaw, The University of Tennessee; Jaimie Tiley, US Air Force

Monday PM — Room: 3016
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Rozaliya Barabash, Oak Ridge National Laboratory; Patrice Turchi, Lawrence Livermore National Lab

2:00 PM Invited
X-Ray Diffuse Scattering for The Study of the Size Distributions of Clustered Defects in Crystalline Materials: *Bennett Larson*[1]; [1]Oak Ridge National Laboratory

The diffuse scattering near Bragg reflections arising from defect clusters in crystalline materials contains detailed signatures of the size, type, size-distribution, and the internal structure of clusters. In this presentation, the fundamental aspects of diffuse scattering from lattice defects will be considered within the coherent wave theory and the local-Bragg scattering interpretation in the so-called "asymptotic" diffuse scattering regime will be discussed. Detailed measurements of separate size distributions for vacancy and interstitial loops in neutron irradiated Cu and for coherent Co precipitates as a function of thermal aging in Cu(1%)Co single crystals will be presented. In addition, the use of submicron diameter x-ray microbeams to perform depth-resolved diffuse scattering measurements of vacancy and interstitial loop distributions in 10 MeV self-ion implanted silicon single crystals will be discussed. Research at ORNL supported by the DOE Office of Science, Basic Energy Sciences, Division of Materials Sciences and Engineering.

2:20 PM Invited
Huang-Scattering as a Probe of Local Defect Structure: *Branton Campbell*[1]; [1]Brigham Young University

Point-like crystal defects often give rise to anisotropic diffuse-scattering distributions that are centered around intense Bragg peaks in electron, x-ray and neutron diffraction data. These anisotropic Huang-scattering distributions are directly related to the long-range strain-fields extending outward from the defect core. By relating these strain fields to the atomic displacements within the defect core via the elastic dynamical matrix, Huang scattering can be used to indirectly probe local defect structure. We will present a convenient method of extracting local displacements from 3D Huang-scattering data that is sufficiently general to handle both arbitrary crystal symmetry and arbitrary defect symmetry. It involves first invoking the linear-continuum elastic approximation to characterize the strain fields, which are then used to extrapolate approximate local atomic displacements. In additional to exploring the strengths and limitations of this approach, we will demonstrate its application to the quantitative analysis of experimental diffuse scattering data.

2:40 PM
Statistical Theory of Diffuse Scattering by Crystals with Nonrandom Precipitates: *Rozaliya Barabash*[1]; Gene Ice[1]; [1]Oak Ridge National Lab

The statistical kinematical theory of diffuse scattering from precipitated alloys is reviewed. Nonperiodic potentials are used to model the scattering from different defect distributions. Fluctuating parts of the potential, resulting in diffuse scattering, is analyzed in the framework of the fluctuation wave method. Interaction of distortion fields from precipitates often causes their self-organization and nonrandom distribution. A comparison with the single defect approximation approach is performed for correlated/uncorrelated precipitates. Correlation and layered precipitates distribution in preferred crystallographic planes, results in specific features of the diffuse scattering intensity. The shape function of coherent precipitates is discussed. Experimental and simulated diffuse scattering intensity maps are used to perform quantitative characterization of the defects structure and distribution.Research is sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Science U.S. Department of Energy.

2:55 PM Invited

Elastic Diffuse Scattering of Binary Alloys with a Large Atomic-Size Difference: *Bernd Schönfeld*[1]; [1]ETH Zurich

Alloys with local order and a relatively large difference in atomic sizes have continued to attract interest of theoreticians and experimentalists. Here, two such alloys, Ni-Re and Ni-Pt, were investigated experimentally. Not much is known on the microstructure in Ni-Re though its fraction in Ni-based superalloys has been steadily increased. From diffuse x-ray scattering of Ni-9.4 at.% Re in combination with small-angle neutron scattering it was now established that local order is present, characterized by 1½0 diffuse maxima. For the system Ni-Pt the superstructures Ni_3Pt and NiPt are known; on the Pt-rich side, however, ordered structures are unknown. A diffuse x-ray scattering experiment of Ni-87.8 at.% Pt was undertaken to investigate the possible presence of $NiPt_7$ suggested by electronic-structure calculations. With the interaction parameters obtained, Monte-Carlo simulations indicated that no $NiPt_7$ superstructure is to be expected. Species-dependent static atomic displacements also determined will be compared with values from the literature.

3:15 PM Invited

Atomic Displacements on a Si-Ge Si Rich Single Crystal: *Jose Rodriguez*[1]; S. Moss[2]; J. Robertson[3]; Rozaliya Barabash[3]; J. Copley[4]; D. Neumann[4]; [1]University of Maryland/National Institute of Standards and Technology; [2]University of Houston; [3]Oak Ridge National Laboratory; [4]National Institute of Standards and Technology

Si-Ge alloys have received a great deal of attention because of the possibility of faster devices that might employ them. However, the lattice mismatch between the two elements has made the growth of epitaxial homogeneous thin films quiet difficult and various strategies have been employed to overcome this. Le Bolloc'h et al. reported phonon measurements on a Si-Ge Si Rich single crystal. He also distinguishes in a radial scan of the elastic neutrons from the alloy the size effect displacement scattering. Using the Disk Chopper Spectrometer (DCS) at NIST we have measured the (110) and (001) planes which clearly shows the Huang Diffuse Scattering (HDS) and Size Effect scattering (SE) due to atomic displacements. The data also shows no SRO contribution. The data permits the extraction of HDS and SE displacements parameters. Phys. Rev. B 63, 035204 (2001).

3:35 PM Invited

Structure and Dynamics of a Si-Rich SiGe Solid Solution: *J. Robertson*[1]; [1]Oak Ridge National Lab

The SiGe system has been intensively studied because the carrier mobility is much higher than in pure silicon and this gives rise to considerable gains in electronic device performance. SiGe alloys have been the subject of intense investigation for several years. Several theoretical studies which are supported by a previous experiment on a polycrystalline solid solution predict a miscibility gap in the Si-Ge system. On the contrary, several long-range ordered structures have been reported in thin films although these were under either compressive or tensile strain as well as an XAFS study on SiGe thin films that indicates complete randomness. Thus, the study of the local atomic environments in this alloy is of fundamental importance. In this talk I will present the results of neutron and X-ray measurements on a homogeneous bulk crystal of $Si_{92}Ge_{08}$ and discuss the nature of its structural disorder and dynamics.

3:55 PM Invited

Small-Angle Scattering of X-Rays and Neutrons - Advances and Challenges: *Gernot Kostorz*[1]; [1]ETH Zurich

Some applications of small-angle scattering in the field of materials science will be discussed. Emphasis will be on experiments pushing the spatial and temporal resolution limits of both tpyes of radiation, exploiting their individual characteristics and their complementarity. Inhomogeneities on a scale from a few interatomic distances to several micrometers and their evolution can be studied in the bulk and at/near surfaces with good accuracy. These experiments serve to develop and test models of micro- and nanostructural features in materials.

4:15 PM Break

4:25 PM Invited

"Small-Angle Scattering for Every Microstructure and Every Problem?" – A Critical Review of Advanced Small-Angle Scattering Techniques: *Jan Ilavsky*[1]; [1]Argonne National Laboratory

Number of small-angle scattering tools and techniques – for both X-ray or neutrons – is available today. Since some of these were developed in the last years, they may not be discussed in commonly available textbooks. Therefore many non-SAS expert researchers may not be aware of their advanced capabilities. Further, to learn these new tools and methods, one has to make use of original manuscripts, sometimes with sketchy techniques description and often-specific materials oriented. Therefore it may be difficult to generalize and apply these novel techniques to wider range of material science problems. This presentation will review different X-ray and neutron small-angle scattering techniques, such as anisotropic Porod scattering, anisotropic multiple scattering, and various types of ultra-small angle scattering. Examples of results, ranging from thermal barrier coatings to hierarchical polymer structures, will be given and applicability to assorted engineering material science problems will be discussed.

4:45 PM Invited

Chemical Order in Alloys: Current Status and Prospect: *Patrice Turchi*[1]; [1]Lawrence Livermore National Lab

The prediction of ordering trends in alloys provides valuable insight on phase formation, phase stability, and phase diagrams. In this context, neutron and X-ray scattering experiments can play a critical role in the verification and validation process, and also challenge theory. After a brief critical review of the quantum-mechanical-based approaches that are currently available for studying short-range order in alloys, examples of predictions will be discussed. They will include: transient ordering phenomenon, ordering in complex alloys such as A15 and sigma, pressure effect on chemical order, and ordering trends in some fcc and bcc-based alloys. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

5:05 PM Invited

Phase Transitions in Ionic Liquid Based Mixtures Studied by Simultaneous X-Ray Diffraction and DSC Measurement: *Hiroshi Abe*[1]; Yusuke Imai[1]; Takefumi Goto[1]; Yukihiro Yoshimura[1]; [1]National Defense Academy

Ionic liquids (ILs) are well known to be new solvents in "green chemistry". The curious nature of ILs is represented in almost zero vapor pressure. Recently, we found anomalous domain growth in $[DEME][BF_4]$-H_2O mixtures. Here, three different types of the domain structures were formed with a small content of H_4O. In spite of a variety of domain formations, crystal structures are the same as pure $[DEME][BF_4]$. In addition, boundaries between complicated domain structures could not provide intrinsic strains. Subsequently, complicated phase diagram is obtained from 0 to 12 mol% H_2O. By simultaneous X-ray diffraction and DSC measurements, various kinds of phase transitions and phases are determined systematically. One of important solid phases is pure amorphous phase around 6 mol%. In Raman spectrum, there are "two dynamic components" in pure amorphous phase. Moreover, cold crystallization with some exothermal peak is observed upon heating above 4 mol%. The results are based on anomalous behaviors of water molecules. Y. Imai *et al.*, J. Phys. Chem. B (2008), in press. Y. Imai *et al.*, Chem. Phys. (2008), in press.

5:25 PM Invited

From Average to Local Structure: A Structural Study of Zeolite-NdY/Se System:: *A. M. Milinda Abeykoon*[1]; W. Donner[2]; M Brunelli[3]; A. J. Jacobson[1]; S. C. Moss[1]; [1]University of Houston; [2]Darmstadt University of Technology; [3]European Synchrotron Radiation Facility (ESRF)

A challenging problem in crystal structure determination is the characterization of the atomic short-range order as observed in the diffuse scattering. The long-range order is characterized by a careful analysis of the sharp Bragg positions and intensities to obtain average atomic positions and equilibrium atom displacements. The short-range order appears as weak diffuse scattering widely distributed throughout the reciprocal space. Since many important crystalline materials have a significant disorder on the atomic scale, a method which is capable of modeling the structure of these materials is necessary. We emphasize the need of two techniques: the Pair Distribution Function (PDF) and the Rietveld method to model the structures of such systems. We will use our results of x-ray studies on zeolite-NdY/Se system to demonstrate this.

5:45 PM

Small Angle X-Ray Scattering Studies of the Pore Structure in Metal and Metal Oxide Foams: *Tony van Buuren*[1]; Trevor Willey[1]; Alex Hamza[1]; Ted Baumann[1]; John Kinney[1]; James Stoken[1]; Jan Ilavsky[2]; [1]Lawrence Livermore National Laboratory; [2]Advanced Photon Source

Although a great amount of study has been devoted to the physical properties of porous structures, it is not clear whether the theoretical models developed

to date can be extended to nanoscopic length scales. Our goal is to quantify the microstructure of highly porous metals and metal oxides to determine how processing of the porous material relates to the structure and ultimately to the mechanical behavior. We will quantify structural changes with a combination of small angle x-ray scattering (SAXS) and high-resolution x-ray imaging. Finite element modeling, using the structures determined above, will be used to study the effects of mechanical loading on the cell structures, and to map out relationships between processing, density, and strength. This work is supported by the US-DOE, under contract DE-AC52-07NA27344, LLNL. APS is supported by the U.S. DOE, BES, Office of Science under contract No. W-31-109-ENG-38.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Electromigration Reliability

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee

Program Organizers: Sung Kang, IBM Corp; Iver Anderson, Iowa State University; Srinivas Chada, Medtronic; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Albert Wu, National Central University

Monday PM — Room: 2020
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: C Robert Kao, National Taiwan University; Albert Wu, National Central Univ

2:00 PM Invited

Stress-Migration Induced by Electromigration in Flip Chip Pb-Free Solder Joints: *King-Ning Tu*[1]; [1]University of California

Abstract: Due to the unique line-to-bump configuration of flip chip, current crowding occurs at the location where the current enters or leaves the solder bump. At the anode, a concentration of compressive stress occurs due to electromigration. The concentrated compressive stress can squeeze out a Sn whisker or hillock, even at temperatures above 100°C. To investigate the stress-migration driven by electromigration, x-ray diffraction by micro beam synchrotron radiation has been performed. Transient stress build-up in the Sn matrix was detected. Since Cu diffuses interstitially in Sn and is much faster than self diffusion of Sn in Sn, intermetallic compound (IMC) formation of Cu-Sn in the anode region has been found. While the IMC may increase the compressive stress, it also may block the diffusion of Sn and slow down the growth of whisker and hillock.

2:20 PM

In Situ Study of Electromigration-Induced Orientation Evolution in Pb-Free Solder Joint by Synchrotron Microdiffraction: *Kai Chen*[1]; Nobumichi Tamura[1]; King-Ning Tu[2]; Yi-Shao Lai[3]; [1]Lawrence Berkeley National Laboratory; [2]UCLA; [3]Advanced Semiconductor Engineering

The rotation of Sn grains in Pb-free flip chip solder joints hasn't been reported in literature so far although it has been observed in Sn strips. In this letter, we report the detailed careful study of the grain orientation evolution induced by electromigration by synchrotron radiation based white beam X-ray microdiffraction. It is found that the grains in solder joint rotate much slower than in Sn strip even if under higher current density. On the other hand, based on our estimation, the reorientation of the grains in solder joints also results in the reduction of electric resistivity, similar to the case in Sn strip, although the change is not detected experimentally. We will also discuss the reason why the electric resistance decreases much more in strips than in the Sn-based solders, and the different diving force for the grain growth in solder joint than in thin film interconnect lines.

2:35 PM

The Direct Measurement of Stress Distribution on Silicon under Thermal Effect and Electromigration in Flip Chip: Albert Wu[1]; *Chun-Yang Tsai*[1]; [1]National Central University

The reliability of the flip chip is affected by electromigration or thermal stress. The variation of stress on silicon chip was directly measured by in-situ synchrotron X-ray. The minute variation of stress level in the solder strips could be resolved by high brightness synchrotron source. The flip chip samples were stressed under various temperatures and current densities. At the edge of the silicon chip, the stress level is the highest at the center than at the edge. The simulation results is provided for the discussion in this paper.

2:50 PM

Electromigration in Tin-Copper, Tin-Silver, and Eutectic Tin-Lead Flip Chip Solder Joints: *Luhua Xu*[1]; Jarrett Liang[1]; Jung-Kyu Han[1]; Yi-Shao Lai[2]; K.N. Tu[1]; [1]University of California, Los Angeles; [2]Advanced Semiconductor Engineering

A comprehensive study of the electromigration reliability of SnCu, SnAg, and eutectic SnPb flip chip solder joints with and without thick UBM at the chip side is reported. The samples were stressed at multiple high temperatures and current densities. The polarity effects on intermetallic compound (IMC) growth and metallization consumption were characterized for both powered and un-powered solder joints. The un-powered solder joints exhibited slower growth in IMCs as well as minor consumption of copper metallization. However, due to thermal gradient from chip to substrate, it also shows polarity phenomena. The powered joints exhibited microstructural changes consistent with a directional diffusion guided by electron wind. Namely, the copper metallization at the cathode experienced accelerated consumption. The propagation of pancake voids was observed in detail, from initiation under the passivated area, to partially propagating across the aluminum line opening, and to complete propagation.

3:05 PM

Effects of Current Density, Environment, and Temperature on Adhesion and Debonding Kinetics of Cu / Barrier Interfaces: *Ryan Birringer*[1]; Roey Shaviv[2]; Reinhold Dauskardt[1]; [1]Stanford University; [2]Novellus Systems Inc.

It is widely accepted that electromigration in modern Cu interconnects is closely related to adhesion and bonding between the Cu line and the top capping layer. While four-point bend thin-film adhesion techniques are commonly used to characterize adhesion in Cu / barrier layer interfaces, the kinetics of sub-critical interface debonding in these films has been largely ignored. In the present study, we propose a new technique for characterizing the kinetics of EM void growth. The method is based on modified versions of well-known thin-film adhesion techniques and is used to quantify sub-critical debonding between Cu and selected barrier layers in the presence of variable current density through the film, environment, and temperature. Selected barrier materials include SiC, SiN, and CoWP. The sub-critical debonding kinetics of these barrier films are largely influenced by the environment, and the sub-critical debonding mechanisms present in ambient, forming gas, and noble gas environments are compared.

3:20 PM

The Study of Flip Chip Electromigration under Extra High Current Density: *Yu-Wei Lin*[1]; Jia-Hong Ke[1]; C. Kao[1]; Yi-Shao Lai[2]; [1]National Taiwan University; [2]Advanced Semiconductor Engineering, Inc.

The electromigration failure mechanism in flip chip solder joints under extra high current density (>1×10^4 A/cm^2) was studied. It is known that the joint temperature under current stressing is strongly associated with the applied current density due to Joule heating effect. Hence, the heat generation and dissipation was found to be a very important factor on the final failure mechanism. In this study, two experimental setups were used. One was with a cooling system to make the chip temperature constant, and the other was not. Without cooling, the temperature increased while increasing the applied current. When the current density was up to 5×10^4 A/cm^2, rapid failure was caused by excessive Joule heating. With cooling, more time to failure was experience at the same current density. Therefore, this method provided a way to de-couple the applied current density and the device temperature.

3:35 PM

Effect of UBM on Electromigration Lifetime of SnAg Solder Joints: *Hsiao-Yun Chen*[1]; Chih Chen[1]; [1]NCTU

Electromigration in flip chip solder joints is investigated under current stressing at 135, 150 and 165°C. By using Kelvin probe, the failure time can be defined as bump with current stressing down by increasing 20% of its original value, therefore direct comparison between 3-µm Cu and 5-µm Cu/3-µm Ni UBM systems can be accomplished. Additionally, by using Al trace TCR effect, it is possible to measure the real stressing temperature. It is found that the solder joints with the 5-µm Cu/3-µm Ni UBM have longer electromigration lifetime. The Ni layer may retard the electromigration failure since it is a good diffusion barrier for Cu. Furthermore, the Ni layer may relieve the current crowding effect.

Three-dimensional simulation on current density distribution has been carried out for these two systems and the results will be presented in the conference.

3:50 PM Break

4:05 PM Invited
Comprehensive Studies on Microstructural Evolution from Current Stressing: Y.C. Lee[1]; Cheng-En Ho[2]; K. Subramanian[1]; *Andre Lee*[1]; [1]Michigan State University; [2]Yuan Ze University

Imposition of high current densities causes material migration due to momentum transfer between electrons and atoms/ions. A clear quantitative understanding of the events that occur under such conditions, especially in multi-phase materials, is far from complete due to several geometry related constraints. Recently, our group has developed a realistic joint configuration to avoid unnecessary complications due to current crowding with the ability to impose a well-described thermal environment. Using these joints effects of current density and temperature on microstructural evolution in two-phase electronic solder joints were studied by Synchrotron X-rays and by Confocal Laser Scanning, Optical, and Scanning Electron Microscopies. These studies have provided unique opportunities to address materials movement, microstructure evolution, as well as solid-state reactions, due solely to the influence of current stressing. Critical current densities at various temperatures for evolution and extents of such events, under various current stressing scenarios were evaluated.

4:25 PM
In-Situ Observation of Stress Evolution on Solder Strips under Electromigration: Albert Wu[1]; *Ciao-Nan Siao*[1]; [1]National Central University

Synchrotron radiation x-ray diffraction was employed for in-situ observation of stress evolution on solder thin film strips under various temperatures and current densities. The minute variation of stress level in the solder strips could be resolved by high brightness synchrotron X-ray. The tensile stress at the cathode and the compressive stress at the anode degraded with time. The kinetics of the atomic diffusion under electron wind forces and the consequent stress distribution is measured; the relationship between the variation of morphology and the changes in the stress level is discussed in this paper.

4:40 PM
Electromigration-Induced Failures at Cu/Sn/Cu Flip-Chip Joint Interfaces: *Hua-wei Tseng*[1]; Chengyi Liu[1]; [1]National Central Unversity

EM-induced failure modes at Cu/Sn/Cu solder joint interfaces were studied. Since the geometry of the flip-chip solder joint, the current density would vary in the both joint interface. Depending on the current-stressing density, different EM-induced failure modes were observed at the cathode joint interface; EM-induced dissolution at the high current stressing site and voiding at the low current stressing site. At the opposite anode interface, Kirkendall void formation also varies with the current distribution. At current crowding site, the current exiting corner, a serious Kirkendall voiding coalesced into a gap between the Cu3Sn/Cu interface. The Cu consumption activation energy without current stressing was determined in this work. With current stressing, the Cu consumption activation energy decreased. We believe that the enhancement of Cu consumption by current stressing is due to the Cu atoms dissolved in the Sn to electromigrate instantaneously toward the anode side.

4:55 PM
Study of Electromigration Phenomena in Ultra-Thin Lead-Free Solder Joints: *Cheng-En Ho*[1]; Wei-Hsiang Wu[1]; [1]Yuan Ze University

Traditional flip-chip solder joints have a line-to-bump configuration consisting of a solder bump with ~100 microns in height and electric circuits with few microns in thickness. Because of the huge divergence in the cross-section between circuit/bump and the electric current takes a ~90-degree turn at the contact, there will be a significant current crowding at the cathode side of solder, where a localized high current density induced a massive electromigration may occur. Failures in the joints resulted from such the current crowding effect mainly included void nucleation and propagation, local melting of solder, and rapid dissolution of metallization pads. To minimize the damages, modification of Cu pillars with ultra-tin solder bumping (20 microns) is being attempted by industry recently. However, little research has been devoted to evaluate its reliability. This study is aimed at gaining a better understanding of electromigration behaviors in lead-free solder joints with ultra-tin solder thicknesses.

5:10 PM
Study of Electromigration-Induced Failures on Ni/Cu Bi-Layer Bond Pad: *Yu-Hsiang Hsiao*[1]; Chengyi Liu[1]; [1]National Central University

EM (electromigration) on Sn(Cu)/Ni/Cu solder joints under 104 A/cm2 at 160° were studied. For the pure Sn/Ni/Cu case, the interfacial compound layer is mainly Cu6Sn5 compound phase. Under EM effect, the interfacial compound Cu6Sn5 compound phase layer would suffer serious EM-induced dissolution. (2) Sn(Cu) alloys, i.e., Sn0.7Cu and Sn3.0Cu, formed a (Cu,Ni)6Sn5 interfacial layer at the joint interfaces. The interfacial (Cu,Ni)6Sn5 compound layer shows a stronger resistance to the EM-induced dissolution. The interfacial (Cu,Ni)6Sn5 compound layer grows with the current stressing time in the Sn0.7Cu/Ni/Cu case.

5:25 PM
In-Situ Studies of Electrical Current Induced Whiskers: *Andre Lee*[1]; K. Subramanian[1]; Cheng-En Ho[2]; Wenjun Liu[3]; [1]Michigan State University; [2]Yuan Ze University; [3]Argonne National Laboratory

Use of high Sn solders for removing Pb from electronic packages has brought about concerns due to the spontaneous growth of metallic whiskers. Although Sn is known to spontaneously form whiskers, the formation and growth of Sn whiskers have never been observed in-situ, due to lack of knowledge of the exact causes and conditions for their formation and growth. Our recent studies on electrical current induced material movements have led us to design a package to study spontaneous formation and growth of Sn whiskers. This package facilitates formation of optically visible whiskers with a constant current of 0.2 amps at room temperature within 3 hours. Such conditions enable in-situ investigations of whisker formation and growth. Micro diffraction X-rays at APS was used to examine the evolution of grain structure, strain distribution during whisker growth in matte Sn coatings at various amplitudes of externally applied electric current and temperatures.

5:40 PM
Electromigration Study of Eutectic SnPb Flip-Chip Solder Joints on Ceramic Substrates: *Chung Kuang Lin*[1]; Chih Chen[1]; [1]National Chiao Tung University

This study investigates electromigration study of eutectic SnPb flip-chip solder joints on ceramic substrates. The under bump metallization (UBM) structure consists of 5-μm Cu / 3-μm Ni under bump metallization (UBM). Under the current stressing by 0.9A at 150°C, we did not find void formation but a large amount of intermetallic compound (IMC) of Cu6(Sn,Ni)5 were formed when the bump resistance increased 10 mO. Three-dimensional electrical simulation by finite element analysis was carried out to simulate the current density distribution in solder joints with slit Cu traces. It is found that the current density was almost uniformly distributed in solder joint for this structure. It is found that the bump resistance only increases 0.07mO when half of the solder bump was transformed into Cu6Sn5 IMC. The reason for the low crowding effect in the solder joints will be discussed in the conference.

5:55 PM
Effective Charge of Electromigration from the Perspective of Electromagnetism: *Peng Zhou*[1]; William Johnson[2]; [1]University of California Irvine; [2]University of Virginia

The mechanism of electromigration is discussed from the perspective of electromagnetism, other than from the traditional view of momentum exchange owing to the collision between electrons and diffusing atoms. It is suggested that the energy associated with electromigration is related to the work done by the electromagnetic fields, and conversion of the non-electrostatic energy, which is transferred by the Poynting's vector from the electric power source, into the chemical energy of the diffusion system. It is also suggested that the momentum transferred to the diffusing atoms is related to the Maxwell stress; at low temperatures, the momentum is transferred via maxwell stress to the chemical impurities and crystal imperfections to give rise to the ''polarity effect''. It is shown from the perspective of electromagnetism, the effective driving force of electromigration has a square dependence on the current density; therefore, the effective charge number is linearly related to the current density.

Peirce-Smith Converting Centennial Symposium: Operational Aspects

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
Program Organizer: Joël Kapusta, Air Liquide

Monday PM Room: 2009
February 16, 2009 Location: Moscone West Convention Center

Session Chairs: Anthony Warner, WorleyParsons HGE; Phillip Mackey, Xstrata Nickel

2:00 PM Keynote
A Century of Converter Operation in Vale Inco's Ontario Operations: Samuel Marcuson[1]; [1]Vale Inco Ltd

Smelting of nickel-copper sulfide ores at Vale Inco's Sudbury operations commenced in 1888 and the first converting plant went into operation in 1892. As the demand for nickel grew, the smelting operations expanded and by the 1960's the Copper Cliff Smelter featured the longest converter aisle in the world with space for nineteen Peirce-Smith converters. During this long period, different converting techniques and vessels were employed. The evolution of converting is reviewed with special emphasis on the modifications made to the Peirce-Smith vessel to accommodate the metallurgical and process needs of the changing times.

2:30 PM
Converting and Casting at Boliden's Rönnskär Smelter 2009 - An Update: Peter Olsson[1]; *Magnus Ek*[1]; [1]Boliden Mineral AB

The Rönnskär Smelter has been faithful to Peirce Smith Converting during its 79 years of existence. Though a visitor to our smelter museum will easily find out that a lot has happened since the beginning – and development continues. In this paper an update on Converting and Casting at the Rönnskär Smelter is presented. Recent developments include an investment into converter process gas handling, but also day-to-day operational aspects such as converter aisle productivity and flexibility, but also minimal EH&S impact.

2:50 PM
Environmental Situation and Enhanced Productivity at Norddeutsche Affinerie AG: Thomas Buenger[1]; [1]Norddeutsche Affinerie AG

The Hamburg smelter of Norddeutsche Affinerie AG is situated near downtown of the city of Hamburg and is therefore a special subject to fullfil environmental requirements according to the german legal regulations. Therefore are investments into increased concentrate throughput every time linked to packages to enhance the emission situation of the smelter. This connection in between environment protection and productivity enhancement should be explained by the example of the Peirce-Smith converter plant of the Hamburg smelter. As an outstanding investment this can be shown on hand of the transition of scrap charging via boot from the front side to a fully automatic and completely sucked scrap charging system.

3:10 PM
Evolution of the Converter Aisle at Xstrata Nickel's Sudbury Smelter: *Bryan Salt*[1]; Enrico Cerilli[1]; [1]Xstrata Nickel

Xstrata Nickel's Sudbury Smelter has systematically developed its converting aisle from a single stage to a counter current multi stage converting process. This has required the development of two unique converters; the slag cleaning vessel and the slag make converter. This has resulted in a unique three stage converting process that enables treatment of secondary materials as well as resulting in a slag that can be discharged without further treatment. The paper will also discuss future changes currently being considered to further improve environmental, plant hygiene and productivity performance.

3:30 PM Break

3:50 PM
Recent Operation and Improvement at the Sumitomo Toyo Peirce-Smith Converters: *Kazuhiro Mori*[1]; Katsuhiko Nagai[1]; Kosei Morita[1]; Osamu Nakano[1]; [1]Sumitomo Metal Mining Company, Ltd.

The Sumitomo Toyo Smelter and Refinery commenced operations in 1971 with three Peirce-Smith (PS) converters in operation. At the time, a "2 hot1 blowing" sequence was adopted (two vessels hot, one blowing and the third unit under repair), with in-stack times reaching 90%, producing five charges per day. Under the 2001 expansion program at the plant, a new "2 hot-2 blowing" operational pattern was adopted at the converters along with the installation of additional converter blowers and crane. In 2005, a fourth converter was installed and a "3 hot-2 blowing" operation commenced. Along with improvements in the anode department, the converters now produce over eight charges per day. The control of sulphur dioxide fugitive gas at the converters has also advanced, and the sulphur fixation rate at Toyo now exceeds 99.9%. This paper discusses the Toyo converter operation and describes improvements in environmental management techniques used at the plant.

4:10 PM
Changing Reality: Continuous Production Control for Optimized Productivity: Wilhelm Wendt[1]; *Willy Persson*[1]; [1]Semtech Metallurgy AB

With rizing price of raw materials and increasing energy and environmental restrictions the optimization of pyrometallurgical processes beyond what can be achieved via modelling and operator experience has come more and more into focus and led to a quest for methods for dynamic process control. Independent of the details of the control mechanism it has to be based on real-time information on the status of the process. Since mid 1990s Semtech OPC Systems are in routine use at a number of smelters to provide continuous on-line information on the instantaneous status of converting processes in Peirce-Smith converters. This information offers an opportunity for the operators to maintain / bring back the process to the desired route. This presentation will discuss experiences from applications to converting of copper and nickel in Peirce-Smith converters. Special emphasis will be laid on de-bottlenecking, e.g., in concentrate injection, revert accumulation, magnetite handling and oxide-skim transfer.

4:30 PM
The Use of Injection Tuyeres Clad Welding with Anti- Abrasive in the Teniente Converter: Luis González M.[1]; Juan C. Davis C.[1]; *Guillermo Guzman D.*[1]; [1]Ventanas Smelter and Refinery, Codelco-Chile

The Teniente converter (CT) of the Ventanas Smelter and Refinery of Codelco-Chile has two tuyeres for dry concentrate injection directly into the melt. The diluted phase injection system can feed 15 to 20 kg of concentrate for 1 kg of air, which creates a very abrasive solid/gas phase. The tuyeres commonly used lasted for about 3.5 days due to the accelerated decay of the shell, and 1.5 hours were necessary for the replacement of new ones in the reactor. Tests were carried out with common shell tuyeres with a tungsten based welding cover, and a life span of 28 days was reached, increasing the Teniente converter operational continuity between 1.5 to 2 %.

4:50 PM
Full Utilization of the Matte Converting Process Energy for Operating Conditions of the RTB Bor: *Bogdan Petkovic*[1]; Boban Todorovic[1]; [1]RTB Bor, TIR Bor, Topionica

In the early 90's a completely unknown problem was encountered at the converter Department: it was a chronic shortage of the cold copper charge needed for the second operating period. A high temperature operating regime was caused which further provoked a chain of consequences such as a drastic drop of the number of operations in campaigns. The consumption of repair bricks reached the quantity of 8.3955 kg / tonne of anode Cu. In 1994, we won the process of cold material smelting during the "copper" blowing [2]. The number of operations per campaign reached 180-200 again. Also in 1994 we won the process of smelting cold materials to obtain "tin" blowing with the purpose of completing their processing cycle [2]. During the period 1994 – 2007 a total of 107,845 tonnes of cold material were remelted to make copper and 56,794 tonnes were re-smelted to obtain "tin". Upon synthesizing these two processes, a completely new model was achieved in 2005, by Bogdan Petkoviæ, which entirely utilized all available energy of the Peirce-Smith (PS) converters of the Bor Copper Smelter and now represents the basis of the time cycle. The cold material now enters the converter plant, copper recovery is increased and the method itself is environmentally friendly.

5:10 PM
Recent Operations of Peirce-Smith Converters in Pirdop Smelter: *Evgeni Marinov*[1]; Ivailo Vasilev[1]; Dimo Kirilov[1]; [1]Cumerio

The Pirdop Smelter was privatised by the Bulgarian government in September 1997. Since then, an investment program has been implemented to increase capacity to 290,000 tpy of copper, while improving the environmental

performance. A significant part of this investment program involved reconstruction of the three Peirce-Smith converters and gas handling system. Between June 2000 and April 2002, converter capacity was increased and primary gas collection systems at the Converters Section were upgraded. The result was a significant reduction in emissions. From 2002 until present, a continuous improvement of operating and environmental performance has been achieved. In 2005, Pirdop received from the Bulgarian Government the Complex Permit under the EU IPPC criteria. An Environmental Compliance Program was accordingly defined aiming for reduction in secondary emissions. In November 2007, a Secondary Gases Cleaning plant was put into operation. This paper describes these projects and the challenges faced by the company.

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials VIII: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Chih-ming Chen, National Chung-Hsing University; Srinivas Chada, Medtronic; Sinn-wen Chen, National Tsing-Hua University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-ho Lee, Hongik University; Daniel J. Lewis, Rensselaer Polytechnic Institute; Kejun Zeng, Texas Instruments; Wojciech Gierlotka, AGH University of Science and Technology; Yee-wen Yen, National Taiwan University of Science and Technology

Monday PM — Room: 2022
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Hans Flandorfer, University of Vienna; Wojciech Gierlotka, AGH University of Science and Technology

2:00 PM Invited ***2009 Hume Rothery Award Recipient***
Alloy Phase Metastability and Microstructure: *John Perepezko*[1]; [1]University of Wisconsin

Alloy phase stability is usually analyzed on the basis of the most stable structure for a given composition as guided by the Hume-Rothery rules and recent first principle calculations. However, some of the most interesting and useful phase reactions involved in microstructure development are based upon metastable structures. Metastability can develop in many processing routes where the initial phase is subjected to high supersaturation, pressure, undercooling or high defect concentration conditions that are encountered in nucleation processes. In these cases, the initial microstructure phase selection is directed by metastable phases and their relaxation towards equilibrium. While metastable structures are often not retained in the final microstructure in bulk volumes, they can still play a role as precursor structures and can be dominant in nanoscale volumes. Several examples from solidification, interface controlled reactions, precipitation and severe plastic deformation are discussed to illustrate the role of metastable alloy phases in microstructure development.

2:20 PM Invited
Development of Thermodynamic Database for Cu-Base Alloy Systems and Micro-Solders: Ikuo Ohnuma[1]; Yoshikazu Takaku[1]; Cui Ping Wang[2]; Xing Jun Liu[2]; *Kiyohito Ishida*[1]; [1]Tohoku University; [2]Xiamen University

The thermodynamic database for Cu-base alloys and micro-solders have been constructed by the CALPHAD (Calculation of Phase Diagrams) method. Based on the thermodynamic assessments on the Cu-X binary and Cu-X-Y ternary systems, the phase diagrams and thermodynamic properties of Cu-base multi-components systems which includes eleven elements of Cu, B, C, Cr, Fe, Ni, P, Si, Sn, Ti and Zn can be calculated. The solder database for eight elements of Ag, Bi, Cu, In, Pb, Sb, Sn and Zn have also been developed, which can be utilized for all combinations of elements and all composition ranges. The elements of Al, Au and Ni are also available for the calculation in the limited composition ranges. The typical examples of the calculations and applications will be presented.

2:40 PM
Au-Sn-Based Lead-Free Solder Alloys: *Leszek Zabdyr*[1]; Anna Wierzbicka-Miernik[1]; Joanna Wojewoda-Budka[1]; Pawel Zieba[1]; [1]Polish Academy of Sciences

Three invariant transition reactions were found among other features of the calculated poly-thermal projection of the liquidus surface close to Sn-corner of the system at temperatures: 252, 289 and 305C, respectively. That and the relatively low gold content makes them the potential candidates as high-temperature lead-free solder materials.Alloy samples of three invariant compositions were prepared, and temperatures of solid-liquid equilibria were determined by DSC and DTA to confirm results of calculations. Structure and chemical composition were then examined using SEM-EDS device. Surface tension was cal-culated for the invariant liquids using Butler approach. Solid alloys of compositions under accord were used to prepare solder joints on copper substrates. Shear test and microhardness measurements were performed, and cross-section through the joints were analysed by SEM-EDS to give an idea about structural characteristic of joint material and possible interaction with substrate material.

2:55 PM
Thermodynamic Modeling Studies of the Cu-In and Sn-In-Cu Systems: *Wojciech Gierlotka*[1]; Sinn-wen Chen[2]; Shih-kan Lin[2]; [1]AGH University of Science and Technology; [2]National Tsing Hua University

Sn-Cu-In ternary system is of interests for lead-free solder applications. A thermodynamic model of this ternary system is developed using the CALPHAD method based on the experimental results in the literature as well as those obtained in this study. In addition to the terminal phases and binary compounds, at 160°C there are one continuous solid solution formed between the η-Cu6Sn5 and η-Cu2In, and the δ 1-Cu41(Sn,In)11 phase. The binary Cu-In system has been remodeled and the homogeneity ranges of the compounds have been described by the sublattice models similar to those used in the Cu-Sn system. Results of the calculation in the ternary system, such as thermodynamic properties of liquid and solid phases, isothermal and isopleths sections, and liquidus projections are compared with the experimental data.

3:10 PM
Size and Substrate Effects upon Undercooling of Pb-Free Solders: *Yu-chih Huang*[1]; Kuang-siang Wu[1]; Sinn-wen Chen[1]; [1]National Tsing Hua University

The melting and solidification temperatures of solders and solders on substrates are determined using differential scanning calorimetry. The solders are Sn, Sn-0.7wt%Cu, Sn-3.5wt%Ag and Sn-3.8wt%Ag-0.7wt%Cu solders, and the substrates are Ni, Cu and Ag. The heating and cooling rates are 10°C/min. The solders are of two different sizes, 5mg and 1mg. With repeated DSC measurements, the statistical distributions of degrees of undercooling are determined. The degrees of superheating are less than 2 degrees at 10°C/min heating rate and are negligible comparing with the degrees of undercooling which could be as significant as 56 degrees. Undercooling is more significant and variation is larger when the solders are of smaller sizes. The undercooling of solders on substrates is less significant than that of solders alone. Among the three kinds of substrates, solders on Ni are with the least undercooling.

3:25 PM Break

3:45 PM Invited
Experimental Methods to Determine Thermodynamic Properties: Examples: *Adolf Mikula*[1]; [1]University of Vienna

For phase stability, phase transformation the thermodynamic properties of the materials involved, must be known. No calculation of phase diagrams, viscosity, surface tension can be carried out without the knowledge of the partial or integral thermodynamic properties.In this lecture it will be demonstrated what type of experimantal methods are available and what kind of data we get from different experiments and how reliable these data are. On a few examples it will be shown, how these data are used to calculate different properties of electronic materials.

4:05 PM Invited
Solidification Behavior of Cu-Ni-Sn: *Hans Flandorfer*[1]; Clemens Schmetterer[1]; Herbert Ipser[1]; [1]University of Vienna

Knowledge about the intermetallic system Cu-Ni-Sn is highly important for nickel-bronze alloys with additions of tin and for lead-free soldering as it concerns the majority of solder/substrate interactions of hitherto used lead-free solders. Detailed information to the solidification and formation of ternary phases is a crucial point for the control of the microstructure and texture of alloys and alloy interfaces.The system Cu-Ni-Sn was investigated by means of XRD, DTA and metallography including EPMA techniques. The occurrence of ordering phenomena at Cu2NiSn along (NixCu1-x)3Sn and at Cu4Ni2Sn5 along (NixCu1-x)6Sn5 has been described in literature. The enthalpy of formation of alloys along these sections was determined by tin solution calorimetry. Ordering should be indicated by a significant deviation from linearity of the respective

deltafH versus xCu (or xNi) curves. According to our results no such behavior could be observed.

4:25 PM
Investigation of Various Properties of Lead Free Solders: *Michael Hindler*[1]; Sabine Knott[1]; Zuoan Li[2]; Clemens Schmetterer[1]; Peter Terzieff[1]; Adolf Mikula[1]; [1]Universitaet Wien; [2]University of Oslo

Different properties of various lead free solders have been investigated. The wetting angles, viscosity, surface tension and thermodynamic properties of Ag-Cu-Sn, Ag-Bi-Sn, Ag-Au-Sn, Au-Cu-Sn and Pd-Sn-Zn have been determined. The thermodynamic properties of the ternary Ag-Au-Sn, Au-Cu-Sn, and Pd-Sn-Zn systems have been determined with the EMF method with a liquid electrolyte as well as with a Calvet type calorimeter. With the data obtained by the calorimetric method the viscosity of the Ag-Au-Sn system has been calculated using the model of Iida et al. The surface tension of the Ag-Bi-Sn and the Ag-Au-Sn system was calculated and will be presented. Additional results of the wetting angle of the Ag-Cu-Sn alloy with various compositions on copper and nickel substrate, as well as the temperature dependency of the wetting angles and some intermetallic structures will be presented.

4:40 PM
Development of Pb-Free Heat Resistant Joints Using Sn-Bi and Ag Powders: *Yoshikazu Takaku*[1]; Yuki Sakurada[2]; Ikuo Ohnuma[1]; Kiyohito Ishida[1]; [1]CREST-JST, Tohoku University; [2] Tohoku University

TLPS (Transient Liquid Phase Sintering) is a candidate method of heat-resistant jointing, which makes use of the reaction between low-melting temperature powder (P1) and reactive powder (P2). During heat treatment above the melting temperature of P1, the molten P1 reacts rapidly with the solid P2, which results in the formation of intermetallic compound (IMC). In this study, the TLPS properties of a combination of eutectic Sn-Bi and Ag powder were investigated. During differential scanning calorimetry measurement, an endothermic reaction occurred at the eutectic temperature of the Sn-Bi (139°C), followed by an exothermic reaction at about 220°C, which was caused by the formation of the Ag3Sn IMC and Bi-rich solid solution phases. After the overall measurement, the reactant contained the Ag3Sn and Bi-rich phases, which melt above 270°C, with a small amount of residual Sn-Bi eutectic phase. These results suggest that the TLPS process can be applied for Pb-free heat-resistant soldering.

4:55 PM
Phase Behavior of Sn- and Zr-Doped Alpha-Al2O3 upon Solution Annealing: *Liu Lung*[1]; Lu Jer-Han[1]; Shen Pouyan[1]; [1]University of NSYSU

α-Al2O3 powders mixed with 8 mole % SnO2 versus ZrO2 were sintered and then solution annealed in air at specified temperatures in order to study the expulsion of a small amount of aliovalent and size-mismatch solute. X-ray diffraction and analytical electron microscopic observations indicated that the Sn-doped alpha-Al2O3 sintered at 1500°C and then annealed at 1000°C contains disk-like Guinier-Preston (G.P.) zones, which are parallel to (0001), (-12-10) and (10-10) planes of the host lattice. By contrast, the Zr-doped alpha-Al2O3 shows (0001) and {01-12}-specific distortion planes in association with coherent defect clusters when sintered (1600°C) and then solution annealed at higher temperatures (1200 and 1350°C) for effective diffusion. The precipitation of (hkil)-specific G.P. zones and lattice distortion of the corundum-type structure upon exsolution of a rather limited amount of Sn4+ and Zn4+, respectively can be rationalized by structure anisotropy and defect chemistry of the hexagonal close packed oxide.

Recent Advances in Thin Films: Applications

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Thin Films and Interfaces Committee
Program Organizers: Nuggehalli Ravindra, New Jersey Institute of Technology; Gregory Krumdick, Argonne National Laboratory; Choong-un Kim, University of Texas; Narsingh Singh, Northrop Grumman, ES

Monday PM — Room: 3011
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Narsingh Singh, Northrop Grumman Corp ES; Gregory Krumdick, Argonne National Laboratory

2:00 PM Introductory Comments

2:05 PM Keynote
Mechanisms of Crystallization and Grain Growth of Amorphous Si Thin Films by Metal-Initiated Crystallization: Nuggehalli Ravindra[1]; *Bhushan Sopori*[2]; Vishal Mehta[1]; Peter Rupnowski[2]; A. Rangappan[2]; [1]New Jersey Institute of Technology; [2]National Renewable Energy Lab

The mechanisms of crystallization and grain growth of amorphous silicon thin Films by metal-initiated crystallization are described in relation to silicon solar cell fabrication.

2:35 PM Invited
Rare Earth Impurity Centers in Silicon for Enhancement of Light Emission and Improving Photovoltaic Efficiency: *Sufian Abedrabbo*[1]; Anthony Fiory[1]; Nuggehalli Ravindra[1]; [1]New Jersey Institute of Technology

While silicon is intrinsically an inefficient light emitter, there is considerable interest in improving its optical emission efficiency through materials modification by incorporation of rare-earth metals. Impurity centers will modify the silicon bandgap enabling an improved solar response when acting as photovoltaic devices. In this work, rare-earth metals impurity centers in silicon are investigated. Erbium is co-evaporated with silicon on silicon substrates along with other proper dopants. The processed samples are investigated optically by photoluminescence and structurally by Rutherford backscattering.

3:05 PM
An Integrated Optical and Electronic Method to Measure Nano deflections in a Silicon Diaphragm: *Ivan Padron*[1]; Anthony T. Fiory[1]; N.M. Ravindra[1]; [1]New Jersey Institute of Technology

The introduction of an embossed diaphragm in the fabrication of a pressure sensor facilitates in the fabrication of a Fabry-Perot optical sensor that permits to measure nano deflections in a silicon diaphragm. A piezoresistive based electronic sensor is introduced as a Q-point stabilization method. An analytical and experimental analysis to study the behavior and performance of the Fabry-Perot pressure sensor is presented.

3:25 PM
Effect of Gas Flow Rate on the Formation of Aligned Nanorods in ZnO Thin Films: Nuggehalli Ravindra[1]; *Sudhakar Shet*[2]; Kwang-Soon Ahn[2]; Yanfa Yan[2]; John Turner[2]; Mowafak Al-Jassim[2]; [1]New Jersey Institute of Technology; [2]National Renewable Energy Laboratory

ZnO thin films are deposited in mixed Ar and N2 gas ambient at substrate temperature of 500oC by rf sputtering ZnO targets. We find that the presence of optimum N2 to Ar ratio in the deposition ambient promotes the formation of well aligned ZnO nanorods. ZnO thin films grown at 25 % N2 gas flow rate promoted aligned nanorods along c-axis exhibit significantly enhanced photoelectrochemical response, as compared to ZnO thin films grown at other N2 to Ar gas flow ratios. Our results suggest that chamber ambient is very important for the formation of aligned nanostructures, which offer potential advantages for improving the efficiency of photoelectrochemical water splitting for H2 production.

3:45 PM

Modeling Epitaxial Quantum Dots Formation and Growth Using Finite Difference Method: *Solmaz Torabi*[1]; Peng Zhou[1]; Shuwang Li[2]; Steven Wise[3]; Axel Voigt[4]; John Lowengrub[1]; [1]University of California; [2]Illinois Institute of Technology; [3]University of Tennessee; [4]Technische Universität Dresden

Self-assembly semiconductor nanostructures such as quantum-dots are a promising inexpensive and effective approach to manufacture novel nanoscale electronic devices. The main goal is the production of large numbers of spatially ordered nanostructures with narrow size distribution via a controlled self-assembly process. Consequently, we need to have a fundamental understanding of the self-organization process (nucleation, growth and coarsening) during epitaxial growth to achieve this goal. For this reason we study the influence of elastic, surface energies and kinetics on heteroepitaxial thin film growth. Numerical studies in 2D and 3D are presented that complement experimental investigations. Here, we present a new approach for modeling strongly anisotropic crystal and epitaxial growth using regularized, anisotropic Cahn-Hilliard-type equations as a model for the growth and coarsening of thin films. A key feature of the new approach is that the interface thickness is independent of crystallographic orientation. We use an adaptive nonlinear multigrid finite-difference method.

4:05 PM

Opto-Electronic Properties of Pthalocyanines: *Fiorella Fuentes*[1]; Nuggehalli Ravindra[1]; Parth Patel[1]; [1]NJIT

An overview of the opto-electronic properties of phthalocyanines is presented in this study. This overview is based on the application of Wemple-DiDomenico and Penn-like models to understand the opto-electronic properties of phthalocyanines in relation to their band structure. The utility of Pthalocyanines to various medical applications is summarized.

4:25 PM Break

4:40 PM Invited

Al and N Co-Doped ZnO Films with Significantly Reduced Bandgap and Enhanced Photoelectrochemical Responses: Nuggehalli Ravindra[1]; *Sudhakar Shet*[2]; Kwang-Soon Ahn[2]; Yanfa Yan[2]; John Turner[2]; Mowafak Al-Jassim[2]; [1]New Jersey Institute of Technology; [2]National Renewable Energy Laboratory

We present results on bandgap narrowing and photoelectrochemical (PEC) response of Al amd N co-doped ZnO thin films. The ZnO:(Al,N) thin films were deposited by sputtering at substrate temperature of 100°C and followed by postannealing at 500°C in air for 2 hours. We found that ZnO:(Al,N) thin films exhibited significantly enhanced crystallinity as compared to ZnO, Al doped ZnO (ZnO:Al), and N doped ZnO (ZnO:N) at the same growth conditions. Furthermore, ZnO:(Al,N) thin films exhibited enhanced N-incorporation and resulted in much reduced bandgap. As a result, ZnO:(Al,N) thin films achieved improved PEC response, as compared to ZnO, ZnO:Al, and ZnO:N thin films. Our results suggest a general way to reduce the bandgap and improve PEC response for wide-bandgap oxides.

5:10 PM Invited

Enhanced Ferroelectric Properties and the Integrated Growth of Hf-Doped Bismuth Titanate Thin Films on GaN Substrates: *Jun Zhu*[1]; Yanrong Li[1]; [1]State Key Laboratory of Electronic Thin Films and Integrated Devices, University of Electronics Science and Technology of China

Hf-doped BIT (BTH) thin films were fabricated on SrRuO3/SrTiO3/TiO2 buffered GaN substrates by pulsed laser deposition. As confirmed by x-ray photoelectron spectroscopy investigation, the oxygen bonded to B-site ions became much stronger after Hf substitution. X-ray diffraction scans, including θ-2θ and ρ-scans, showed that both films were highly (104)-oriented. Compared to the BIT films, the BTH films have significantly enhanced electrical properties with 3 times larger remanent polarization (2Pr=45.7μC/cm2), 0.7 times smaller coercive field (2Ec=184 kV/cm) and better fatigue endurance(11.4% degradation). These results showed that equal-valence B-site Hf-substitution is effective to improve the electrical properties of BIT. By inserting effective buffer layers, BIT film can epitaxially deposited on GaN substrates. The integration growth of ferroelectric films with GaN may supply a possible way to realize multifunctional electronic devices.

5:40 PM

Synthesis and Characterization of ZnO:GaN Thin Films for Photoelectrochemical Water Splitting: Nuggehalli Ravindra[1]; *Sudhakar Shet*[1]; Kwang-Soon Ahn[2]; Yanfa Yan[2]; John Turner[2]; Mowafak Al-Jassim[2]; [1]New Jersey Institute of Technology; [2]National Renewable Energy Laboratory

ZnO:GaN thin films with significantly reduced bandgaps were synthesized by using ZnO and GaN targets at 100°C followed by postdeposition annealing at 500°C in ammonia for 4 hr. All the films were synthesized by rf magnetron sputtering on F-doped tin oxide-coated glass. We found that ZnO:GaN thin films exhibited narrowed bandgap, as a result showed improved PEC response, as compared to ZnO thin film. Furthermore, ZnO:GaN thin films with various bandgap were realized by varying the N2 mass flow rate in mixed N2 and O2 chamber ambient.

Recycling of Electronic Wastes: Mechanical Recycling and Pyrometallurgical Recycling

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS Materials Processing and Manufacturing Division, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Lifeng Zhang, Missouri University; Fay Hua, Intel Corp; Oladele Ogunseitan, University of California, Irvine; Gregory Krumdick, Argonne National Laboratory

Monday PM — Room: 2024
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Oladele Ogunseitan, University of California-Irvine; Fay Hua, Intel Corp

2:00 PM Introductory Comments

2:05 PM

Green Combustion of Waste Printed Circuit Boards: *Lifeng Zhang*[1]; Xiangjun Zuo[1]; [1]Missouri University of Science and Technology

In this paper, the pyrometallurgical recycling of Printed Circuit Boards (PCB) was executed. The mechanisms of thermal degradation and combustion were investigated using TG/DTA and MS. Some chemical powders, such as Na2CO3, NaHCO3, NaOH and CaCO3 were used to control the exhausted toxic gas, such as Br2. Finally a green combustion process for the recycling of waste PCB was proposed.

2:25 PM Question and Answer Period

2:35 PM

Industrial Recycling of Electronic Scrap at Boliden's Rönnskär Smelter: *Theo Lehner*[1]; Hans Henriksson[1]; [1]Boliden Mineral AB

Recycling plays a central part in supplying the smelters with raw materials. Boliden has developed proprietary technology to extract metal values from complex secondary raw materials with minimal impact on the environment as well as on the workers health and safety. Recycling plays also a central part in the political and public perception of our business. Secondary raw materials collected on a global scale include secondary metal concentrates, metal scrap, electronic scrap; to mention a few. In the presentation the operations and performance will be presented and the challenges as well as opportunities along the road map will be discussed.

2:55 PM Question and Answer Period

3:05 PM

Size-Dependent Melting Characteristics of Lead-Free Solder Alloys in Microelectronics: *Rami Chukka*[1]; NRMR Bhrargava[1]; [1]A U College of Engineering

Reduction of melting point for lead free solder alloys helps (Sn–Ag–Cu, Sn–Cu and Sn–Ag alloys) to work efficiently for soldering of modern fine pitch electronic component design. Because lead free solders generally have liquidus points of 220ºC or higher, compared to the 183°C melting point of eutectic tin-lead solder, that much of heat certainly damage electronic devices. Present work involves in size reduction of Tin based alloys to nano scale level in order to increase the surface area of crystalline materials it eventually results in lowering melting point. Nanoparticles of Sn3.5Ag0.5Cu, Sn3.5Ag and Sn0.7Cu lead free

solder alloys were produced by mechanical attrition using high energy planetary ball mill. The melting temperature depression due to nano size effect was calculated with differential scanning calorimeter (DSC) analysis and particle size is analyzed by using XRD data.

3:25 PM Question and Answer Period

3:35 PM
Thermal Behavior of Mixed Household Portable Batteries: Denise Espinosa[1]; *Jorge Tenório*[1]; [1]University of São Paulo

The recycling of waste of electric and electronic equipment (WEEE) is one of the main modern challenges. Among the variety of WEEE, batteries can be set apart due to their composition and their growing demand along with portable devices. The objectives of this work are to characterize the thermal behavior of a sample of mixed types of batteries using a thermobalance (TGA). The results of tests showed that Cd evaporated up to 800ºC, Zn evaporation occurred mainly above 900ºC and manganese oxides were prereduced to MnO at temperatures above 900ºC.

3:55 PM Break

4:15 PM Invited
Preparation of Cadmium Ingot and Nickel Powder from Spent Cd-Ni Batteries: Li Changdong[1]; *Xu Shengming*[2]; Huang Guoyong[1]; Zhang Lifeng[3]; Xu Gang[2]; Tan Jingjin[1]; [1]Foshan Brunp Nickel & Cobalt Technology Co., Ltd; [2]Tsinghua University; [3]Missouri University of Science and Technology

Cadmium ingot and Nickel powder were synthesized by high-temperature distillation, solvent extraction and liquid phase reducing method with Spent Cd-Ni Batteries as raw material. The impacts of the temperature in the distilling stage , the concentration of H2SO4 in the leaching stage, the pH value in the extracting stage and the dosage of hydrazine in the reducing stage were discussed respectively; moreover, the samples were characterized by the means of X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic absorption spectrometry (AAS) and inductively coupled plasma-atomic emission spectrometry (ICP-AES). The results showed that the recoverying efficiencies of Cd and Ni reach 99.5% and 99.3%; and the purities of Cd ingot and Nickel powder are higher than 99.90% and 99.80% respectively.

4:35 PM Question and Answer Period

4:45 PM
Materials Recovery from Electronics Scrap via Mechanical Separation and Froth Flotation Technology: *Joseph Pomykala*[1]; Bassam Jody[1]; Jeffrey Spangenberger[1]; Edward Daniels[1]; [1]Argonne National Laboratory

Argonne National Laboratory has developed a two-stage process for recovering materials from end of life electronics. The electronics are first mechanically processed to recover the residual metals and to produce a concentrated polymer fraction. Applying a developed froth flotation technology to the concentrated polymer fraction resulted in the separation and recovery of various polymers. This technology, which separates overlapping density materials from one another in an aqueous solution, has been successful in the separation and recovery of polymers such as acrylo-nitrile-butadiene-styrene (ABS) from polystyrene. This work was sponsored by the U.S. Department of Energy Office of Energy Efficiency and Renewable Energy. This paper describes three case studies of different sourced materials processed through the Argonne National Laboratory pilot plant facility. Results indicated that over 90% of the metals were recovered, the polystyrene was recovered at high concentrations and the ABS polymers could be recovered at concentrations greater than 95%.

5:05 PM Question and Answer Period

Shape Casting: Third International Symposium: Processes

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Solidification Committee, TMS: Aluminum Processing Committee
Program Organizers: John Campbell, University of Birmingham; Paul Crepeau, General Motors Corp; Murat Tiryakioglu, Robert Morris University

Monday PM — Room: 2011
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Alan Druschitz, University of Alabama at Birmingham

2:00 PM Introductory Comments

2:05 PM
Influence of Hydrogen Content and Bifilm Index on Feeding Behaviour of Al-7Si Alloy: *Derya Dispinar*[1]; Arne Nordmark[1]; Jorunn Voje[2]; Lars Arnberg[3]; [1]SINTEF; [2]Elkem Aluminium; [3]Norwegian University of Science and Technology

The relationship between 'hydrogen-porosity' and 'Sr modification-porosity' has long been investigated. In this study, this phenomenon has been investigated in terms of bifilm content. A feeding test with deliberately inadequate feeding to promote some degree of shrinkage porosity has been used to compare feeding and porosity of Al-7Si alloy in gravity die casting. A melt with three different hydrogen contents was prepared by degassing first and then upgassing to low, mid and high hydrogen levels (0.1, 0.2 and 0.3 respectively) with Ar-10%H_2 mixture. Reduced pressure test samples were taken for bifilm index calculation and 10 tensile test bars were cast into sand moulds for mechanical testing. The results showed that feedability was increased with B-grain refining (SiBloy alloy) than the conventional Ti-B grain refined. However, pore distribution was scattered in both cases when the alloys were Sr-modified.

2:30 PM
Oxide Entrainment Structures in Horizontal Running Systems: *Carl Reilly*[1]; Mark Jolly[1]; Nick Green[1]; [1]The University of Birmingham

During the transient phase of filling a casting running system surface turbulence can cause the entrainment of oxide films into the bulk liquid. Research has shown that these are detrimental to the material's integrity. Common mechanisms for this entrainment include returning waves, arising during filling of the runner bar, and plunging jets, found when pouring into a basin. One of these, the returning wave, has been studied in greater depth, using real-time X-ray and process modelling techniques alongside the application of physical principals. It has been concluded that when developed, returning waves cannot attain the more stable and less entraining tranquil flow regime desirable in the running system of castings.

2:55 PM
Degassing: A Critical Stage in the Manufacturing of Al-Si-Cu Alloys for Automotive Castings: *Eulogio Velasco*[1]; Rocio Valdes Lopez[2]; Jose Nino[2]; [1]Texas State University; [2]NEMAK

In the manufacturing of aluminum blocks for automotive engines, porosity requirements on sealing surfaces and other critical areas are so stringent that special controls and special processes are required to remove hydrogen and inclusions from the molten aluminum. Several trials and adjustments in the degassing parameters and melting practices were evaluated at a production foundry. To evaluate the degassing process, density measurements were performed and to evaluate the inclusion content, fluidity and PreFil™ measurements were performed. Additionally, the mechanical properties of the casting in the heat treated condition were determined. The relationship between the density as determined by the reduced pressure test (RPT) and actual hydrogen content as determined by an AlScan unit was established for A319 aluminum alloy. The results of the mechanical property tests showed a direct relation with the metal quality of the alloy.

3:20 PM
Heat Treatment of A356.2 Aluminum Alloy: Effect of Quench Rate and Natural Ageing: *Manickaraj Jeyakumar*[1]; Mohamed Mousa[1]; Mohamed Hamed[1]; Sumanth Shankar[1]; [1]LMCRC-McMaster University

A356.2 aluminum alloy is a popular commercial alloy used for structural shaped castings in automotive applications. The heat treatment of this alloy is critical to obtain the desired mechanical and performance properties. The three stages of heat treatment include, solutionizing, quenching and artificial ageing. In this study, the effect of quenching in water at 80°C and forced air at 33.5 m/s velocity is quantified. Further, the effect of natural ageing treatment prior to the artificial ageing is also quantified. It is observed that the mechanical properties of the castings are significantly affected by both the rate of quenching and the natural ageing treatment. Mechanical properties and hardness values are presented for standard tensile test specimen cast using gravity permanent mold process and subjected to various heat treatment conditions.

3:45 PM Break

3:55 PM
Process Parameters Study for Net-Shape Steel Casting: Von Richards[1]; *Simon Lekakh*[1]; Darryl Kline[1]; K. Chandrashekhara[1]; Jian Chen[1]; [1]Missouri University of Science & Technology

The objective of this research was the experimental study and computational modeling of process parameters of net-shape steel casting with complicated geometry. Gas permeability, burst pressure (pressurize water), and mechanical and physical properties at room and high temperatures for ceramic shells were evaluated for different ceramics. The experimental data was used to simulate stresses in the ceramic shell, mold filling, and solidification using ABAQUS and MAGMA software. Models were verified by pouring steel into ceramic molds of differing geometries and monitoring with electrical sensors and thermopiles connected to a high-speed DAQ. The results will be used for the optimization of an industrial process.

4:20 PM
Improving Build Speed in Rapid Freeze Prototyping through Increase of Heat Transfer: *Ming Leu*[1]; Sriram Isanaka[1]; Von Richards[1]; [1]Missouri University of Science and Technology

The heat transfer in the Rapid Freeze Prototyping (RFP) process has been significantly increased for improvement of build speed and part accuracy. RFP is a solid freeform fabrication process in which water droplets are deposited and solidified layer-by-layer to form three-dimensional ice patterns for investment casting. A mechanism has been devised to cool the substrate to as low as -140°C. Chilling plates were developed to enable effective transfer of heat with the aid of conduction. To ensure that deposited water does not freeze to the chilling plate, various surface coats were investigated. The most effective interface material was identified using contact angles measured using high resolution digital photography. The experimental results were substantiated with model based simulations performed using Fluent and Gambit. The improvements in build speed and dimensional accuracy after incorporating the above changes were measured and presented.

4:45 PM
Cooling Properties of Frozen Sand Molds for Casting of Lead Free Bronze: *Hiroyuki Nakayama*[1]; Shuji Tada[1]; Toshiyuki Nishio[1]; Keizo Kobayashi[1]; [1]National Institute of Advanced Industrial Science and Technology

A frozen mold is an advanced sand mold provided by freezing the mixture of sand and water. Bronze cast plates, which had dimensions of 30x150x5 mm, were produced through the frozen mold casting process. The temperature transition of the cast plates just after pouring were measured at three positions, near the gate, the center and the far side, by high speed recorder with sampling time of 1 ms. The temperature rapidly decreased on the initial stage. Subsequently, the slight temperature raise was observed in the near side of the gate. However, this phenomenon was not observed in the far side, where the temperature of melt was already decreased. In contrast, the above temperature raise was not observed at any positions in the casting by a green sand mold.

Structural Materials Division Symposium: Advanced Characterization and Modeling of Phase Transformations in Metals in Honor of David N. Seidman on his 70th Birthday: Thermodynamics of Phase Transformations

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Chemistry and Physics of Materials Committee
Program Organizers: Robert Averback, University of Illinois, Urbana-Champaign; Mark Asta, University of California, Davis; David Dunand, Northwestern University; Ian Robertson, University of Illinois at Urbana-Champaign; Stephen Foiles, Sandia National Laboratories

Monday PM — Room: 3000
February 16, 2009 — Location: Moscone West Convention Center

Session Chair: Mark Asta, University of California, Davis

2:00 PM Invited
The Early Years: *David Brandon*[1]; [1]Israel Institute of Technology

David Seidman has had a remarkably successful career, marrying atomistic studies of phase stability to kinetics and thermodynamics. But David's seminal years were spent at the university of Illinois, completing his doctorate under the leadership of Bob Balluffi, and at Cornell, 'learning' field-ion microscopy and developing the expertise that would later allow him to build his first atom probe and then expand these facilities at Northwestern, to create the world's leading international center for atom probe tomography. I met David Seidman when he was a graduate student at Illimois, where his room-mate was a former student of mine, Piers Bowden. My last 'professional' interaction with him was when I spent three weeks at Cornell, as a rather ineffectual 'consultant' on field-ion microscopy. Our shared interests in microstructural characterization would not have sufficed to maintain our friendship, but our shared interest in the State of Israel certainly has.

2:25 PM Invited
Thermodynamic Properties of Grain Boundaries from Atomistic Simulations: *Jeffrey Hoyt*[1]; D.L. Olmsted[2]; S. Jindal[3]; M. Asta[3]; A. Karma[4]; B.B. Laird[5]; [1]McMaster University; [2]Sandia National Laboratories; [3]University of California, Davis; [4]Northeastern University; [5]University of Kansas

Molecular dynamics (MD) and Monte Carlo simulations on an embedded atom method model of pure Ni have been used to study two thermodynamic properties of grain boundaries at elevated temperatures. First, premelting of a high energy Σ 9 twist boundary has been investigated and, in agreement with the classic experiment of Hsieh and Balluffi, premelting has been observed at temperatures very close to the bulk melting point. In addition, by monitoring the width of the liquid-like layer over the course of lengthy MD runs, the disjoining potential between the two solid-liquid boundaries is derived. In agreement with phenomenological models, the disjoining potential was found to decay exponentially with the premelted layer width. Second, a lattice switch Monte Carlo technique has been used to compute the excess boundary free energy as a function of temperature for the Σ 3 coherent twin.

2:55 PM Invited
Chemical Short-Range Ordering in Liquid-Phase Ni Alloys: *Dallas Trinkle*[1]; Mark Asta[2]; Christopher Woodward[3]; [1]University of Illinois, Urbana-Champaign; [2]University of California, Davis; [3]Air Force Research Laboratory

First-principles modeling provides new predictive capabilities and atomistic-scale insight into the behavior of alloys in the liquid phase. In support of a current effort aimed at the development of validated mathematical criteria for predicting the formation of solidification defects in Ni-based superalloys, ab-initio molecular dynamics (AIMD) simulations have been performed for elemental, binary and ternary alloys of Ni with Al, W, Re, and Ta, as well as a RENE-N4 multi-component superalloy, to compute equations of state, measure diffusion, and quantify short-range chemical and structural order at temperatures of 1830 and 1750 K. Structural analysis based on radial distribution functions augmented with common-neighbor analysis and bond angle distributions reveal a strong tendency for icosahedral short range order for Ni-W and Ni-Re alloys.

Finally, short-range chemical ordering and neighbor distances for solutes are compared with elemental liquids for binary, ternary, and complex alloys.

3:25 PM Break

3:45 PM Invited
Atomistic Modeling of Interfacial Thermodynamics: *Y. Mishin*[1]; T. Frolov[1]; [1]George Mason University

We present thermodynamic relations for the free energy, stress, strain, formation volume, segregation and other excess properties of interfaces in non-hydrostatically stressed solid phases in forms convenient for their atomistic calculations. Cahn's method of determinants permits easy access to all interface properties from "raw" atomistic data without having to compute the interface profiles. The equations are applied to examine the temperature and composition dependencies of interface free energy, interface stress, interface strain and interface segregation for a few free surfaces, grain boundaries, coherent phase boundaries and solid/liquid interfaces with different crystallographic orientations. The method is also used to study surface pre-melting and the orientation dependence of the solid/liquid interface stress by molecular dynamics and grand-canonical Monte Carlo methods. Extensions of the method to multi-component systems with the substitutional, interstitial and mixed solubility mechanisms are discussed.

4:15 PM
Modeling of Diffusion along Triple Junction Lines: *T. Frolov*[1]; Y. Mishin[1]; [1]George Mason University

It has long been believed that diffusion along triple junction lines in metals is much faster than grain boundary diffusion. The anomalously high diffusivity in some nano-crystalline materials was sometimes attributed to a contribution of triple junctions. There have been very few experimental measurements and no atomistic calculations of triple-junction diffusion. As a result, no reliable self-diffusion coefficients along triple junctions in metals are currently available. We present results of molecular dynamics simulations of triple-junction and grain-boundary diffusion in copper over a wide temperature range. The unexpected result is that, although the diffusion coefficients in triple junctions are larger than in high-angle boundaries, they are of the same order of magnitude at all temperatures studied (700-1320 K). Thus the role of triple-junction diffusion in polycrystalline materials might be overestimated. Possible consequences of this finding are discussed. We have also studied pre-melting of triple-junctions and its effect on the diffusivity.

4:30 PM Invited
Computational Approach to Phase Transformations at the Nanoscale: *Alfredo Caro*[1]; Paul Erhart[1]; Babak Sadigh[1]; Magdalena Caro[1]; [1]LLNL

The computational study of phase transformations is today a standard approach that provides detailed pictures of complex processes at scales going from the continuum down to the electronic structure. Since modeling phase transformation at the nanometer scale requires atomic resolution, there is an increasing interest on developing models for atomic interactions that translate the information obtained at the electronic scale into reliable classical potentials, able to carry information about thermodynamics of multicomponent systems. Concurrently, adequate tools to resolve the time and length scales required for these studies are being developed. Codes for massively parallel Metropolis Monte Carlo algorithm are available today and provide insight into equilibrium properties. The situation is not yet so developed for massively parallel kinetic Monte Carlo that would also provide kinetic information. In this talk I will review our work in nanophase alloys, from small clusters to bulk nanophases, developing potentials for alloys and codes for large systems, and our applications for grain boundary and surface segregation, corrosion, and radiation damage in steels.

5:00 PM Invited
Free Energy Calculations for Rreactions of Lithium-Ion-Battery Electrode Materials in Acid: Li(1+x+y)Mn(2-x)O4 Spinel: *Roy Benedek*[1]; [1]Argonne National Laboratory

The lithiated transition metal oxides employed as cathodes in lithium-ion batteries react with aqueous acid by dissolving, or exchanging protons for lithium. We have previously developed a method to calculate free energies for reactions in acid of lithiated transition metal oxides by applying first principles calculations for the solid phases and tabulated thermochemical data for aqueous species. In the present work, this approach is applied to calculate the reactions of LiMn2O4 spinel, as a function of excess or deficiency of lithium. Our results show that, as expected, the dissolution reaction driving force is maximum for Li2Mn2O4, for which all Mn is available for disproportionation to divalent and tetravalent states. We find however, that the driving force for dissolution decreases less for overlithiated compositions than might be expected if disproportionation were the only factor involved. Unlike the dissolution reaction, protonation is unfavorable at almost any composition.

5:30 PM
Antiferromagnetic Transition and Martensitic Transformation in Mn-Rich g-MnFe Alloy: *Ji Zhang*[1]; [1]Shanghai Jiao Tong University

This paper attempts to pay attention to research the effect of antiferromagnetic transition on martensitic transformation. Observation of Transmission Electron Microscopy (TEM) and the measurement from Dynamic Mechanical Analysis (DMA) showed that there are (011) fct twins of martensitic transformation in the alloy. Experiments of electrical resistance and Differential Scanning Calorimeter (DSC) show that the martensitic transformation temperature (Ms) and reverse martensitic transformation temperature (Af) in the Mn-rich g-MnFe alloy is closed to each other or almost coincide, and direct and reverse martensitic transformation are similar to the second order typed-continuous transition. Because there is the coupling between first order martensitic transformation and second order antiferromagnetic transition, the temperature shape memory effect of the alloy showed a hysteresis-free characteristics in dilatation measurement. And there also is a magnetic-field induced shape strain (MFIS) under applied magnetic field and the maximum MFIS reached 1.6% at the applied field of 3.8 T.

5:45 PM
Rigorous Simulations of Kirkendall Effects in Polycrystalline Solids: *Hui-Chia Yu*[1]; Anton Van der Ven[1]; Katsuyo Thornton[1]; [1]University of Michigan

The Kirkendall effect stems from the biased interdiffusion in which the two atomic species have different exchange rates with vacancies that mediate diffusion. The vacancy injection/annihilation at their sources/sinks result in the so-called Kirkendall shift and deformation. In this study, we investigate the effect of grain boundaries acting as vacancy sources and sinks on the Kirkendall effect through computer simulations. The vacancy source effects on biased diffusion due to net vacancy flux and intermixing are analyzed. The result shows a new mechanism for the enhanced grain boundary diffusion. Furthermore, phase field simulations are employed to study the Kirkendall deformation based on the treatment that assumes grain boundaries to be discretely distributed vacancy sources/sinks. The results demonstrate the local expansion and contraction near grain boundaries lead to a different deformation pattern from the conventional model that assumes a continuous vacancy source/sink within the bulk of solids.

Synergies of Computational and Experimental Materials Science: Three-Dimensional Materials Science II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Katsuyo Thornton, University of Michigan; Henning Poulsen, Risoe National Laboratory; Mei Li, Ford Motor Co

Monday PM — Room: 3003
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Jonathan Madison, The University of Michigan; Peter Voorhees, Northwestern Univ

2:00 PM Invited
Combining Serial Sectioning, EBSD Analysis, and Image-Based Finite Element Modeling: *Alexis Lewis*[1]; David Rowenhorst[1]; Andrew Geltmacher[1]; George Spanos[1]; [1]Naval Research Laboratory

Recent work combining 3D data obtained from serial sectioning, electron backscatter diffraction (EBSD), and finite element modeling (FEM) of materials microstructures has led to new advances in the fundamental understanding of structure-property relationships in three dimensions. Current techniques, both experimental and computational, will be described, with emphasis on imaged-based FE methods which use experimental data from these 3D reconstructions as the initial input conditions for simulations of the mechanical response of

3D microstructures to various externally applied loading conditions. Specific examples from work on a beta-Titanium alloy are utilized to illustrate the capabilities of these experimental and modeling techniques, the challenges and the solutions associated with these methods, and the types of results and analyses that can be obtained by the close integration of experiments and simulations.

2:40 PM

Modeling the Influence of Microstructure on Flow Stress in Ti-6Al-4V Alloy by Neural Networks: *N. S. Reddy*[1]; C. H. Park[2]; Y. H. Lee[3]; Y. I. Son[4]; C. S. Lee[2]; [1]Alternative Technology Laboratory, Pohang University of Science and Technology; [2]Department of Materials Science and Engineering, Pohang University of Science and Technology; [3]Wire Rod Research Group; [4]Agency for Defence Development

Neural networks (NN) were used to model flow stress in Ti-6Al-4V alloy with equiaxed, maretnsite and widmanstätten microstructure as initial microstructures. Continuous compression tests were performed over a wide range of temperatures (700-1100°C) with strain rates of 0.001-100 s-1 and true strains of 0.1-1.4. These tests have been focused to obtain flow stress data under varying conditions of strain, strain rate, temperature, and initial microstructure to train NN model. The feed-forward neural network consisted of two hidden layers with a sigmoid activation function and back propagation training algorithm used. The NN model was successfully trained across (α+ β) to β phase regimes and across different deformation domains. Sensitivity analysis was carried on trained model to study the quantitative effect of microstructure on flow stress. Results show that the NN model can correctly reproduce the flow stress in the sampled data and it can predict well with the non-sampled data.

3:00 PM

Microstructural Analyses Using 3D Image-Based Finite Element Modeling: *Andrew Geltmacher*[1]; Alexis Lewis[1]; Muhammed Qidwai[2]; David Rowenhorst[1]; George Spanos[1]; [1]Naval Research Laboratory; [2]SAIC

Image-based finite element (FE) models derived from 3D reconstructions of real material microstructures have been used to simulate the mechanical response in several advanced engineering alloys. In the work presented here, 3D image-based FE modeling of a single-phase beta Titanium alloy was used to determine the relationships between microstructure, crystallography, grain morphology, and mechanical response. Initial simulations show high stress concentrations at particular grain boundaries and junctions, and the combined 3D microstructural and crystallographic information at these areas of high local stress is used to determine correlations between the measured microstructure and the simulated response. Results will be presented for a number of representative volumes sampled from a larger reconstructed volume which contains thousands of grains. The effects of volume sampling size, simulation parameters, and mesh generation techniques on the observed material response will also be discussed.

3:20 PM

Numerical and Experimental Investigation of Deformation Behavior of a Duplex Microstructure of a γ-TiAl Alloy Using Crystal Plasticity and Two Scale Modeling Approach: *Mohammad Rizviul Kabir*[1]; Liudmila Chernova[1]; Nikolay Zotov[2]; Marion Bartsch[1]; [1]German Aerospace Center; [2]Ruhr-Universität Bochum

The 3-dimensional microstructure of a duplex γ-TiAl has been modeled in a parameterized FE-model using a two scale approach (FE2-approach) for micro and macro coupling. The most important microstructural features, such as α_2 and γ-phases, their volume percents, orientations, lamellar and globular arrangements, were representatively incorporated. The microstructure information was experimentally determined by SEM, TEM and EBSD analysis. For describing the micromechanics of the phases a continuum based crystal plasticity model was used. The model parameters were validated on the microscopic level by nano-indentation testing and on the macroscopic level by tensile tests at room temperature. The global deformation behavior was explained fairly well by the slip interactions, the local stresses, and the local strains predicted by the modeling approach. The model can be used for optimizing the microstructure of polycrystalline materials for components.

3:40 PM Break

4:00 PM Invited

Combined 3D X-Ray Microscopy and Dislocation Dynamics Simulation Investigation of the Fundamental Aspects of Deformation in Copper: *Bennett Larson*[1]; Jie Deng[2]; Anter El-Azab[2]; [1]Oak Ridge National Laboratory; [2]Florida State University

Submicron resolution 3D x-ray microscopy measurements have been combined with dislocation dynamics simulations to initiate an investigation of the fundamental aspects of deformation in metals. 3D x-ray microscopy measurements of local plastic deformation were performed with 0.5 µm resolution (~0.3 µm beam size) on initially dislocation free Cu single crystals that were deformed in compression to strains varying from 1% to 7.6% along the [100] direction. Dislocation dynamics simulations were performed for [100] axial deformation of fcc Cu for strain values ranging up to 1.6%. The overlapping strain magnitudes for the measurements and simulations provide a direct and quantitative link between nondestructive 3D x-ray microscopy measurements and first principles simulations of deformation. Quantitative comparisons between the measured and simulated local lattice curvatures will be presented in graphical and statistical form. Research supported by the US DOE Office of Science, Basic Energy Sciences, Division of Materials Sciences and Engineering.

4:40 PM

X-Ray Synchrotron Tomography for Three Dimensional (3D) Microstructure Visualization and Modeling of Deformation in Metal Matrix Composites: *Flavio Silva*[1]; Jason Williams[1]; Nikhilesh Chawla[1]; Pedro Portella[2]; Bernd Mueller[2]; [1]Arizona State University; [2]Federal Inst for Mat Rsch & Testing BAM

Current analytical and numerical techniques simplify the complex and heterogeneous microstructure of composite materials. These simplifications make modeling and analysis more efficient and straightforward, but fail to accurately predict the effective properties and local damage behavior which are inherently dependent on microstructure. We report on a novel methodology that addresses the critical link between microstructure and deformation behavior, by using a three-dimensional (3D) virtual microstructure as the basis for a robust model to simulate damage caused by deformation. The approach involves capturing the microstructure by novel and sophisticated x-ray tomography techniques, followed by image analysis, 3D reconstruction of the microstructure, and incorporation into a powerful finite element modeling code for simulation. We will present a case study based on uniaxial tensile deformation of SiC particle reinforced Al alloy matrix composites. In particular, the damage in the form of particle fracture will be described.

5:00 PM

Assessing the Sensitivity of FEM-Based Crystal Plasticity Models to Microstructures: A Multi-Scale Crystal Plasticity Model Combined with Experimental Methods: *Remi Dingreville*[1]; Corbett Battaile[1]; Luke Brewer[1]; Elisabeth Holm[1]; [1]Sandia National Laboratories

In this paper, we examine the effect of the microstructure on the elasto-plastic response of polycrystals by combining a non-local crystal plasticity simulations with experimental microscale characterization. The morphology and deformation behavior of polycrystals are characterized using electron back scatter diffraction (EBSD) and in situ tensile testing, and finite element simulations of the deformation are performed using an augmented kinematics framework and a non-local crystal plasticity constitutive treatment allowing a natural description of microstructure by featuring low angle sub-grain boundaries and high angle grain boundaries. Comparisons between experimental observations and computational simulations are provided at the macroscopic and microscopic scale. The contrasts between the experimental investigations and modeling technique highlight the strong and weak points of the theoretical framework used.

5:20 PM

A Failure Surface Calibrated with Mechanical Testing, Finite Element Analyses, and Metallography: *Matthew Hayden*[1]; Xiaosheng Gao[2]; Charles Roe[1]; [1]Naval Surface Warfare Center; [2]The University of Akron

Ductile fracture is a complex phenomenon currently implemented in structural codes as empirical fits of material testing. Most often, these fits do not fully capture the effects of stress state, nor are they tied to material microstructure. This study develops a three-dimensional failure surface calibrated to 5083-H116

aluminum with a suite of tension, torsion, and compression tests over a broader range of stress states. Finite element models coupled to material microstructural topology analyze each specimen geometry to calculate local stress state and failure strain at the region of failure initiation.

Transformations under Extreme Conditions: A New Frontier in Materials: Melting and Solidification II

Sponsored by: The Minerals, Metals and Materials Society, ASM International, ASM Materials Science Critical Technology Sector, TMS Materials Processing and Manufacturing Division, TMS/ASM: Phase Transformations Committee
Program Organizers: Vijay Vasudevan, University of Cincinnati; Mukul Kumar, Lawrence Livermore National Laboratory; Marc Meyers, University of California-San Diego; George "Rusty" Gray, Los Alamos National Laboratory; Dan Thoma, Los Alamos National Laboratory

Monday PM — Room: 3001
February 16, 2009 — Location: Moscone West Convention Center

Session Chairs: Robert Hixson, Naval Postgraduate School; Jorg Wiezorek, University of Pittsburgh

2:00 PM Invited
Dynamics of Ultrafast Melting and Solidification in Metals: *Robert Averback*[1]; Wai-Lun Chan[1]; David Cahill[1]; Yinon Ashkenazy[2]; [1]University of Illinois; [2]The Hebrew University of Jerusalem

Melting and solidification in metals have been investigated using a combination of experimental and simulation methods. The experimental work measures the velocity of the advancing melt front in Ag following excitation with a femtosecond laser and the subsequent resolidification as a function of undercooling. The liquid-solid transitions are followed using third harmonic generation of light. Critical to understanding these experiments is the thermal transport of energy under such extreme conditions. MD simulations were used to examine resolidification behavior as a function of undercooling in a number of metals, both FCC and BCC. We illustrate that the interface kinetics can be divided into three temperature regimes, with the lowest being controlled by diffusion. We also demonstrate a correlation in the low-temperature regime between the migration process controlling the crystallization and equilibrium point defect properties.

2:35 PM
Microstructural Changes in Al Thin Films during Pulsed Laser Induced Rapid Lateral Solidification: Andreas Kulovits[1]; John Leonard[1]; *Jorg Wiezorek*[1]; [1]University of Pittsburgh

Sputtered Al films (70-140nm thick) with and without silica cap-layers were melted using a single excimer laser (KrF) pulse with 28ns duration. SEM and TEM studies revealed RS microstructures consisting predominantly of through-film thickness high aspect ratio grains (~10-20μm long, 1μm wide) with an in-film-plane columnar structure, containing a variety of crystal defects. XRD and SEM EBSD measurements were used to quantify texture changes. Theoretically estimated re-solidification times of 100 to 500ns imply extremely fast solidification front velocities (~102m/s). The thin film sample geometry we successfully used for pulsed laser melting of Al and also Cu, Ag, Cr, Ni is suitable for dynamic in-situ experimentation using a novel in-situ TEM instrument or Dynamic TEM (DTEM). The DTEM uniquely offers the combination of nanoscale temporal and spatial resolution critically required for studying directly by experiment the dynamics associated with these unique and extremely rapid phase transformations.

2:55 PM
A Computational Model for Thermal Response of Semi-Transparent Materials to Laser Processing: *Jeffrey Colvin*[1]; James Stölken[1]; [1]Lawrence Livermore National Lab

Lasers are widely used to modify the internal structure of semi-transparent materials for a wide variety of applications, including waveguide fabrication and laser glass damage healing. The diffusion approximation used in past models to describe radiative cooling is not adequate for these materials, particularly near the heated surface layer. In this paper we describe a new computational model based upon solving the radiation transport equation by the Pn method with ~1000 photon energy bands. The model accounts for the temperature-dependent absorption of infrared laser light and subsequent redistribution of the deposited heat by both radiative and conductive transport. We present representative results for fused silica irradiated with 1-2 W for 10 s pulse durations. This work was performed under the auspices of the US Department of Energy by Lawrence Livermore National Laboratory under contract No. DE-AC52-07NA27344, with support received from LDRD Project #08-ERD-057.

3:15 PM Invited
Experimental and Theoretical Research on Shock-Induced Phase Transformation in LSD: *Wenjun Zhu*[1]; Jianbo Hu[1]; Xianmin Zhou[1]; Jun Li[1]; Xinlin Cui[1]; Chenda Dai[1]; Hongliang He[1]; [1]Laboratory for Shock Wave and Detonation Physics Research

Shock-wave has advantage to generate high pressure condition, where materials can undergo rich pressure-driven phase transformations. However, it is critical to measure the phase transformation pressure and to determine the new phase structure as well as phase boundaries due to very short time. We present three examples to show recent experimental and theoretical progresses on shock-induced phase transformation in our lab (LSD). (1) The classic configuration of reverse-impact experiments has been modified to enhance the accuracy of high-pressure sound velocity measurements. The shock induced bct to bcc phase transformation with slight change of volume in tin is distinctly detected through sound velocity measurements. (2) The phase transformation pressure for LiTaO3 and the structure have been determined by combination methods of shock velocity (D) versus particle velocity (Up) relation measurements and first-principle calculations. (3) Effects of defects to shock-induced phase transformation in iron have been investigated by molecular dynamics (MD) simulations.

3:50 PM Break

4:05 PM Invited
Shock Wave Exploration of the High Pressure Phases of Carbon: *Marcus Knudson*[1]; Mike Desjarlais[1]; Dan Dolan[1]; [1]Sandia National Laboratories

The high energy density response of diamond has gained interest of late due to its relevance to planetary astrophysics and the possible use of diamond an ablator material in inertial confinement fusion capsules. Recently, experiments utilizing an ultra-high flyer plate capability at the Sandia Z accelerator were performed to determine the Hugoniot and the shock melting properties of polycrystalline diamond. Composite aluminum/copper flyer plates were used to shock load diamond samples to pressures ranging from 5 to 14 Mbar. Multiple samples and fast diagnostics provided Hugoniot measurements with ~1% accuracy in density. This work provides compelling evidence for the existence of a diamond-bc8-liquid triple point along the coexistence region of the Hugoniot, at a pressure and density of ~850 GPa and ~6.5-6.6 g/cc. These high precision Hugoniot measurements at multi-Mbar pressures allow for high fidelity comparisons with recent quantum molecular dynamics calculations, and provides the first experimental evidence of a high pressure solid phase of carbon beyond that of diamond.

4:40 PM Invited
Dynamic Solidification of Water under Quasi-Isentropic Compression: Heterogeneous Nucleation and Beyond: *Daniel Dolan*[1]; [1]Sandia National Laboratories

Solidification is well established under static compression, but has proven elusive under dynamic compression. Creating solidification states with dynamic compression is challenging, and these states are short lived, whereas freezing is generally a slow phase transition. Using quasi-isentropic compression, it is possible to bring liquid water from ambient conditions to states deep within the ice VII domain on nanosecond time scales. Under these conditions, water can be made to solidify in two ways. If an appropriate nucleator is present, metastable water transforms to a mixed phase over the course of 10-100 ns. Much faster solidification occurs if compressed water is driven beyond its metastable limit. Both types of transformation have been observed experimentally in water, and will be discussed in this presentation. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

5:15 PM

Liquid-Solid, Pressure-Induced Phase Transition for Water: Simulations: *Daniel Orlikowski*[1]; Jeff Nguyen[1]; Neil Holmes[1]; [1]Lawrence Livermore National Laboratory

Hydrodynamic simulations of liquid water undergoing a pressure-induced phase transition into ice VII are presented. From initial ambient conditions, the liquid system is quasi-isentropically compressed into its solid phase. To develop this modeling effort, we have used unique quasi-isentropic, light-gas gun data for water that have measured a phase-fraction along this path. Combining this information with the equilibrium equation of state (EOS), we have evaluated a possible kinetic model for its liquid to solid phase transition. Specifically, the simulations use two tabular single phase EOS's. To mitigate between the single phase EOS's during the mixed-phase intervals, a thermodynamic linear mixing scheme is used to compare to a simple kinetic model containing an time constant determined from experiment. The simulation models in one dimension the entire experimental setup, accounting for the wave interactions throughout the impactor and target, which are compared with experiment.

5:35 PM

Transient Ionization of Shock Compressed Water Near Planetary Isentropes: *Nir Goldman*[1]; Evan Reed[1]; Will Kuo[1]; Laurence Fried[1]; Christopher Mundy[2]; Alessandro Curioni[3]; [1]LLNL; [2]Pacific Northwest National Laboratory; [3]IBM Research

We report herein first principles simulations of water under shock loading near the isentropes of Neptune and Uranus. Accurate description of the chemical mechanism for the ionic conductivity at high pressures and temperatures is of particular importance to models of the planetary dynamo mechanism in these planets. Using a novel simulation technique for shock compression, we are able to make excellent comparison to the experimental results for the Hugoniot pressure, temperature and density final states. Our simulations resolve controversy by showing that a unimolecular mechanism for electric conduction dominates at high pressures along the shock Hugoniot. Near the approximate intersection of the Hugoniot and the planetary isentrope we observe high concentrations of negatively charged species that contribute electronic states near the band gap. Our results provide a microscopic picture of the chemistry at planetary depths of ca. 6000 km and greater.