2011 Aluminum Plenary Symposium: The 125th Anniversary of the Hall-Héroult Process

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizer: John Johnson, JCG

Monday AM
February 28, 2011

Room: 6B
Location: San Diego Conv. Ctr

Session Chair: John Johnson, Johnson's Consulting Group

8:30 AM Plenary
Paul Héroult: The Early Days of the European Aluminium Industry: *Claude Vanvoren*[1]; [1]Rio Tinto Alcan

Paul Héroult story is indistinguishable from the early days of the European aluminium industry in France and Switzerland. The personal engagement of the co-inventor of the electrolytic process, a true entrepreneur, tells a lot about his conviction on the bright future of the light metal. The lecture will come back on those adventurous early days and illustrate how they gave birth to an industry which since, relentlessly, devoted effort to innovate and facilitate access to a material which developed from semi-precious to commodity status in less than 125 years.

9:05 AM Plenary
Charles Martin Hall and Warren Haupin: Over 100 Years of Technical Innovation: *Gary Tarcy*[1]; [1]Alcoa

Two scientists at Alcoa stand above all others. The first, of course is Charles Martin Hall, one of the co-inventors of the electrolysis process that is still used today. C. M. Hall was also one of the co-founders of what was originally the Pittsburgh Reduction Company that later became the Aluminum Company of America (ALCOA). Looking back on some of his early work and in particular his invention one only wonders what he would think of what became a worldwide metals industry that in volume is only surpassed by steel. Although the process is essentially the same as it was invented 125 years ago. A second Alcoa scientist, Warren Haupin, was one of the essential people to increase the understanding of the process that has led to a multitude of incremental improvements over the last 60 years. The lecture will discuss the contributions and some antidotal information about both of these scientists.

9:40 AM Plenary
The Rise and Fall of the Knowledge Base for Aluminium Smelting – The Last 50 Years: *Barry Welch*[1]; [1]University of New South Wales- Australia, and Welbank Consulting Ltd - New Zealand

In the second half of the 1950's the space race was with us. Cheaper more efficient production of light metals (such as titanium, magnesium and aluminium) as well as the refractory metals and materials - including zirconium and titanium diborides for example - that would withstand extremely high temperatures became a major research thrust. As a flow-on from the European and Russian research eminence in these areas during the 1930's and 1940's, a few academic Western-world research "cells" (to pinch a George Bush phrase) specialising in the structure, properties and electro chemistry of molten salts were being established and active. The activities were heavily biased to molten fluorides included research aiming at developing more efficient nuclear reactors (ORNL) and a better understanding of aluminum smelting electrolyte. "Young-bloods" trained in academic institutions (especially the University of Toronto, the Technical University of Norway and Imperial College -London Penn State Univ., and MIT) spearheaded and intense research that developed an excellent understanding of the fundamentals of the properties of molten electrolytes and the electrochemistry. The glowing example was Kai Grjotheim's thesis in 1956. The Symposium on Aluminium organised by TMS in 1962 was the first one that brought together some of the academic understanding and industrial issues associated with aluminium smelting. Inspiring papers by Eric Ransley on titanium diboride (with concepts quite different to those that are being considered in the decades since), the reactivity and properties of carbon anodes and cathodes, and the options for maximising current efficiency stood alongside one on Bus-bar designs which initiated magnetic modelling of the cells - then computing was in its infancy. From these and other papers presented the growth in design features that enabled an increase in amperage took off. The impact of some of the "characters" who became leaders in the early 1960s, together with their contributions will be traced and highlighted. As also will be the decline that started following the change in emphasis introduced by aluminium industry leaders in the second half of the 1980s, to the point to day where it is harming performance and displaying a greater lack of understanding of core fundamentals than existed 50 years ago!

10:20 AM Break

10:30 AM Plenary
The Early Years of Light Metals, TMS: *Nolan Richards*[1]; [1]Richards & Associates

The symposium held in New York, 1962, "Extractive Metallurgy of Aluminum", under the auspices of the Metallurgical Society of AIME, was a ground breaking event in the U. S.A. for the entire aluminum industry. As a result of two-years of organizing, fifty five contributors, thirty one embracing the production of aluminum, freely shared results of research done within their companies or universities with this tentative opening up of communication and sharing that had occurred previously only in a few conferences in Europe. The amicable interactions and success of this meeting led to the beginning of annual meetings organized by the Light Metals Committee, the Metallurgical Society (TMS) of the American Institute of Mining Metallurgical and Petroleum Engineers Inc. (AIME). Before this conference, technologists involved in improving the operation, design and safety of alumina reduction cells, apart from their in-house, guarded data, had recourse to the monographs of J.W. Richards (1896), T.G. Pearson 1955), Kai Grjotheim (1956)and excepting the Russian literature, a few papers in The Journal of the Electrochemical and Faraday Societies. While it took a while, the consensus was that sharing non-competitive technical and discovering some of the trends in the strategies of competitors was that these aspects were all conducive to advancing the competitiveness of the whole aluminum industry, raising the awareness of the effectiveness of in-house R&D and enhancing the innovations and creativity of all technologists. Therefore, in 1971, again in New York, now the TMS Light Metal, began its series of annual meetings involving the international aluminum industry and academia. There participation increased to ninety-seven contributors with sixty four concerned with the reduction process. This presentation explores the first eight or nine TMS Light metals symposia with the objective of categorizing the topic fields, the extent to which they were "cutting edge", comments on how they influenced the electrolytic production of aluminum and inspired competitive research with improved funding, the attraction of future technologists into the industry and nurturing of "centers of excellence" for research in the Hall Héroult Process, novel processes and advanced materials.

10:55 AM Plenary
Russian Aluminium Industry: *Peter Polyakov*[1]; Victor Mann[2]; [1]Siberian Federal University; [2]Russian Aluminum Industry

1. Prehistory: Professor Fedotyeff's, Professor Pushin's investigations.2. Soviet Union Early stage: 1932 – Volkov Smelter (PB); 1933 – Zaporozhye Smelter (PB); 1939 – Ural Smelter (HSS). 3. Scientific research: Professor M. Rappoport – Sodium intercalation; Professor A. Belyaev – bath for Al-cells; Professor M. Vetyukov – carbon technology; Professor Korobov – heat transfer; Candidate Science Forsblom A. Tsyplakov – magnetic fields; Professor Kaluzhsky – from VSS to PB; Professor P. Polyakov – Al-cell as a living creature. 4. After WW II. Huge Smelters: 1964 – Krasnoyarsk (VSS); 1966 – Bratsk (VSS); 1985 – Sayanogorsk (PB); 2007 – Khakas (PB). 5. Current scientific investigations: Soderberg technology improvement; PB-cells development; Al laboratory studies.

11:35 AM Plenary
Brief Overview of Environmental Control within the Primary Aluminium Industry: *Erik Keul*[1]; [1]ALSTOM Environmental Norway

The Aluminium Industry has come a long way with respect to Environmental Control during the 125 years with the Hall-Heroult process. The initial smelters were small and any adverse effects were either of no concern or were not detected. As the smelters grew in size improved potroom ventilation was the major method for reducing exposure of the workers to fumes as well as for protection of equipment. The first noticeable damage to vegetation was detected in St. Jean de Maurienne in 1909. Environmental Control within the industry was basically a volountary effort and evolved due to concern about worker's health and obvious damage to the local environment, but has been helped along due to legislations. Various types of roof scrubbers were introduced in the 1930's and were applied by some smelters as the only treatment systems until the 1970's. None of these had reached the expected removal efficiencies due to inadequate sampling methods. Hooded cells were also introduced in the 1930's and in the following decades the concentrated fumes were either dispersed by tall stacks or cleaned by cyclones, electrostatic precipitators, wet scrubbers or a combination of these. Since the late 1960s dry scrubbers operating with alumina for removal of gaseous fluorides and particulates have been the state of the art emission control technology. Mega-smelters may introduce local environmental impact from SO2 which would need to be treated in additional downstream scrubbers such as seawater scrubbers for coastal smelters. As per IAI the reduction in fluoride emission can be divided into three generations: 1st generation plants 1940 – 1955 12 – 15 kg per tonne; 2nd generation plants 1955 – 1975 2 – 6 kg per tonne; 3rd generation plants 1975 – today 0.3 – 1 kg per tonne. Today fluoride emission is not a major concern and due to legislation as well as to potential loss of costly raw materials all new smelters employ efficient pot hooding combined with dry scrubbers at +99% collection efficiencies. The focus today is on SO2 removal, on greenhouse gases like CF4/C2F6 and on PAHs from the green-mills and baking kilns. Future legislation (in EU) will likely enforce reductions in particulate emissions in addition to CO2 limitations.

12:00 PM Plenary
The Future for Aluminium Smelting: *Mark Taylor*[1]; J.J.J. Chen[1]; [1]University of Auckland

The future of aluminium smelting is not assured. A primary driver of this situation is that the reasons for continuously reducing real margin for primary aluminium are not openly acknowledged, or even agreed. Is it the total cash cost of production ? Or is it the community perception first, which causes a loss of public support for investment in infrastructure and resources to support heavy industries like aluminium smelting? Of course this is not a new question and many countries including USA and Europe have been confronted with the competing requirements of energy or resource intensive industries. In this competition, the wider community must prevail eventually. And as these competitive situations play out in the customary adversarial manner between business and Government, the new question is becoming: Is there another way for the business community to think about the place of energy intensive industries in our interconnected, interdependent societies where energy and other natural resources are apparently in short supply? In this paper we discuss specific energy consumption across the industry, the perceived barrier in reducing it, and enabling factors. Lastly we explore the possibility that aluminium smelters could become an integral part of the network for power supply in the community, and across communities and countries.

2011 Functional and Structural Nanomaterials: Fabrication, Properties, Applications and Implications: Nanomaterials: General

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee
Program Organizers: Jiyoung Kim, Univ of Texas; David Stollberg, Georgia Tech Research Institute; Seong Jin Koh, University of Texas at Arlington; Nitin Chopra, The University of Alabama; Suveen Mathaudhu, U.S. Army Research Office

Monday AM
February 28, 2011
Room: 8
Location: San Diego Conv. Ctr

Session Chairs: Jiyoung Kim, University of Texas at Dallas; Suveen Mathaudhu, US Army Research Lab

8:30 AM Introductory Comments

8:35 AM Keynote
Materials Research Support at the National Science Foundation: *Alan Ardell*[1]; [1]National Science Foundation

The NSF/DMR perspective on materials research and education will be presented. DMR invested ~$303M in FY10, supporting people, ideas, and instrumentation primarily through awards to the nation's colleges and universities. Apart from DMR, there is also substantial support for materials and materials-related research and education from other programs in NSF. Specific new opportunities – including the 4+ year-old program in biomaterials and the SOLAR and EFRI initiatives – will be discussed. There are also avenues for collaborative research, nationally and internationally, via different types of special funding programs such as the new CEMRI and MIRT, which have replaced the MRSEC program, and the established MWN program. Other The projects supported by the MMN program in FY10 and the budgetary outlook for FY11 will be described. General information on all the programs in DMR can be found on the DMR Web page: <http://www.nsf.gov/mps/divisions/dmr/>.

9:05 AM Keynote
Nanostructured Materials Development by the Center for Nanostructured Materials Technology: *Sang-Hee Suh*[1]; [1]KIST, Center for Nanostructured Materials Technology

Over the last 10 years Korea has put large effort on nanotechnology development. Since Korean government started Korea Nanotechnology Initiative, we have seen tremendous increase in nanotechnology related publications, patents filed, new venture companies, and commercial products. Sponsored by the Ministry of Education, Science and Technology of Korea, the Center for Nanostructured Materials Technology has been managing one of the biggest programs for developing nanotechnologies in Korea. This Program aims to develop various nanostructured materials with superior properties by creating new materials or by applying nanotechnology to the already existing materials. The R&D areas covered by the Program include nanostructured materials for structural applications, environment and energy applications, and information technology applications. By the program, more than 30 original nanotechnologies have been developed, including 11 platform technologies. 22 technologies have been transferred to industry with many of them commercialized. Commercialized products include cutting tools with nano-layered coating; carbon nanotube/metal nanocomposites; amorphous structured thermal spayed ceramic coating; nano-membrane with dual functionalities of catalytic reaction; and separation and nano-cathode materials for Li rechargeable batteries. The author will introduce some of the platform and commercialized technologies developed by the program.

9:35 AM Invited
Nanomaterials: Research, Development and Technology (R&D&T) Roadmaps - 2020: *Marcel Van De Voorde*[1]; [1]TU Delft

An overview will be given of nanomaterials Science and Technology in the world. Fundamental research will be the backbone for future industrial success including new theories, design of third generation nanoMaterials, macro, and mico-nano computer modelling. Potential nanoMaterials will be highlighted. Research-development-technology "nanoMaterials" roadmaps: 2010 - 2025 for: Breakthroughs in communication and information; Grand challenges in life science and medical applications; NanoMaterials with tailored functionalities for new generation energy sources; Role of nanoMaterials in transportation: aeronautics and car industry; Potentials for the chemical industry; NanoConsumer products. Roadmaps 2010 – 2020 for nanoscience environment and climate change and safety: Man-Made environment; Climate Change; Nanopotentials in Safety; (Toxicology) and Security. Guidelines for a prosperous modern university - industry and welfare for the society: Initiate "university – science/technology institutes -industry" partnerships; Create International NanoMaterials R&D&T Centres; Promote Nanoindustrialisation: Technology Parks, nanometrology and standardisation. Initiate International Infrastructures for nanoMaterials R&D&T; and Familiarise the Society with the new Culture of NanoMaterials Technology. Countries and Industries investing in NanoMaterials Science and Technology: Model for a global nanoMaterials landscape: NanoMaterials - science, technology and nanoindustrialisation are complex topics and cannot be subject anymore for one institute or industry, or nation. The success rate will depend on joining brilliancies. Models for research and industrial collaboration will be elaborated and mechanisms for execution proposed.

10:05 AM Break

10:20 AM Invited
Nanomaterials Control for Biotech Applications: *Sungho Jin*[1]; [1]UC San Diego

There has been a strong trend in recent years toward convergence of nanotechnology and biotechnology for advanced biomedical applications. For example, biomagnetics is potentially a huge field for science and applications of magnetism and magnetic materials. Magnetic nanoparticles (e.g., superparamagnetic Fe-oxide) are one of the most versatile nanotech/biotech materials actively investigated for targeted cancer treatment, cell sorting and manipulations, guided drug delivery, bio-imaging and medical diagnosis. Nanowires and nanopore arrays are also useful for a variety of biotech applications. These are all very rapidly advancing fields. For practical applications for biomedical devices, proper engineering of these nanomaterials is essential. In addition, some aspects of nanotoxicity need to be understood for safe bio applications. This talk describes materials science aspects of these nano-biomaterials, their control, and implications for safe, therapeutic applications.

10:50 AM Invited
Can Single-Atom Change Affect Electron Transport Properties of Molecular Nanostructures such as C60 Fullerene?: Xiaoliang Zhong[1]; Ravindra Pandey[1]; Alexandre Rocha[2]; *Shashi Karna*[3]; [1]Michigan Tech; [2]Universidade Federal do ABC; [3]Army Research Lab

At the nanoscale, even a single atom change in the structure can noticeably alter the properties, and therefore, the application space of materials. We examine this critical behavior of nanomaterials using fullerene as a model structure in (i) endohedral (B@C60 and N@C60), in which the doping atom is encapsulated inside the fullerene cage, and (ii) substitutional (BC59 and NC59), in which the doping atom replaces a C atom on the fullerene cage, configurations. The calculated results reveal that the conductivity for the doped fullerene is higher than that of the pristine fullerene. In the low-bias regime, the current-voltage characteristic of the endohedral as well as the substitutional configurations are very similar. However, as the external bias increases beyond 1.0 V, the substitutional BC59 fullerene exhibits a considerably higher magnitude of current than all other species considered, thus suggesting that it can be aneffective semiconductor in p-type devices.

11:20 AM Invited
Atomic Layer Deposition - A Modern Tool for Nanoscience: *Mato Knez*[1]; [1]Max-Planck-Institute MSP

Atomic layer deposition (ALD) is a thin film deposition technique which was developed in the 1970s to meet the needs for processing thin film electroluminescent displays (TFEL). Technically and chemically it is similar to chemical vapor deposition (CVD). However, in contrast to CVD, ALD incorporates as a specific feature the separation of the chemical reaction into two half-reactions. Being a non-line-of-sight deposition technique, ALD allows for good coating conformality even with 3D nanostructured substrates or structures with a high aspect ratio. Our mission is to make use of the precision of ALD for fabrication or functionalization of a variety of nanostructures, optical coatings, encapsulation, replication or even infiltration of soft materials with metals. This talk will step through a selection of strategies on how to modify existing nanostructures or even change some physical properties of the structures making use of ALD for coating and infiltration, respectively.

11:50 AM
Advancing The Science of Nanomanufacturing: *Khershed Cooper*[1]; [1]Naval Research Laboratory

Nanomanufacturing is the fabrication of materials, components and devices with nano-scale features and their integration into viable engineering systems. A key ingredient is the enhancement or creation of new properties and functions by nano-scale physical phenomena. Nanomaterial building blocks, such as, quantum dots, nanotubes, nanowires, nanopillars and nanoparticles can be manipulated by top-down (e.g., atomic layer deposition) and bottom-up (e.g., self-assembly) processes to build novel nanodevice structures and useful nanosystems. The challenges for nanomanufacturing are achieving functionality, quality, repeatability, scalability and affordability. To address some of these challenges, basic research in areas such as nanoDDM (direct digital manufacturing), massively parallel processing, high-throughput (e.g., roll-to-roll) processing and system-level integration is needed. These concepts will be described in this paper and illustrated with research examples.

12:20 PM Concluding Comments

2nd International Symposium on High-Temperature Metallurgical Processing: Energy Efficient New Metal Production Technology

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee, TMS: Energy Committee
Program Organizers: Jiann-Yang Hwang, Michigan Technological University; Jerome Downey, Montana Tech; Jaroslaw Drelich, Michigan Technological University; Tao Jiang, Central South University; Mark Cooksey, CSIRO

Monday AM
February 28, 2011
Room: 18
Location: San Diego Conv. Ctr

Session Chairs: Jiann-Yang Hwang, Michigan Technological University; Anton Vernigora, Baikal Mining Company LLC

8:30 AM
A Breakthrough Application of Electricity at High Temperature for Steel Production: Molten Oxide Electrolysis: *Antoine Allanore*[1]; Luis Ortiz[1]; Donald Sadoway[1]; [1]MIT

In the search for more energy efficient processes together with a mitigation of GHG emissions, the use of a high-temperature electrolytic process to produce iron from its molten oxide is of interest as an alternative to traditional iron making. The intensive use of electricity for a steelmaking process is not new, but its application in electrochemical reactors can lead to a drastic reduction of its emission of pollutants together with the production of a new product: pure liquid iron. A description of the thermodynamics of the iron

oxide conversion to metal will be presented. This approach shows that at high temperature, electricity is a suitable energy vector for both the heat and work required for the reaction. The key physico-chemical parameters that govern the corresponding energy balance of the electrolysis process will then be discussed, to depict the process design conditions for best energetic efficiency.

8:50 AM

Intrinsic Hydrogen Reduction Kinetics of Magnetite Concentrate Particles Relevant to a Novel Green Ironmaking Technology: Haitao Wang[1]; *Moo Eob Choi*[1]; Hong Yong Sohn[1]; [1]University of Utah

A novel ironmaking technology is under development at the University of Utah. This technology produces iron directly from iron ore concentrate by gas-solid suspension reduction. Hydrogen is the main reducing agent for high reactivity and for the elimination of carbon dioxide release in this ironmaking process. The direct use of concentrates allows bypassing the problematic pelletization/sintering and cokemaking steps in the steel industry. Intrinsic kinetics of the suspension reduction of magnetite concentrate particles by hydrogen have been measured. Experiments were carried out in the temperature range of 1423 - 1673 K, other experimental variable being hydrogen partial pressure, the amount of excess hydrogen and particle size. Under most experimental conditions, 95% reduction was attained within several seconds, which presents sufficiently rapid kinetics for a suspension reduction process. The reaction kinetics followed the nucleation-and-growth equation, and a rate equation that contains all the effects of the experimental parameters has been obtained.

9:10 AM

A Laboratory Investigation of the Reduction of the Iron Carbonate Bearing Ore to Iron Nugget by Means of the ITmk3 Technology: *Nikolay Panishev*[1]; Rafkat Tahautdinov[1]; Anton Posazhennikov[1]; Vasily Bastrygin[1]; [1]Magnitogorsk Iron & Steel Works

The Bakal (South Ural, Russia) deposit of iron ore bearing iron carbonate with the capacity of more than 1 billion tones belongs to the MMK. This ore cannot be fully processed via blast furnace technology because of high content of MgO. According to the investigations carried out in the USA and Japan in 1999-2004 the ITmk3 RHF technology is a breakthrough in ironmaking technology. Four iron ore types (hematite, magnetite, high and low Al2O3/SiO2) were tested. The main objective of the investigation is to establish optimum operation conditions for the production of iron nuggets from iron carbonate bearing ore via the ITmk3 by means of the lab scale testing. Green pellets were processed via a lab tube furnace to simulate RHF conditions. This preliminary test work provides valuable information which may be used for large-scale testing in a commercially sized RHF.

9:30 AM

Prospects of Making Directed Reduction Iron by Microwave Heating: *Linqing Dai*[1]; [1]Kunming University of Science and Technology

Based on selectively summarized the characteristics of microwave heating and the research advances on making directed reduction iron by microwave heating.It was pointed out that microwave heating on making directed reduction iron has a good future by analyzing the development direction of directed reduction iron technology

9:50 AM

Behavior of Coal-Based Direct Reduction Reaction of Iron Oxide Pellets by Microwave Heating: *Zhu-cheng Huang*[1]; Hua Wang[1]; Bing Hu[1]; Hu Peng[2]; Guang-bin Xia[2]; [1]Central South University; [2]Changsha SYNO-THERM Co., Ltd

The temperature rising characteristics and reaction of the reduction of iron oxide pellets with anthracite fines were studied using a MW-L0316V microwave oven and Leica-DM-RXP polarizing microscope. The results show that iron oxide pellets and anthracite fines have good microwave absorbing properties, and high temperature and reducing atmosphere in a relatively short time for reduction of iron oxide pellets can be achieved. The reduction reaction, which follows the unreacted core model, is rapid and the metallization increases from 13.41% to 56.57% as the reduction end point temperature increases from 850° to 950°. The iron is formed, fine grain crystals of iron are transferred and the contraction of metal phase occurs firstly at the surface of pellets, which hinders the reduction reaction at the center of pellets. The rate of reduction reaction increases slowly as the reduction end point temperature increases from 950° to 1050°.

10:10 AM

Sustainable Developments in High Temperature Mineral and Metals Extraction and Processing: *Florian Kongoli*[1]; Edward O'Brien[1]; S. Llubani[1]; Ian McBow[1]; [1]FLOGEN Technologies Inc

Sustainability has become a more frequent prerequisite for any project in any field of life. Any major development project has started to include sustainability as a criteria in evaluating its objectives and outcome. However for heavy multibillion dollar industries such as high temperature mineral processing and metals extraction, sustainability is a major criteria since the impact this industry has in the world and its sustainable future is much more pronounced than in many other fields. Environment, energy, residues, efficiency, etc are all important factors related to sustainability of high temperature mineral and metals extraction and processes. Improving or updating these characteristics make these industries more sustainable. However high temperature minerals and metal extraction and processing industries face the competition of low temperature minerals extraction and processing such as hydrometallurgical, electrochemical routes. Low temperature processes sometimes look more popular that high temperature ones. The real question is: Which one of these 2 different routes is more sustainable for the future? Are low temperature processes capable of replacing high temperature ones and if yes are they more sustainable? This paper will give an overview of aspects of sustainability of various high and low temperature mineral and metal processing routes in terms of environmental protection and residues, productivity, energy etc. and will offer a glimse of the future that waits us.

10:30 AM

Carbothermal Reduction of Titanium Concentrate at High Temperature: *Run Huang*[1]; Chenguang Bai[1]; Xuewei Lv[1]; Guibao Qiu[1]; Lei Lei[1]; [1]College of Materials Science and Engineering, Chongqing University

The TiO2-rich slag and pig iron can be produced from ilmenite concentrate by the electric furnace process. In this study, ilmenite concentrate was reduced in a vertical furnace equipped with a weighting data acquisition system. The effects of reducing agent (carbon) amount on the iron reduction were studied. The phase transformation, morphology and chemical compositions of reduced samples were investigated by X-ray diffractometry(XRD), scanning electron microscopy(SEM) and energy disperse spectroscopy(EDS), respectively. It was found that the reductive degree of ilmenite concentrate increased with the increase of reducing time. The phases of reduced samples were mostly iron, rutile, Ti3O5 and Fe2TiO5. The mass loss percentage increased with increasing the carbon amount from 8% to 12%. When the carbon amount exceeded 12%, the mass loss percentage decreased with the increasing of carbon amount.

10:50 AM

A Simulation Study on Flue Gas Circulating Sintering (FGCS) for Iron Ores: *Tao Jiang*[1]; Zhenyu Fan[1]; Yuanbo Zhang[1]; Bin Xu[1]; Guanghui Li[1]; Xiaohui Fan[1]; [1]Central South University

Iron ore sintering process is the main source of SO2 generated in the steel industry, of which the discharge amount of SO2 emission accounts for about 60% of the total. Aiming at the features of sintering flue gases and existing problems during the flue gas desulphurization, a technical route of flue gas circulation sintering (FGCS) for iron ores has been put forward. Under the simulated experimental conditions, effects of FGCS process on the main output and quality indexes and SO2 emission rule are researched using simulating flue gases. Compared the novel FGCS with conventional sintering process, SO2 in the final sintering flue gas is obviously enriched and the total exhaust gases can be reduced evidently. The decreasing of oxygen content in circulating gases has bad effect on the sintering indexes. Increasing the oxygen potential is beneficial to obtain high quality sinters. This investigation shows that the FGCS process is promising.

11:10 AM

Energy And Exergy Analysis of Different Technologies of Copper, Zinc and Lead Production – Entropy Generation and Thermoecological Cost: *Bozena Boryczko*[1]; Adam Holda[1]; Zygmunt Kolenda[1]; [1]AGH University of Science and Technology

Copper, zinc and lead can be produced with different technologies. Most popular copper technologies are shaft furnace and flash smelting. Zinc and lead are produced by shaft furnace or by hydrometallurgical method. Main problem of all metallurgical technologies is the consumption of exergy connected with inevitable depletion of nonrenewable natural resources. One of the most important (from thermodynamic point of view) measure of their depletion is Cumulative Exergy Consumptionas the basis of calculation of thermoecological cost (defined as the cumulative exergy consumption of non-renewable natural resources). Difference in exergy consumption is the result of different kinds of driving energy. In shaft furnace technologies, driving energy is the chemical exergy of coke and for flash smelting or hydrometallurgical processes, electric energy plays principal rule. Because of those different exergy sources, the CexC values can differ significantly. The concept of thermoecological cost allows to choice technology with the lowest value of CexC.

11:30 AM

Optimizations of Preparation for U3O8 by Calcination from Ammonium Durante Using Response Surface Methodology: *Bingguo Liu*[1]; Jinhui Peng[1]; Daifu Huang[1]; [1]Kunming University of Science and Technology

The conditions of technique to prepare U3O8 by calcinations from ammonium diuranate were optimized, and the response surface design was applied to analyze the influence on the total uranium and of calcination temperature, calcination time mass of sample. A quadratic equation model for the total uranium and of U3O8was built and effects of main factors and their corresponding relationships were obtained. The analysis of variance shows that calcination temperature and calcination time significantly affected the value of total uranium and of U3O8. The optimal calcination conditions were as follows: calcination temperature931.83 K, calcination time 24.32min and 43.89 g. Under these conditions the value of total uranium and of U3O8 was 84.78% and 28.02%, respectively. The validity of the model was confirmed experimentally and the results were satisfactory.

11:50 AM

Microwave Field Attenuation Length and Half-power Depth in Magnetic Materials: *Zhiwei Peng*[1]; Jiann-Yang Hwang[1]; Xiaodi Huang[1]; Matthew Andriese[1]; Wayne Bell[1]; [1]Michigan Technological University

The equations for determining microwave field attenuation length and half-power depth in magnetic materials were derived from Maxwell's equations. The microwave field attenuation length and the half-power depth for a magnetite concentrate were calculated and the temperature dependence of them was determined. It is demonstrated the microwave field attenuation length and the half-power depth highly depend on temperature and decrease with increasing temperature up to 900 °C. The variations of field attenuation length and the half-power depth with temperature indicate the microwave power attenuates much faster than the field strength in materials. The evaluations of field attenuation length and microwave half-power depth can be used to characterize the microwave dissipation behaviors in the sample and optimize the dimensions of the material in microwave heating.

12:10 PM

Vanukov Furnace Technology: Application Experience for Processing Different Types of Raw Materials and General Development Trends: Valentin Bystrov[1]; Valery Paretsky[2]; Anton Vernigora[1]; Rostislav Kamkin[1]; *Alexander Mamaev*[1]; Alexander Kuznetsov[1]; [1]National University of Science and Technology (MISIS); [2]State Research Center of Russian Federation "Gintsvetmet" Insitute

Vanukov Furnace Technology is an efficient, proven pyrometallurgical injection technology, extensively used in Russia and Kazakhstan for a number of different applications. Technology was most widely adopted for processing copper sulfide concentrates for matte in smelters of Norilsk, Revda (Russia) and Balkhash (Kazakhstan). Also the following applications were developed and tested in industrial scale: treatment of sulfide lead and lead-zinc concentrates, laterite nickel ore, production of cast iron, treatment of antimony gold-containing ores and municipal solid waste. In this paper, current experience of Vanukov Furnace application to these technologies is described with a number of general development trends and new ways of applications.

Advances in Science-Based Processing of Superalloys for Cost and Sustainment: Processing Advancements via Modeling and Simulation

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: High Temperature Alloys Committee, TMS: Advanced Characterization, Testing, and Simulation Committee

Program Organizers: Donna Ballard, US Air Force; David Furrer, Pratt & Whitney; Paul Jablonski, US Department of Energy; Christopher Woodward, Air Force Research Laboratory; Jeff Simmons, AFRL; Mark Blodgett, Wright-Patterson AFB

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Session Chairs: Paul Jablonski, US Department of Energy; Lee Semiatin, US Air Force Research Laboratory

8:30 AM Introductory Comments

8:35 AM Keynote

Nickel-Based Superalloys: Construction, Use and Validation of Numerical Models: *Roger Reed*[1]; [1]University of Birmingham

In recent years, the use of numerical modelling in the field of high temperature materials – particularly for the superalloys – has increased markedly. There are now very tangible advantages to be gained from modelling work, e.g. for (i) the invention of new grades of alloy, (ii) the analysis and optimisation of manufacturing processes (casting, welding, heat treatment etc) and (iii) the design of high-integrity components such as turbine blades and discs. In this presentation, examples of successful modelling work in each of these areas will be given. The advantages, limitations and future expectations are given. The overarching observation is that the field has grown from one of mere academic curiosity to one which is of great importance to the viability of businesses operating in the high temperature world.

9:15 AM Invited

Air Force Research Laboratory R&D in Science-Based Superalloy Processing: *Lee Semiatin*[1]; Donna Ballard[1]; David Mahaffey[1]; Jonathan Miller[1]; Jaimie Tiley[1]; Todd Turner[1]; Donald Weaver[1]; Adam Pilchak[2]; JP Thomas[3]; Kyle McClary[4]; Peter Lee[5]; [1]US Air Force Research Laboratory; [2]UTC, Inc; [3]ATI Allvac; [4]Wright State University; [5]Imperial College London

Basic research at AFRL to develop science-based understanding and predictive models for microstructure evolution during the thermomechanical and solidification processing of superalloys will be summarized. For cast-and-wrought superalloys, the physics of homogenization heat treatment and recrystallization during hot working have been established. These observations have been interpreted using 2D diffusion analysis, crystal-plasticity FEM to quantify deformation nonuniformity, and a mesoscale modeling technique that treats the effects of dislocation generation/annihilation and boundary migration on the kinetics of recrystallization. With respect to powder-metallurgy superalloys, the evolution of grain structure during TMP will be described. Specifically, the effect of hot working/heat treatment conditions and second phases on the final grain size and the status of associated models will be presented. Last, the effect of solidification conditions on the propensity for lateral dendrite growth during directional solidification will be described. Phenomenological (processing-map) and

mesoscale (cellular-automaton) models to describe such phenomena will be briefly reviewed.

9:45 AM
Recrystallization and Grain Growth of a Hot Pack Rolled Nickel-Base Superalloy: *Adam Pilchak*[1]; S. Semiatin[1]; Donna Ballard[1]; Donald Weaver[1]; [1]Air Force Research Laboratory

Microstructure and texture evolution of a hot pack rolled and annealed Ni-Al-Cr-base superalloy were investigated with electron backscatter diffraction. In the as-rolled condition, the microstructure consisted of elongated, unrecrystallized grains that were in orientations consistent with typical rolling deformation texture components. Subsequent heat treatment recrystallized the microstructure which resulted in an overall decrease in the intensity of the various deformation texture components, but not the development of new orientations. Grain growth kinetics during subsolvus heat treatment were quantified and analyzed on the basis of existing models for grain growth in the presence of second phase pinning particles and texture controlled grain growth. Grain growth behavior during supersolvus heat treatment was also investigated. The results were compared to simulations performed using 3D Monte-Carlo methods incorporating misorientation dependent grain boundary energy and mobility.

10:05 AM Break

10:20 AM
HIP of Ni-Base PM Superalloys- Back to the Future: *Charles Barre*[1]; Viktor Samarov[1]; Dmitry Seliverstov[1]; [1]Synertech PM Inc.

HIP of complex shape parts from PM superalloys is coming back for critical applications This comeback is based on a suit of the advanced technologies related to this consolidation process. These technologies include: - Advanced computer process modeling that enables to provide very complex shapes and a significant reduction of development and manufacturing cost. - Enhancement of mechanical properties due to the control of the powder size and inclusions during manufacturing of powder and filling of the HIP capsules - Shape control during HIP due to the adequate design of the HIP tooling and filling procedures - Control of the quality for the net shape surfaces and their enhancement for the environmental compatibility - New technologies of non-destructive testing for the complex shape components The paper analyses the developed technological tools and numerous applications.

10:40 AM Invited
Microstructural Level Modelling of Freckle Initiation during Directional Solidification: *Peter D. Lee*[1]; Lang Yuan[1]; [1]Imperial College London

Freckles are severe channel-like segregates commonly observed in directionally solidified and single-crystal Ni-base superalloy turbine blade castings, causing part rejection. Freckles arise from the segregation of solute from the solidifying dendrites, altering the density of the interdendritic liquid, causing a Rayleigh instability initiated flow. To predict the on-set of flow the interaction of thermal gradients, microstructure, and permeability all need to be modelled. However, prior work has focused on macrosegregation, with the onset of flow initiated due to numeric noise. In this study, a microstructural model is coupled with a flow solver to provide direct simulation of freckle simulation. The predictions are compared to the extensive prior experimental results in the model binary alloy system, Pb-Sn. Solute channel formation was predicted under different solidification conditions (based on temperature gradient and cooling rate). Using the Rayleigh number, a good correlation between the numerical simulations and experimental was found.

11:10 AM Invited
Heat Extraction and Structure Evolution in LMC Single-Crystal Growth: *Jonathan Miller*[1]; Lang Yuan[2]; Michael Eisman[3]; Peter Lee[2]; Tresa Pollock[4]; [1]AFRL/RXLM; [2]Imperial College; [3]Wright State University; [4]University of California, Santa Barbara

The liquid-metal-cooling (LMC) process for growth of single-crystal superalloys provides enhanced heat extraction, higher cooling rates and refinement of dendritic structure. However, some processing conditions cause substantial lateral heat extraction that promotes lateral growth of dendrites – the formation of long secondary dendrite arms overgrowing favorably aligned primary dendrites. The conditions under which lateral growth occurs during solidification of alloys CMSX-486 and René N4 have been studied experimentally and with solidification modeling at the macro- and meso-scales. Solidification experiments have been conducted in a LMC furnace that utilizes liquid tin as the cooling medium and a floating ceramic baffle. A mold geometry was designed to evaluate a range of thermal conditions and assess the tendency for lateral growth. Correlations between dendritic structure, solidification-front curvature, solidification rate and thermal gradient will be presented. The presence of lateral growth is strongly dependent on the solidification-front curvature at the casting surface.

11:40 AM
Residual Strain Measurements in a Single-Crystal Nickel-Based Superalloys Turbine Blade using Neutron Diffraction: *Stephane Pierret*[1]; Alexander Evans[1]; Ania Paradowska[2]; Thomas Etter[3]; Helena Van Swygenhoven[1]; [1]Paul Scherrer Institute; [2]ISIS; [3]Alstom

Rafting in nickel-based superalloys describes the directional coarsening of the gamma prime precipitates when subjecting the alloy to high temperature creep deformation. But rafting has also been observed at very localised areas in a non-crept fully heat-treated single-crystal (SX) Ni-based superalloy turbine blade. The influence of macro residual stresses and the associated inhomogeneous plastic strain fields on the rafting of the precipitates has been investigated using neutron diffraction in a 2nd generation SX Ni-based superalloy turbine blade following cooling from the solution heat treatment. A novel approach was used to localise the measurements in the partially hollow blade, by combining a 3D digitisation with a 3D reconstructed image obtained by neutron tomography. Complementry hard energy synchrotron diffraction followed on a cross-section of the blade at the position where the maximum residual strains were determined with neutrons. This revealled the variation of the diffraction peak width and the lattice parameter misfit.

12:00 PM
Numerical Simulation of Directional Solidification of Turbine Blade by LMC Process: Ning Tang[1]; *Qingyan Xu*[1]; Baicheng Liu[1]; [1]Tsinghua University

Directionally solidified blade castings are widely used in turbines, because the directional solidified microstructure leads to outstanding high temperature properties. A very strict process control is needed to avoid stray grains. In recent years, liquid metal cooling (LMC) is used as a new process in manufacturing of the blade casting, and it still needs to be improved. Numerical simulation of the LMC process is helpful to optimize the technical parameters and reduce the development cycle and cost. In this paper, a model of a LMC process considering the convection between the shell and the cooling metal was established, which was validated by the experimental results. Different processes of LMC solidification were simulated. The results indicated that higher withdrawal rate or lower cooling liquid metal temperature can obtain higher temperature gradient and lead to thinner mushy zone. However, if the withdrawal rate is too high, the mushy zone becomes more concave.

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: Subodh Das, Phinix LLC; Zhengdong Long, Kaiser Aluminum; Tongguang Zhai, University of Kentucky

Monday AM
February 28, 2011

Room: 14A
Location: San Diego Conv. Ctr

Session Chair: Zhengdong Long, Kaiser Aluminum

8:30 AM
Scandium/Zirconium Modified Aluminum Alloys for Improved Mechanical Properties: *Jennifer Gaies*[1]; [1]NSWC Carderock Division

Due to the need for high speed lightweight ships there is an increased use of aluminum alloys in ship construction. These applications require materials with the following combination of properties: high welded strength, ductility and elongation, corrosion resistance, and fatigue and corrosion fatigue resistance. The most commonly used aluminum alloy families in marine applications are the Al-Mg (5XXX series) alloys which derive their strength from work hardening and are not heat-treatable. Reduced as-welded strengths limit their practical structural applications. The high temperatures introduced during the welding process reverse some of the effects of the strengthening mechanisms so aluminum alloys which can retain the greatest amount of post-welded strength are, therefore, greatly desired. Small additions of Sc and Zr have been shown to improve mechanical and corrosion properties of aluminum alloys and their effect as additions to commercial 5XXX alloys are investigated.

8:50 AM
Continuous Cast Superplastic Grade Aluminum Sheet: *Ravi Verma*[1]; [1]General Motors

Thermo-mechanical processing of a twin-belt continuous cast (CC) Al 5083 slab is investigated with the objective of developing superplastic (SP) grade aluminum sheet. The effect of small variations in the Mn content of the alloy on its SP formability is also investigated. The optimized process is capable of breaking down the as-cast structure of the CC slab to a fine equiaxed microstructure (grain size 7 μm) suitable for superplastic forming. Compared to the conventional Direct-Chill (DC) process, which uses massive rolling to achieve the necessary grain refinement, the developed process achieves similar level of grain refinement in much less rolling reduction. The SP grade sheet produced with the developed process exhibit tensile ductility of 302% at 500ºC and 10^{-3} s^{-1} strain rate, which is impressive for this initial material. This work demonstrates the technical feasibility of producing high quality SP grade aluminum sheet based on low-cost, twin-belt continuous cast technology.

9:10 AM
Study of Aluminum Sensitization at Moderately Elevated Temperatures: *William Golumbfskie*[1]; Catherine Wong[2]; [1]Naval Surface Warfare Center, Carderock Division; [2]NAVSEA

Structural aluminum alloys are seeing increased use in marine applications, with the shipbuilding industry looking to reduce weight. The 5xxx series is an ideal candidate, combining high specific strength, corrosion resistance, and weldability. A particular concern is sensitization. Aluminum becomes sensitized when beta phase (Al3Mg2) is precipitated at grain boundaries, which may lead to stress corrosion cracking. Sensitization is believed to occur when aluminum is exposed to temperatures greater than 50°C. This study will investigate the effects of exposure at lower temperatures (>30°C) on sensitization. Such "moderately" elevated temperatures are of great importance as ship decking and superstructures experience such temperatures while in service. The ASTM G67 mass loss test is being used to quantify the extent of sensitization. The new data will aid in determining the degree of sensitization for in-service 5xxx alloys and estimate the lifetime until the material has fully sensitized.

9:30 AM
Forming Limit Diagrams for AA5182 after Preform Annealing: Jingjing Li[1]; *John Carsley*[2]; Theresa Lee[2]; S. Jack Hu[1]; [1]University of Michigan; [2]General Motors

Preform Annealing is a process of forming aluminum sheet panels in which a blank is partially stamped, heat treated and then stamped to the final desired shape. Because strain from the first deformation event is recovered by annealing, the process significantly improves product formability. The effects of prestraining along different strain paths and subsequent annealing on the ultimate forming limits of aluminum alloy 5182-O were investigated by determining forming limit diagrams (FLDs) using digital image correlation (DIC) and circle grid analysis (CGA). Aluminum panels were prestrained in uniaxial, equibiaxial and plane strain tension at 15 and 20% equivalent strains, perpendicular to the rolling direction; and annealed at 350°C for short (10 seconds) and long (20 minutes) durations. Calculations of total effective forming limit curves (FLC) were compared to experimental CGA and DIC results. The improvement in FLCs shows the advantage of annealing that can extend the formability of strained materials.

9:50 AM
A Positron Annihilation Study of Hot Band of a Continuous Cast AA 2037 Al Alloy after Annealing: Yichu Wu[1]; *Tongguang Zhai*[2]; [1]University of Wuhan; [2]University of Kentucky

Continuous cast AA 2037 Al alloy hotband was annealed at temperatures ranging from 25 to 500ºC for 3h. Vacancy-solute interactions and precipitates in the samples during annealing were investigated using positron annihilation techniques (coincidence Doppler broadening (CDB) and lifetime, respectively) and TEM. It was found that there were vacancy-Cu and vacancy-Cu-Mg complexes at room temperature. Positron mean lifetime peaked at 200ºC because of the formation of semicoherent precipitates. The measured CDB ratio curve, characteristic to Mn in shape, indicated the formation of vacancy-Mg-Mn complexes or Mn clusters in these precipitates. Above 250ºC, coarsening and dissolution of semicoherent precipitates resulted in decrease in mean lifetime and disappearance of Mn signal, but Cu signal increased clearly with annealing temperature. Over 350ºC, Cu signal started to saturate, indicating that Cu dissolved might agglomerate and cluster with vacancies, while new increase in mean lifetime might be attributed to the formation of incoherent phase.

10:10 AM Break

10:25 AM
Aluminium-Titanium Alloys for Thermal Spraying: *Christopher Wheatley*[1]; [1]CJ Wiretech Limited

Arc-spraying using pure aluminium wire (TSA) has been accepted for many years in the oil industry for barrier protection of steel structures in aggressive marine environments. Aluminium-titanium alloys provide an even better physical barrier which exhibits extremely good resistance to erosion and this is achieved with no detrimental affect on corrosion properties. The combination of wear resistance and the macroscopic surface roughness from thermal spraying provides a surface of extremely low slip potential which can remain 'non-slip' for over 20 years at 1000 footfalls per day. Data of slip testing for pedestrians and vehicles is presented and this is supported by case studies in real life situations. Data from corrosion testing in laboratory conditions demonstrates that in some cases the aluminium-titanium alloys sprayed onto steel structures can give improved protection against corrosion in very severe conditions.

10:45 AM
Hot Tensile Behaviour and Constitutive Analysis of Al-5,5Zn-1,2Mg/Zr Alloys: *Paola Leo*[1]; Emanuela Cerri[1]; Hugh McQueen[2]; [1]Università del Salento; [2]Concordia University

In this study hot tensile behaviour of Al-5,5Zn-1,2Mg and Al-5,5Zn-1,2Mg-0,16Zr alloys deformed in the as-cast state has been analyzed. Tensile tests have been performed in the range 250°C-350°C and 10-5s-1 to 10-3s-

1. At the lowest test temperature the stress-strain curves shows a gradual softening after a stress maximum or peak while at the other temperatures a plateau is attained which is lower as temperature T rises and strain rates decrease. For each fixed temperature T, the ductility is almost independent of strain rate for Al-5,5Zn-1,2Mg whereas, for the Zr modified alloy, it decrease slightly with ε. The constitutive analysis by the sinh equation give QHW=218kJ/mol and QHW=271 kJ/mol for unmodified and Zr modified alloys respectively. These values of activation energy are quite high and similar to that of other 7000 alloy containing Cu. The microstructure of all tensioned samples is characterized by cavitation phenomena.

11:05 AM
Production of Continuous Cast 3105 Coil-Stock for Thin Gauge Roller Shutters: *Dionisios Spathis*[1]; John Tsiros[1]; [1]Hellenic Aluminium industry (ELVAL SA)

An optimum industrial process was established through trials of continuous cast 3105 alloy for roller shutters. Several thermomechanical processes were investigated regarding final gauge mechanical properties, bendability and surface quality. Softening curves were determined through laboratory annealing trials at intermediate and final gauge in order to choose the proper coil anneal temperature regime.

11:25 AM
Microstructure Evolution of the Modified AA 5083 Alloy: From the As-Cast State to the Final Product: *Endre Romhanji*[1]; Miljana Popovic[1]; Tamara Radetic[1]; [1]University of Belgrade

In this work, we characterized the microstructure evolution of industrially processed, commercial AA 5083 with a small Zr addition throughout complete processing route: from the as-cast state to the final hot rolled product. We investigated the effect of multiple stage thermal soaking of the cast alloy and homogenization of the industrially hot-rolled alloy on recrystallization and recovery during the final hot rolling. Microstructural changes during the thermo-mechanical treatments were followed by electrical resistivity measurements, optical and TEM. Two populations of the Mn-based secondary phases were identified in as-cast state. Coarse, eutectic particles containing Fe precipitated at grain boundaries, while finer Mn particles alloyed with Cr formed within grains. Although further processing affected the particle morphology and distribution, both types of precipitates remained distinct and had significant, but different effect on recrystallization process. This work is supported by Ministry of Science and Technological Development, Republic of Serbia under contract number E!4569.

Aluminum Rolling: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee
Program Organizer: Kai Karhausen, Hydro Aluminium Rolled Products GmbH

Monday AM — Room: 16A
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chair: Kai Karhausen, Hydro Aluminium

8:30 AM Introductory Comments

8:35 AM Keynote
Simulation of Particle Effects on Recrystallization in Commercial Al Alloys: *Günter Gottstein*[1]; Volker Mohles[1]; Carmen Schaefer[1]; [1]RWTH Aachen University

Depending on particle size and precipitation kinetics, second phase particles can affect recrystallization in various ways. Large particles which exist prior to recrystallization can cause particle stimulated nucleation which makes itself felt by an acceleration of recrystallization and a change of the recrystallization texture. A complex case arises when recrystallization and precipitation occur concurrently. This will have a significant influence on the recrystallization kinetics as well as grain size and texture. By means of finite element modeling and recrystallization simulation with the adaptive cellular automata code CORe the various cases were studied. The results will be discussed with respect the predictive power of recrystallization modeling in such cases.

9:05 AM
Texture Evolution during Symmetric and Asymmetric Rolling of Al-Si-Mg Alloys Fabricated by Twin Roll Casting: *Jaehyung Cho*[1]; Hyung Wook Kim[1]; [1]Korea Institute of Materials Science

6K21 aluminum alloys (1%Si-0.6Mg) fabricated by twin roll casting were cold-rolled using differential speed rolls. Effect of speed ratio and reduction in area on texture evolution was examined using XRD. Overall reduction in area was 75%. Loads measured in the rolls were decreased with speed ratio. As-casted alloys had texture gradient from the surface to the center. {111}// ND textures were found in the surface and weak rolling textures were located in the center. The initial textures changed into rotated cube, {100}<011> and rolling textures, a (Goss-Brass) and b (Brass-S-Copper). Shear texture, rotated cube, was found in both symmetric and asymmetric rolling processes. Various rolling parameters were investigated to generate shear texture. Roll speed ratios greater than 1.4 produced shear texture throughout the whole thickness direction.

9:30 AM
An Investigation of Deformation Behavior of Bimetal Clad Sheets by Asymmetrical Rolling at Room Temperature: *Li Xiaobing*[1]; Zu Guoyin[1]; Deng Qiang[2]; [1]School of Materials and Metallurgy, Northeastern University; [2]Cold rolling company, Pangang group company LTD.

The different thickness metals mild steel, aluminum and copper were bonded with each other by means of asymmetrical rolling at room temperature. The deformation behaviors, bonding conditions and interfacial layer thickness of the clad sheets were discussed. According to the slab stress in the plastic deformation region at the roll gap, the relations of bonding condition and metal flow were analyzed. The influence of cross shear on the bonding due to the roll speed mismatch is obvious. The large speed mismatch makes a good bonding and drops the critical reduction. The improvement of bonding is achieved with the increase of the total rolling reduction. The reduction of both layers increases in direct proportion with the total reduction, and the difference between hard and soft metals gradually diminishes. The large initial thickness ratio of hard and soft metal is unhelpful for the bonding due to the inconsistent deformation of bimetal.

9:55 AM Break

10:15 AM
Parameter Study within the Through-Process-Modeling Chain of AA8xxx-Alloys and Its Validation: *Thiemo Brüggemann*[1]; Volker Mohles[1]; Carmen Schäfer[1]; Günter Gottstein[1]; Kai Karhausen[2]; [1]Institute; [2]Hydro Aluminium Deutschland GmbH

Optimization of alloys and whole process chains for aluminum sheets and foils is a major goal of the aluminum industry. Since experimental methods are cost- and time-intensive, simulation tools are to be used to predict effects of material and process parameter changes on the product. Within this work the behavior of a through-process-modeling-chain is tested for its reaction on various parameter changes within the group of 8xxx-Aluminum alloys. The modeling chain consists of a thermally coupled roll gap model with subsequent simulation of rolling- and recrystallization-texture by grain-interaction and cellular automata models, respectively. Parameter studies will include microchemistry changes such as solute contents and particle sizes and radii. Data from industrial trials and experiments will be used for validation.

10:40 AM
Coil Build up Compensation during Cold Rolling to Improve Off-Line Flatness: *Lourival Almeida Neto*[1]; Tuggan Ayhan[2]; [1]Achenbach Buschhütten GmbH; [2]Assan Alüminyum

During rolling, overlaps of material with a convex profile at recoiler cause greater tension in strip center fibers than in fibers of the edges, which is

known as coil build up. The high tension on central area is understood by the automation system as zones tighter than reference commanding its actuators to put back such zones on target, loosing the correspondent strip fibers. The difficulty is that the buckles created by the flatness controller are not seen at operator's screen. A coil build up compensation based on material profile was developed in order to have the cause of center buckles after unwinding reduced to the coiling process. From 24 coils with thickness ranging between 0.7 and 2.0mm, 100% presented better offline flatness. Flatness carpets related to profile suggested a paradigm breaking: perhaps we have to accept rolling with not so good flatness in order to have the desired offline flatness.

11:05 AM
Through Process Effects on Final Al-Sheet Flatness: *Stefan Neumann*[1]; Kai Karhausen[1]; [1]Hydro Aluminium Dts. GmbH

In the process of rolled products to achieve a defined flatness of the strip in ever decreasing tolerances becomes more and more a key challenge. The typical process chain of strip material consists of hot rolling, cold rolling, annealing as well as coil winding and unwinding at various stages. Some Aluminium alloys exhibit the strong tendency to reduce internal stresses by creep and relaxation. This transformation of elastic into plastic strains frequently leads to considerable changes of shape and flatness of the product over the different steps of the process chain. This contribution introduces a model based through process analysis with emphasis to the evolution of strip flatness via the most decisive production stages. Case studies from aluminium strip production will be used to illustrate the impact of different process parameters.

11:30 AM Concluding Comments

Approaches for Investigating Phase Transformations at the Atomic Scale: Transformation Kinetics and Mechanisms

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
Program Organizers: Neal Evans, Oak Ridge National Laboratory; Francisca Caballero, Spanish National Research Center for Metallurgy (CENIM-CSIC); Chris Wolverton, Northwestern University; David Seidman, Northwestern University; Rajarshi Banerjee, University of North Texas

Monday AM
February 28, 2011
Room: 32B
Location: San Diego Conv. Ctr

Session Chairs: S. Babu, Ohio State University; Neal Evans, ORNL & Univ. Tennessee, Knoxville

8:30 AM Keynote
Solid-State Diffusion and Transformation Kinetics: *Georges Martin*[1]; [1]Retired

The recent dramatic increase in efficiency of atomic scale observation and modeling techniques opens new routes to the understanding of diffusion controlled phase transformations. The kinetic pathway for phase separation in ternary Ni(Cr, Al) supersaturated solid solutions is discussed*. First principle calculations and empirical fits to simple experiments give the parameters for kinetic Monte Carlo simulations of long sequences of vacancy jumps in the solution. The latter result in the precipitation of the □' Ni3(Al1-x, Crx) phase (L12), and perfectly reproduce quantitative and qualitative features revealed in real alloys by 3DAP. The same parameters are also used to measure the Onsager and diffusion matrices in the model solid solutions; several intriguing features of the observed pathways are shown to result from kinetic couplings between the diffusion fluxes of the alloy components, a coupling ignored by existing models. *Collaboration with David Seidman et al. NWU.

9:10 AM Invited
Atomistic Modeling of Diffusive Phase Transformations Kinetics by Monte Carlo Simulations: *Frederic Soisson*[1]; [1]CEA Saclay

The kinetics of diffusive phase transformations are essentially controlled by the concentrations, jump mechanisms and jump frequencies of point defects, and by the way they depend on the local atomic configurations. Atomistic Kinetic Monte Carlo (AKMC) simulations are nowadays one of the most efficient tool for taking into account such details and therefore for predicting the kinetic pathways, especially when associated with ab initio calculations, which provide a reliable way to compute the key physical properties and the corresponding Monte Carlo parameters. We present a few recent AKMC simulations of phase transformations in model systems of industrial interest, especially in iron-based alloys. Comparison with experimental kinetics using 3D atom probe and neutron scattering techniques is emphasized.

9:35 AM Invited
Chemical Mixing at Hetero-Interfaces Forced by Severe Plastic Deformation: *Pascal Bellon*[1]; Robert Averback[1]; Nhon Vo[1]; Xuan Zhang[1]; Elvan Ekiz[1]; Yinon Ashkenazy[1]; Daniel Schwen[1]; Mohsen Pouryazdan[2]; Horst Hahn[2]; [1]University of Illinois; [2]Karlsruhe Institute of Technology

Severe plastic deformation (SPD) can lead to the stabilization of supersaturated solid solutions in the case of moderately immiscible elements such as Cu and Ag, and to self-organization into lamellar structures in the case of highly immiscible elements, such as Cu and Nb. In this work, we contrast the different mechanisms operating in Cu-Ag and Cu-Nb alloys subjected to SPD, including high pressure torsion testing and ball milling. Combining experiments and molecular dynamics simulations, we show that the atomic mixing in Cu-Ag, surprisingly, increases linearly with strain as the elements are brought into solution. In contrast, Cu-Nb alloys can either remain phase separated or they can mix, depending on the initial crystallographic orientation of the Cu-Nb interfaces. Special orientations such as Kurdjumov-Sachs, for example, are remarkable stability against mixing, whereas others are not. In these latter cases, local amorphization is often observed at the interfaces.

10:00 AM Break

10:15 AM Keynote
THE INSTITUTE OF METALS LECTURE AND ROBERT FRANKLIN MEHL AWARD WINNER: The Ubiquitous Interfacial Free Energy in Phase Transformations: *David Seidman*[1]; [1]Northwestern University

The interfacial free-energy of a grain boundary or a heterophase interface is a ubiquitous and important quantity in all microstructures, and its short and long-term stability or lack of stability is intimately related to this fundamental quantity. Interfacial free energy affects important physical phenomena such as nucleation, growth and coarsening of different phases in multicomponent alloys, which are integral to the decomposition of a single-phase solid-solution. In this talk I focus on the diverse roles played by interfacial free energy in phase transformations in multicomponent metallic alloys, which are studied experimentally by atom-probe tomography and transmission electron microscopy. The results of simulations employing lattice kinetic Monte Carlo, where the phase transformation is mediated by a vacancy mechanism, will be presented and compared in detail with experimental observations. First-principles calculations of interfacial energies in the same metallic alloys will be discussed in terms of their relevance to all of the above.

10:55 AM Invited
Atomic Scale Investigation of Alpha Nucleation in the Beta Matrix of Titanium Alloys: Soumya Nag[1]; Robert Williams[2]; Arun Devaraj[1]; Peter Collins[1]; Gopal Viswanathan[3]; Rajarshi Banerjee[1]; *Hamish Fraser*[2]; [1]University of North Texas; [2]The Ohio State University; [3]Air Force Research Laboratory

The solid-state decomposition of the beta phase of titanium alloys, leading to the precipitation of the equilibrium alpha phase, is a rather complex phenomenon involving multiple competing compositional and structural instabilities. The generally accepted mechanism of alpha precipitation

in these alloys is via classical nucleation and growth. Coupling advanced characterization techniques such as synchrotron-based in situ x-ray diffraction, aberration-corrected high-resolution (S)TEM, and atom probe tomography, the early stages of alpha precipitation at the atomic to near-atomic scale have been investigated in two titanium alloys, a commercial beta alloy, Ti-5Al-5Mo-5V-3Cr-0.5Fe (TIMETAL-5553 or Ti-5553), and a model ternary Ti-Mo-Al system. These experimental observations indicate that depending on temperature the decomposition can take place via a novel pseudospinodal mechanism that involves homogeneous alpha precipitation at a composition far-from equilibrium, followed by a continuous change in the compositions of both the parent beta and the product alpha phases towards equilibrium.

11:20 AM

In Situ Studies and Simulations of Rapid, Self-Propagating Phase Transformations in Nanolayer Foils: *Timothy Weihs*[1]; Sara Barron[1]; Todd Hufnagel[1]; Steve Kelly[1]; Michael Falk[1]; Rong Xu[1]; Omar Knio[1]; Francesco Rizzi[1]; Geoffrey Campbell[2]; Judy Kim[3]; [1]Johns Hopkins University; [2]Lawrence Livermore National Lab; [3]Oxford University

Exothermic formation reactions are known to self-propagate in multilayer foils with nanoscale layers and large, negative heats of mixing, such as Ni/Al, Nb/Si and Ti/B multilayers. The exothermic reactions can travel at velocities greater than 10 m/s and they reach temperatures as high as 3000 K. The combination of rapid propagation and high temperatures produces extremely fast heating rates (~10^7 K/sec) within the reaction front, while the nanoscale layers provide steep chemical gradients. Here we investigate the impact of both extremes (heating rate and chemical gradient) on reaction paths and phase morphologies using in situ X-ray diffraction, Dynamic TEM, Molecular Dynamic Simulations, and numerical modeling. The sequence of phase transformations and the phase morphologies that appear under fast and slow (~1 K/sec) heat rates are shown to be different and the impact of steep chemical gradients is assessed. Work performed under auspices of USDOE OBES by JHU under contract DE-SC0002509 and by LLNL under contract DE-AC52-07NA27344.

11:35 AM

Molecular Dynamics Studies of Phase Transformations during Rapid Heating of Nanolayer Foils: Rongguang Xu[1]; *Michael Falk*[1]; [1]Johns Hopkins University

A series of molecular dynamics studies of multilayer metallic floils undergoing rapid heating due to exothermic reaction reveal a number of salient details regarding the sequence of reaction. In simulations of Ni-Al foils melting is followed by the sequential precipitation of a number of intermetallic phases. The sequence of phase formation is observed to be sensitive to the interlayer mixing prior to the onset of the reaction. In simulations of Ni-Zr foils spontaneous amorphization precedes the formation of an ordered intermetallic. Atomic scale diagnostics are used to quantify the fraction of the material that has undergone phase transformation, identifying both atoms in ordered alloy and solid solution phases. This work was performed under DOE contract DE-FG02-09ER46648.

11:50 AM

Interplay between Interfacial Segregation and Diffusion Shapes the Growth of Gold-Catalyzed Silicon Nanowires: *Moneesh Upmanyu*[1]; Hailong Wang[1]; Luis Zepeda-Ruiz[1]; [1]Northeastern University

The vapor-liquid-solid for nanowire synthesis route has gained relevance of late as it in principle allows direct control over nanowire morphology and composition during synthesis. Here, we present classical computations on a small diameter, gold-catalyzed <111> silicon nanowire that for the first time allow access to the atomistics underlying the growth process. We find a direct effect of the interfacial segregation, at the catalytic particle surface and at the faceted particle-nanowire interphase, on the diffusive kinetics that builds up the supersaturation necessary for interphase nucleation. The silicon-rich surface layer suppresses diffusion to an extent that the supersaturation is carried by the bulk of the particle. They key structural aspect is the Au segregation at the interphase. The enhanced undercooling is kinetically offset by Au metastability around nucleated steps that arrests lateral flow such that the growth is no longer layer-by-layer. We discuss the implications controlled growth of nanowire arrays.

12:05 PM

Ta Clustering and Microstructural Evolution in the A1 to L10 Fe52PtX(Ta1-X) Phase Transformation: Diondra Means[1]; Billie Wang[1]; *Gregory Thompson*[1]; [1]University of Alabama

FePt magnetic films have application in ultrahigh magnetic storage media. Their chemical ordering phase transformation and microstructure can be controlled by the addition of ternary elements. Controversy has existed on wither Ta additions promote or hinder the A1 to L10 ordering transformation. A series of Fe52Pt48, Fe52.3Pt46.3Ta1.4 and Fe52Pt40.7Ta7.3 thin films were sputter deposited and subsequently annealed. The as-deposited films had a change from a {111} fiber texture to {002} with the Ta additions. Annealing at 550°C facilitated the L10 order in Fe52Pt48 and Fe52.3 Pt46.3Ta1.4 films. Upon annealing at 750°C, all three compositions L10 phase transformed. Atom probe tomography revealed nano-scale clustering in the annealed Ta containing films. The formation of these clusters appeared to be a necessary initial step to allow the L10 ordering reaction to occur. Additionally, clustering at grain boundaries provided a Zener-based pinning mechanism for grain growth measured by TEM.

Biological Materials Science: Bio-Inspiration and Bio-Inspired Materials I: Hard Biomaterials

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Biomaterials Committee
Program Organizers: Jamie Kruzic, Oregon State University; Nima Rahbar, University of Massachusetts, Dartmouth; Po-Yu Chen, University of California, San Diego; Candan Tamerler, University of Washington

Monday AM
February 28, 2011

Room: 15A
Location: San Diego Conv. Ctr

Session Chairs: Jamie Kruzic, Oregon State University; Candan Tamerler, University of Washington

8:30 AM Introductory Comments

8:35 AM Keynote

Nature-Inspired Hybrid Structural Materials for Bone Repair: *Antoni Tomsia*[1]; [1]Lawrence Berkeley Lab

While the need for organ repair is being partly addressed by transplantation, the development of a systematic method to generate new organs would transform medicine. So far we have made little progress in engineering bone and cartilage even at relatively small scales. A critical component of this engineering approach is the development of porous 3D structures — scaffolds — that will provide cell support and guide tissue regeneration. Ceramics will play an important role in the development of new scaffolds but this will require materials and structures able to combine the optimum environment for bone formation with the optimized mechanical response. We will describe how new processing techniques can be combined with novel design principles taken, in part, from natural material such as nacre or bone to fabricate ceramic-based scaffolds and composites for bone repair. The critical design and material parameters as well as future research directions will be discussed.

9:15 AM

Bone Regeneration in CaP Scaffolds: The Complementary Roles of BMP-2 and Microporosity: *Amy Wagoner Johnson*[1]; Samantha Polak[1]; Sheeny Lan Levengood[2]; Aaron Maki[1]; Matthew Wheeler[1]; Sherrie Clark[1]; [1]University of Illinois at Urbana-Champaign; [2]University of Wisconsin, Madison

BMP-2 is a well-known stimulant for bone regeneration. Microporosity (<20um), has also been shown to influence regeneration in CaP scaffolds

that contain both macro (>100um) and microporosity. This study shows that BMP-2 and microporosity have different, yet complementary, roles in bone regeneration in biphasic calcium phosphate (BCP) scaffolds. Microporosity increases the volume of bone (BV), but does not influence the specific surface area (SSA), in a study using a pig mandible model. In contrast, BMP-2 influences SSA, but not BF. Both make regeneration more uniform throughout scaffolds. Bone concentrates at the defect margin in scaffolds without microporosity or BMP-2. Additionally, BMP-2 and microporosity accelerate healing up to four-fold. Thus, microporosity may be as influential in healing large defects as BMP-2, as demonstrated by rapid and uniform regeneration. Mechanisms controlling regeneration for these two variables will be discussed.

9:35 AM

Enhanced Functions of Osteoblasts on Biomimetic Nanohydroxyapatite-Grafted Chitosan Scaffolds for Bone Tissue Engineering: *Dilip Depan*[1]; Bhupinder Girase[1]; Pavan Challa[1]; Devesh Misra[1]; [1]University of Louisiana at Lafayette

We describe here three dimensional biodegradable chitosan-nanohydroxyapatite (nHA) composite scaffolds with improved mechanical, physico-chemical, and biological properties compared to pure chitosan scaffolds for bone tissue engineering. High and medium molecular weight chitosan scaffolds with 0.5, 1, and 2 wt % fraction of nHA were fabricated by freezing and lyophilization. The nanocomposite scaffolds were characterized by a highly porous structure with interconnected pores and the pore size was similar for the scaffolds with varying content of nHA. The nanocomposite scaffolds exhibited greater compression modulus, slower biodegradation rate and reduced water uptake, but the water retention ability was similar to pure chitosan scaffolds. Favorable biological response of pre-osteoblast (MC 3T3-E1) on nanocomposite scaffolds includes improved cell adhesion, higher proliferation, and well spreading morphology in relation to pure chitosan scaffold. The study underscores chitosan-nHA composite as a potential scaffold material for bone regeneration.

9:55 AM

Structure and Mechanical Properties of Bioinspired Inorganic/Polymer Multi-Layer Composites: *Gustavo Hirata*[1]; Sandra Payán[1]; Yu-Chen Chan[2]; Po-Yu Chen[3]; Jenq-Gong Duh[2]; Joanna McKittrick[2]; [1]Centro de Nanociencias y Nanotecnología-UNAM; [2]National Tsing Hua University; [3]University of California, San Diego

Abalone nacre has mechanical properties that are far beyond its constituents, CaCO3 and biopolymer, due to the well-defined structure at varying hierarchical levels. Abalone-inspired ZrN/PMMA multi-layer composites are synthesized by combining DC-magnetron sputtering and pulsed laser deposition on silicon substrates in a single chamber. SEM and high resolution TEM images show uniform polymeric layers (10-50 nm) sandwiched between nanocrystalline ZrN layers (0.3-0.5 μm). The thickness ratio of the inorganic/polymer layers is the same as that in abalone. Multi-layered ZrN/PMMA thin films with increasing interfacial roughness were also made to mimic the nano-sized asperities and bridges observed in abalone. Nanoindentation and nanoscratch tests are conducted on the synthetic thin films and abalone nacre followed by SEM and AFM characterization. Failure mechanisms are discussed and comparisons are made. This research is supported by the NSF Grant DMR 1006931.

10:15 AM Break

10:25 AM Invited

Mimicking Bone Formation Using Anionic Polymeric Process-Directing Agents: Taili Thula[1]; *Laurie Gower*[1]; [1]University of Florida

Bone is a hierarchically-structured composite which imparts it with unique mechanical properties and bioresorptive potential. These properties are primarily influenced by the underlying nanostructure of bone, which consists of nanocrystals of hydroxyapatite embedded and uniaxially aligned within collagen fibrils. There is also a small fraction of non-collagenous proteins in bone, and these are thought to contribute both to bone's formation as well as its mechanical properties. In our in vitro model system of bone formation, polyanionic peptides and proteins are used to mimic the role of the non-collagenous proteins. Intrafibrillar mineralization of collagen can be achieved using a polymer-induced liquid-precursor (PILP) mineralization process, which yields a nanostructured composite closely mimicking the nanostructure of bone. This talk will focus on our studies comparing the effectiveness of different polymeric process-directing agents, as measured by the kinetics of mineralization, degree of mineral content, and depth of penetration of mineral into dense-packed collagen matrices.

10:55 AM

Bionanomineralization through Genetically Engineered Peptides and Fusion Proteins: *Candan Tamerler*[1]; [1]University of Washington & Istanbul Technical University

Biomimetic synthesis of nanoinorganics have been a major focus in hard tissue engineering as well as developing a new generation of hybrid materials through environmentally-benign processing. Bio-based molecular building blocks are, therefore, developed for practical water-based pathways to novel biologically compatible structures. Here we use recombinant DNA technologies and biochemical conjugation to produce single or multifunctional peptides and fusion proteins and enzymes that can controllably bind to a given solid material including variety of materials, and in particular to those relevant for medical applications. Examples of the use of solid-binding peptides are given with specific implementations in self-mineralization and in forming hybrid molecular scaffolds in tissue restoration and regeneration, surface biofunctionalization of implants with antibacterial bifunctional peptide coatings, and conjugated linkers towards directed enzyme immobilization. Research is supported the NSF-MRSEC at UW, NSF-BIOMAT, TUBITAK-NSF IRES Joint Projects, and TR-SPO.

11:15 AM

Engineered Mineral-Directing Peptides for Hard Tissue Engineering: *Mustafa Gungormus*[1]; Hanson Fong[1]; Joel Schneider[2]; Candan Tamerler[1]; Mehmet Sarikaya[1]; [1]University of Washington; [2]National Cancer Institute-Frederick

During normal development of hard tissues, mineral morphogenesis is heavily influenced by soluble extracellular matrix (ECM) proteins. Polymer scaffolds, while providing architectural support, lack the molecular control over mineralization. We demonstrate use of engineered peptides to functionalize scaffolds or defect sites to exert control on mineral formation at molecular level. Mineralization-directing peptides can be engineered to interact with the mineral surfaces or precursor ions to regulate the mineral formation mimicking the ECM proteins synthesized during tissue development. In vitro solution based or cell mediated mineralization assays have shown that a precise control can be achieved over the mineral morphology and the formation kinetics. Moreover, these peptides do not require any hostile chemistry for functionalization and show advert cytotoxic effects. The results presented here indicate that these peptides may find substantial use in tissue engineering for successful restoration and regeneration of hard tissues. Research is supported by the NSF-MRSEC at UW.

11:35 AM

Structural Characterization of the Mineral Phase in Bony Tissues: A Comparative Study: *Po-Yu Chen*[1]; Maria Lopez[1]; Ekaterina Novitskaya[1]; Marc Meyers[1]; Joanna McKittrick[1]; [1]University of California, San Diego

Bone is a hierarchically structured composite of type-I collagen fibrils and mineral crystallites. We examined structural features of the mineral phase in some unique bony tissues, including antlers, turtle shells, armadillo carapaces and fish scales. A chemical method to completely remove the protein constituents without altering the original structure has been developed and verified. TEM observations of carbonated apatite crystallites derived from various bony tissues showed similar platelet-like geometry. The hierarchical structure of untreated and deproteinized samples was characterized by micro-computed tomography, optical microscopy, SEM, and AFM. The most significant finding was that the minerals retain structural integrity after completely removing protein constituents. High resolution SEM images showed minerals formed sheet-like structure, aligned in a coherent manner

along the orientation of collagen fibrils. Organized porous structure (vascular channels, lacunae, canaliculi) was observed. Comparisons between bony tissues were made. This research is supported by NSF Grant (Ceramics and Biomaterials Program) 1006931.

11:55 AM

Bone Demineralization and Deproteination Studies: A Biochemical-Kinetic Focus: *Ana Castro*[1]; Ekaterina Novitskaya[2]; Po-Yu Chen[2]; M. del Pilar Sánchez-Saavedra[1]; Gustavo Hirata[3]; Joanna McKittrick[2]; [1]CICESE; [2]UC San Diego; [3]CNyN-UNAM

There is an increasing interest on the study of bone demineralization and deproteination due to their different clinical applications and, the Material Science, also. In the present work, cortical and cancellous bovine femur bones were demineralized and deproteinated. Demineralization was performed at different temperatures and concentrations of hydrochloric acid. Deproteination was carried out using 6% NaOCl at distinct temperatures. The goal of this work was to calculate the kinetic parameters of the demineralization and deproteination reactions. Demineralization and deproteination were found to follow first-order kinetics. The rate of demineralization increased with both HCl concentration and temperature. Three different stages were clearly identified during the demineralization reactions. The activation energy for demineralization increased with increasing HCl concentration. Concerning on deproteination, the activation energy calculated was different between cortical and cancellous bovine femur bones. This work provides a better understanding of the details of bone demineralization and deproteination reactions.

Bulk Metallic Glasses VIII: Alloy Development and Application I

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

Program Organizers: Gongyao Wang, University of Tennessee; Peter Liaw, Univ of Tennessee; Hahn Choo, Univ of Tennessee; Yanfei Gao, Univ of Tennessee

Monday AM
February 28, 2011

Room: 6D
Location: San Diego Conv. Ctr

Session Chairs: Jan Schroers, Yale University; Marios Demetriou, California Institute of Technology

8:30 AM Keynote

Millisecond Thermoplastic Processing of Bulk Metallic Glasses: *William Johnson*[1]; Georg Kaltenboeck[1]; Marios Demetriou[1]; Joe Schramm[1]; [1]California Institute of Technology

A novel method is introduced for millisecond time scale experiments and processing of bulk metallic glass forming liquids. Electrical energy is used to uniformly heat BMG cylinders, plates, and sheets to a predefined temperature in the undercooled (or equilibrium) liquid at rates of order 10^6 K/s. Excellent temperature uniformity is achieved throughout the sample. The sample can be deformed under mechanical loading or injected into mold cavities to form net shapes. Measurements are carried out in millisecond time scales under "near adiabatic" conditions. The platform can be used to measure liquid specific enthalpy h(T), heat capacity c(T), viscosity, crystallization kinetics, etc.. High speed video and infrared thermal imaging are used to collect data. The new "Rapid Discharge Heating and Forming" (RDHF) method overcomes limitations created by intervention of crystallization in glass forming liquids enabling measurements and processing of both marginal and superior glass forming alloys.

9:00 AM

Bulk Metallic Glass: The Smaller the Better: *Golden Kumar*[1]; Jan Schroers[1]; [1]Yale University

Despite their superb properties, bulk metallic glasses (BMGs) find limited utility due to their macroscopic brittle nature, high costs, and difficulty of processing, particularly when complex shapes are desired. As a consequence of strong size-dependent mechanical properties of BMGs, abovementioned drawbacks can be mitigated when BMGs are used in miniature parts (< 1 cm), an application which takes advantage of BMGs' enhanced plasticity at small length scales as well the insignificant material cost associated with such parts. As an alternative to traditional metal processing techniques, thermoplastic forming (TPF)-based microfabrication methods have been developed which can process some BMGs like plastics. We will discuss the properties and fabrication of BMGs for application in small-scale devices including miniature parts, nano-imprinting, and self-assembly substrates.

9:10 AM Invited

The Production of Iron Based BMGs by Spray Forming: *Claudemiro Bolfarini*[1]; Walter Botta[1]; Claudio Kiminami[1]; [1]Universidade Federal de São Carlos

This paper describes results obtained by spray forming of iron-based alloys, whose compositions derived from rapid solidification studies aiming to obtain amorphous structures. Atomization with high gas to metal flow ratio is necessary for amorphous phase formation, and most important for keeping it by avoiding excessive reheating and consequent crystallization during deposit build-up. The amorphous regions of the deposit were formed by the participation of a great numbers of amorphous particles that were kept in the supercooled liquid region for a sufficient time in order to allow bonding of one to each other and viscous flow under the influence of the gas pressure and impacting droplets. The [(Fe0.6Co0.4)0.75B0.2Si0.05]96 Nb4 al alloy presented the highest glass forming ability among the Fe-based alloys investigated, showing a high volume percent of amorphous phase formation up to 4mm thickness of the deposit, a similar value obtained for this alloy when processed by copper mould casting.

9:30 AM

Development of Mass Production System for Zr-Based Bulk Glassy Alloys: A. Inoue[1]; *Y. Yokoyama*[1]; [1]Institute for Materials Research

For the implementation of high-reproducibility and high-quality of bulk metallic glasses (BMGs), mass production system of Zr-based BMGs was newly developed. This mass production system is composed of three machines; weighing-, master alloying- and casting-machines. The automatic weighing is composed of three processes, materials feeding, cutting, and weighing, and these three processes are automatically controlled by LabVIEW in PC. The weight deviation of automatically measure system is about 1 % in this process, we are trying much higher accuracy of this value to be less than 0.5 %. Recently. full-automatic arc furnace was commercialized by DIAVAC ltd. [http://www.diavac.co.jp/] using a patent developed by YKK co. ltd. and Tohoku university. The usage of full-automatic arc furnace suppresses the human error and dependence human's skill. By using this machine, we can control the homogeneity of alloying as a relative evaluation. This developed mass production system will accelerate the standardization of BMGs.

9:40 AM Invited

Ductility Enhancement and Size Enlargement of Bulk Glassy Alloys: H. Tokunaga[1]; K. Fujita[1]; T. Yamasaki[2]; A. Yavari[3]; P. Liaw[4]; A. Inoue[5]; *Y. Yokoyama*[5]; [1]Department of Machine Engineering; [2]Department of Material Science and Engineering; [3]SIMAP-CNRS; [4]University of Tennessee; [5]Institute for Materials Research

We examined hypoeutectic Zr-Cu-Al bulk glassy alloys exhibit high-fracture toughness and almost no degradation in mechanical properties due to structural relaxation. Furthermore, in the quaternary Zr-Ni-Cu-Al alloys, the hypoeutectic Zr70Ni16Cu6Al8 bulk glassy alloy with extremely low Young's modulus (70 GPa) and high Poisson's ratio (0.39) enables to show the distinct tensile plastic elongation at room temperature. We also examined tensile plasticity of the hypoeutectic BMG at low temperature with various strain rate. Size enlargement of metallic glass has been performed by combination of compositional optimization and production process optimization. The former focused on the glass forming ability, and the later focused on the homogenizing and purification of master alloy to achieve vitrification. The quantity of master alloy should be limited for

homogenizing. Homogenized small master alloys promote easy handling of the melting and casting processes, whereas we have to fabricate the large number of master alloys.

10:00 AM Break

10:10 AM Invited

Bulk Metallic Glasses Form Like Plastics: *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.

10:30 AM

A Novel Preparation Method for Mg-based BMG Matrix Composite with the In-Situ Ti Dispersoids: Hideki Oka[1]; Takeshi Wada[1]; Kunio Yubuta[1]; *Hidemi Kato*[1]; Akihisa Inoue[1]; [1]Tohoku University

The Mg-Cu-RE (rare earth) bulk metallic glass (BMG) is known for its high specific strength (~2.2×10^5 Nm/kg), which is higher than the conventional AZ91 alloy (~1.4×10^5 Nm/kg). However, the fracture toughness of the alloy is comparable to the ceramics, which is a serious disadvantage for the practical use. For improving the fracture toughness of the alloy, Mg-Cu-RE BMG matrix composites have been developed by dispersing the ex-situ particles such as Fe, TiB_2, SiC, Ti and so on. Although the plastic elongation of the Mg-based BMG is highly enhanced by these particles, the composite effect is achieved only in the compressive mode. In this work, we intend to introduce a novel method of dispersing in-situ α- & β-Ti particles in a Mg-Cu-Gd BMG based on an earlier work by Li et al. (Mater. Trans. 48(2007)3193) to improve the fracture toughness of the brittle Mg-based BMG.

10:40 AM Invited

Novel Net-Shape Processing of Metallic Glass by Rapid Joule Heating: *Marios Demetriou*[1]; Georg Kaltenboeck[1]; Joseph Schramm[1]; William Johnson[1]; [1]California Institute of Technology

In an effort to process metallic glass into net shape, thermoplastic methods that take advantage of the vitreous state of the undercooled liquid have been explored. These methods are limited by the relatively low stability of the liquid against crystallization, which prohibits processing within the optimum viscosity range of 10-1000 Pa-s utilized in conventional thermoplastic processes. Consequently, thermoplastic forming of metallic glass typically requires very high pressures, often approaching the flow stresses of tools. Recently, a joule heating method for processing metallic glass has been developed. The method takes advantage of the unique electrical properties of the metallic glass to heat rapidly and uniformly across the entire undercooled liquid region bypassing crystallization. Processing in the viscosity range of 10-1000 Pa-s is achievable by this method, which promotes low-cost fabrication of high-precision net-shape metallic glass parts. The rapid joule heating and forming method constitutes a powerful platform for processing metallic glass.

11:00 AM Invited

The Effect of Purification on the Glass-Forming Ability of Pd-Cu-Si Alloys: *Ke-Fu Yao*[1]; Sheng-Bao Qiu[1]; Hong-Yu Ding[1]; Yang Li[1]; [1]Tsinghua University

In present work, the Pd-Cu-Si alloy melt has been purified by fluxing with B2O3 at high temperature and effect of melt purification on the glass-forming ability has been studied. It has been found that with the purified Pd-Cu-Si ingots glassy bars with a diameter of 8 mm have been successively prepared by water quenching method and the glassy bars with a diameter of 4 mm have been prepared by copper mould casting. But without purification even for 2mm glassy bar it could not be prepared. It has also been found that the glassy alloy prepared with purified ingots exhibits much higher thermal stability than that prepared with the ingots without purification. The present results indicate that melt purification can effectively enhance the glass-forming ability of Pd-Cu-Si alloys through removing the inhomogeneous nucleus by fluxing method.

11:20 AM Invited

Processing and Tensile Tests of Amorphous Wires: *Yong Zhang*[1]; [1]University of Science and Technology Beijing

The amorphous wires of Cu50Zr46Al4 and Co36Fe36Nb4Si4.8B19.2 alloys were prepared by the In-rotation water melting spinning and Taylor-Ulitovsky technique, respectively. The amorphous wires of Cu50Zr46Al4 alloy can be in diameter of 50-120 \956m and length of 1 m, while the amorphous wires of Co36Fe36Nb4Si4.8B19.2 alloy by Taylor-Ulitovsky method can be in diameter of 10-50 \956m and length of 5 m or more, and with glass coated. The tensile strength of the Cu50Zr46Al4 amorphous alloy wires is about 1800 MPa, that of Co36Fe36Nb4Si4.8B19.2 alloy is about 2400 MPa. The tensile strength of the glass coated amorphous wire of Co36Fe36Nb4Si4.8B19.2 alloy linearly increases from 800 MPa to 2400 MPa with the ratio of the diameter of the inner amorphous wire to the outer diameter with the glass coating from 0.3 to 0.9. The wire forming parameters were compared with the glass forming ability of the alloys.

11:40 AM Invited

Formation and Properties of New Zr-Based Bulk Glassy Alloys with High Glass-Forming Ability: *Wei Zhang*[1]; Yanhui Li[2]; Chuang Dong[2]; Akihisa Inoue[1]; [1]Institute for Materials Research, Tohoku University; [2]School of Materials Science and Engineering, Dalian University of Technology

Although Zr-Al-Ni alloy system is the first Zr-based glassy alloys reported in 1990 [1], the glass-forming ability (GFA) and mechanical properties have not been investigated yet systematically. Here we report on the thermal stability, GFA and mechanical properties of Zr-Al-Ni glassy alloys with high Zr concentrations. The glassy rods with critical diameter (dc) above 10 mm were obtained in a wide composition range. The maximum dc of ~ 15 mm was formed for an off-eutectic Zr60Ni25Al15 alloy, which has the largest supercooled liquid region and □ value. Compression tests revealed that the bulk glassy alloys exhibit high yield strength of over 1.65 GPa and a distinct plastic strain [2]. The effects of additional alloying elements on the thermal stability, GFA, mechanical properties, and corrosion resistance of Zr-Al-Ni glassy alloys are also investigated.[1] A. Inoue et.al., Mater. Trans. JIM. 31, 177 (1990).[2] Y.H. Li et.al., Intermetallics. (2010), on line.

12:00 PM Invited

High Strength Amorphous and Nanocrystalline Ni-W Electrodeposits: *Tohru Yamasaki*[1]; Masako Sonobe[1]; Kazutaka Fujita[2]; Takeyuki Kikuchi[1]; Hye Jung Chang[3]; Do Hyang Kim[3]; [1]University of Hyogo; [2]Ube National College of Technology; [3]Yonsei University

Structure and tensile plastic deformation behaviors of amorphous and nanocrystalline Ni-W electrodeposited alloys have been examined. By controlling the surface roughness of the Cu-substrate, a high strength nanocrystalline Ni-16.9 at % W alloy with mesoscale Ni-segregated network structure has been prepared by electrodeposition. When the W-content was increased to 22 at. %, the alloy has consisted of amorphous phase in general. Nominal tensile strength of the Ni-16.9 at % W alloy was attained to about 2,900 MPa with the total strain before fracture was about 3 % containing the large plastic strain of about 1.0 %. A large necking phenomenon of about 30% at the fracture surface was observed, resulting the high true tensile strength at fracture of about 4,100 MPa. These results have suggested that the Ni-segregated mesoscale structure is effective for improvement of the tensile plasticity of high strength nanocrystalline alloys.

12:20 PM
Synthesis of Plastic Mg-Based Bulk Metallic Glass Matrix Composites by the Bridgman Solidification: *Liang Zhang*[1]; [1]Nanjing University of Science and Technology

The microstructures of the in situ Mg-based bulk-metallic-glass-matrix composites synthesized by the copper-mold suction casting are inhomogeneous, and the optimized mechanical properties were usually realized through alloy composition design. In this paper, Mg-based bulk-metallic-glass-matrix composites containing uniform flake-shaped precipitates with a fixed composition of Mg70Cu8.33Y8.33Gd8.33Zn5 are synthesized by the Bridgman solidification. The sizes and volume fractions of precipitates in the composites are controlled by adjusting the withdrawal velocities. Large plasticity can be obtained by only controlling the cooling condition. The Bridgman solidification is an effective way not only to fabricate the homogeneous BMG-matrix composites but also to tailor and optimize its mechanical properties.

Characterization of Minerals, Metals and Materials: Characterization Methods and Synthesis Techniques

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee
Program Organizer: Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF

Monday AM
February 28, 2011
Room: 14B
Location: San Diego Conv. Ctr

Session Chairs: Jian Li, CANMET-MTL; Sergio Monteiro, UENF

8:30 AM
Materials with Controlled Microstructural Architecture (MCMA) Fabricated by Electron Beam Melting (EBM): *Sara Gaytan*[1]; Diana Ramirez[1]; Lawrence Murr[1]; Edwin Martinez[1]; Jose Martinez[1]; Daniel Hernandez[1]; Brenda Machado[1]; Frank Medina[1]; Ryan Wicker[1]; [1]UTEP

While traditional or contemporary materials science and engineering utilizes various processing technologies including thermo-mechanical or mechanical processing to create and manipulate microstructures to control properties and establish performance features, there are currently no processing routes for the selective or systematic organization of microstructures to create architectures. In this paper we demonstrate the novel development of carbide (M23C6) precipitate architecture in the EBM fabrication of Co-base alloy components, and an oxide (Cu2O) precipitate-dislocation architecture in the EBM fabrication of Cu components. These architectures are characterized by special arrays of precipitate columns extending (generally) in the EBM build direction for the additive (powder layer) manufacturing process. Microstructures and associated architectures have been examined by optical metallography, and scanning and transmission electron microscopy.

8:45 AM
Quantifying Damage Accumulation during Ductile Plastic Deformation Using Synchrotron Radiation: *Reeju Pokharel*[1]; Jonathan Lind[1]; Xi Tan[1]; Robert Suter[1]; Anthony Rollett[1]; [1]CMU

High Energy X-ray Diffraction and absorption tomography were used to study void nucleation process in polycrystalline materials, during ductile fracture. The experimental techniques were developed at the Advanced Photon Source beam line 1-ID. Preliminary tests were performed on the tabletop microtomography system at Carnegie Mellon University. 1mm diameter Cu wire was selectively electropolished to form a neck of ~200um in the middle, to localize the strain. Synchrotron diffraction and tomography data were collected while performing in-situ uniaxial tensile test. These non-destructive techniques were able to more accurately confirm the position of the void nucleation sites in relation to the crystallographic orientation and microstructural features, such as, grain boundaries, triple points, and quadruple points. This study will aid in quantifying anisotropic microstructural distribution of strain and understanding damage accumulation, which are the main sources of failure in polycrystalline materials.

9:00 AM
Design and Implementation of Low Cost Measurement System for Determination of Thermal Conductivity Coefficient: *Juan Calderón*[1]; Nora Ramírez[1]; Leobardo López[1]; [1]ITESM Campus Toluca

One of the most important parameters to characterize the thermal behavior of any material is the coefficient of thermal conductivity. The present work proposes a design of a low cost measurement system in order to determine this coefficient with the lower possible error. The system is based on ASTM C 518 – 04 and integrated by two plates. A control system checks in real time plates temperatures in order to determine the effective power applied to the system. The contact faces of the plates contain a group of temperature sensors connected to a data acquisition system that process and records the temperature in each measurement point. With the power applied to the system, the measurement of temperatures and a process of calibration it is possible to determine the coefficient of thermal conductivity.

9:15 AM
Material Performance of TBCs at High Temperature in Moisture-Containing Environments Using a Load-Based Micro-Indentation Technique: *Jared Tannenbaum*[1]; Bruce Kang[1]; Mary Anne Alvin[2]; [1]West Virginia University; [2]National Energy Technology Laboratory

A load-based micro-indentation technique has been developed for damage assessment and non-destructive spallation detection of TBCs at room temperature. This micro-indentation technology has been further extended to the development of a high temperature (HT) test methodology. Elastic modulus calibration tests performed on H13 Tool Steel to 500°C and Haynes 230 at 1000°C displayed excellent agreement with reported values. Moreover, indentation creep tests of Haynes 230 at 1200°C were found to be in agreement with known creep exponents as well. Finally, a HT thermal flux indentation apparatus was assembled for conducting TBC turbine component testing under high temperature moisture-containing environments (e.g. =50% steam with controlled gas content temperatures up to 1250°C). Description and design considerations of this test apparatus are discussed. Preliminary tests of RenéN5/MCrAlY/APS TBC coupons in =50% steam/air environments with in-situ HT micro-indentation testing are conducted. Furthermore, the coupon is removed and examined for damage assessment at periodical intervals.

9:30 AM
Raman Spectroscopy of C-A Domain Switching in (001) BaTiO3 Single Crystals under Uniaxial Loading: Carolina Diliegros Godines[1]; *Juan Muñoz - Saldaña*[1]; Molly Gentleman[2]; Amy Bolon[2]; Luis Gutierrez Ladron de Guevara[1]; [1]Cinvestav-Queretaro; [2]Texas A&M University

Ferroelastic domain switching occurs progressively as a function of applied stress in ferroelectric-ferroelastic materials such as BaTiO3. The first steps of switching were characterized using 325 nm polarized confocal Raman spectroscopy on a (001) oriented BaTiO3 single crystal with 90º-aa and cc domain arrangements under compressive loading. Direction of the polarization vectors for a-domains from the unloaded crystal were unambiguously identified by Raman mapping, through observation of orientation intensity gradients due to contribution from subsurface a and c domains. A similar gradient was observed by measuring the elastic modulus across 90º-ac domains by nanoindentation. A set of mappings was recorded from the loaded surface as a function of applied stress that shows the switching sequence. Under this mechanical configuration out-of-plane domains from the surface become unstable and switch to in-plane domains. Raman spectroscopy also allowed identification of the preferential switching sites and A+x, A-x, A+y or A-y domains.

9:45 AM
Synthetic Generation of Annealing Twins in Three-Dimensional Microstructures: *Lisa Chan*[1]; Michael Groeber[2]; Gregory Rohrer[3]; Anthony Rollett[3]; [1]EDAX-TSL; [2]Air Force Research Laboratory; [3]Carnegie Mellon University

In face-centered cubic metals, the density of high symmetry boundaries is often maximized in order to improve grain boundary dependent properties. Twinning events have been found to be very effective in increasing the density of these high symmetry boundaries that are denoted by low sigma values for Coincident Site Lattice relationships. In this study, annealing twins are generated in synthetic microstructures by using the Σ3 cluster distribution as the target. To illustrate the capabilities of the method, a 3D dataset for a nickel alloy was collected using serial sectioning in a dual-beam FIB with OIM™. Twin-related grains were synthetically removed from the structure and subsequently regenerated. The statistics used to quantify the differences between the twin-regenerated and experimentally observed microstructures are texture, five-parameter grain boundary character distribution, and the number and area fractions of Σ3 boundaries.

10:00 AM Break

10:15 AM Invited
Experimental Characterization of Negative Thermal Expansion in Oxides: *Fernando Rizzo*[1]; Monica Ari[1]; Bojan Marinkovic[1]; Paula Jardim[1]; Roberto de Avillez[1]; Fabio Furlan Ferreira[2]; [1]PUC Rio de Janeiro; [2]ABC Federal University

For several technological applications such as Fiber Bragg gratings sensors, illumination system, integrated circuits, electronic components, optical devices, shock thermal resistance, etc, controllable thermal expansion materials, pure or composites are required. It has been the driving force in the search for crystal phases with unusually low, zero, or even negative thermal expansion (NTE). Some open-framework classes of known ceramics, such as AO2, AMO5, AM2O7, A2O5 and A2M3O12 have emerged as potential sources of crystal phases with low or NTE. The A2M3O12 family is especially attractive due to the chemical flexibility that results in a large range of variation of linear coefficients of thermal expansion (αl) after phase transition from monoclinic to orthorhombic structure. Both structures consist of corner-sharing AO6 octahedra and MO4 tetrahedra. It is assumed that the low-energy transverse thermal vibration of the oxygen in the A–O–M linkage within lower-density orthorhombic structure produces NTE.

10:45 AM
Digital Construction and Characterization of Reticulated Porous Microstructures from Sacrificial Templates: *Jason Kulpe*[1]; Stephanie Lin[2]; Jason Nadler[2]; [1]Georgia Institute of Technology; [2]Georgia Tech Research Institute

A numerical method has been developed to predict the microstructural characteristics of reticulated metal foam. The porosity of this foam is derived from sacrificial template particles of known particle size distribution, which are incorporated with other precursor components. In this approach, a porous structure is numerically generated starting with Powell's sphere packing algorithm. The resulting stochastic structure has characteristics similar to those of corresponding metal foam. Deterministic inter-sphere connections are calculated to more accurately model the pore morphology that evolves during thermochemical synthesis. Estimations of pore morphology and specific surface area of the simulated material are compared to lab-synthesized materials composed of equivalent constituent compositions. In addition, the effect of template particle size, inter-sphere connections, and the geometric constraints on pore morphology and permeability are investigated to refine microstructural evolution and design.

11:00 AM
Thermodynamic Measurement of CaO-P2O5-SiO2 System: *Takashi Nagai*[1]; Hisao Kimura[1]; Masafumi Maeda[1]; [1]The University of Tokyo

Hot metal pretreatment for de-phosphorization is a common practice in the iron and steel industry, and has been developed as an effective refining process. Although a molten flux was used traditionally in this process, a multi-phase flux, which contained liquids and solidus phases, is used today in order to avoid use of harmful element for environment such as florin. In addition, it is possible to reduce wasted flux by use of this flux because P2O5 is incrassated to solidus phase in the flux. Since the solidus phase is based on CaO-P2O5-SiO2 system, thermodynamic properties of this system are necessary to improve this process optimally. However, the available thermodynamic information on this system is limited. In this study, activity of P2O5 in CaO-P2O5-SiO2 system was measured by double Knudsen cell mass spectrometry. And the optimal condition of this process was considered.

11:15 AM
Applying Modular DOE to Improve Material Characterization and Performance: *Paul Funkenbusch*[1]; [1]University of Rochester

Design Of Experiments (DOE) methods are routinely applied in many engineering applications. However, they have not been as extensively used for material characterization and, when applied, they have often been in the form of standard product/process optimization methodologies (e.g. "Taguchi methods") that may not be well adapted to material engineering applications. The modular approach to DOE seeks to build experimental designs tailored to specific applications, by allowing the designer to make appropriate choices for each of four design element (array design, choice of the characteristic response, incorporation of variability, and data analysis). In this presentation, the use of a modular approach to build experimental designs suitable for characterizing material performance is illustrated using a variety of examples. These include development of an erosion testing protocol, identification of tool material property effects on precision grinding performance, and characterization of diamond bur performance degradation in dental burs.

11:30 AM
Selective Removal of Thiophene from Liquid Fuels over Nickel-Based Nanocrystalline Zinc Oxide: *Mohammad Islam*[1]; Jewel Gomes[1]; Hylton McWhinney[2]; Doanh Tran[1]; Sameer Pallavkar[1]; Md Islam[1]; George Irwin[1]; David Cocke[1]; [1]Lamar University; [2]Praire View A&M University

Low temperature deep desulfurization of fuels via selective adsorption of sulfur compound is receiving increasing attention worldwide in order to satisfy the upcoming environmental regulations and fuel specifications. The Ni/ZnO adsorbents were prepared by thermal decomposition and incipient impregnation method from their nitrate precursors. A model fuel (20 mL) consisting of pentane (C5H12), heptane (C7H16) and thiophene was prepared and adsorption experiments were performed by a batch method under ambient conditions. The physicochemical behaviors of the adsorbents were obtained using BET, XRD, XRF, TG-DSC, SEM/EDAX, and XPS. The sulfur concentration in the mixture was monitored by UV-vis spectrophotometry. The adsorption reaction confirmed that ZnO played a crucial role in taking up S element to convert it into ZnS. The deposition of nickel on ZnO is an innovative approach which takes advantage of the selectivity of Ni towards S species and the high adsorptive capacity of ZnO support.

11:45 AM
Extending the Effective Range of Wilkinson's Method Via a Geometry-Based Pattern Center Correction Algorithm: *Calvin Gardner*[1]; Brent Adams[1]; David Fullwood[1]; [1]Brigham Young University

Electron Backscatter Diffraction based Orientation Imaging Microscopy has generated worldwide interest in the localized recovery of the elastic displacement gradient tensor in diverse crystalline materials. Wilkinson's cross-correlation based method, with angular resolution of ±0.006°, is sufficiently precise to determine the sought-after tensor. However, the method is limited by reliance on the comparison of two like oriented patterns that must be of similar pattern center, collected within ~75 micrometers of one another. Moreover, if the absolute pattern center is not known, the method's accuracy decreases correspondingly. Both a single crystal germanium scan and a series of simulations are presented to demonstrate the problems of pattern center variation and imprecise absolute pattern center. An analytic algorithm based in system geometry is employed to correct for pattern center variations and the effective range of Wilkinson's method is extended to ~500

micrometers. Additionally, an analysis of the necessary absolute pattern center precision is presented.

12:00 PM

Thermodynamical Studies on the Carbothermal Reduction and Nitride Preparing for Vanadium Nitride: *SS Yu*[1]; WX Li[1]; ZL Ji[1]; NX Fu[2]; ZT Sui[2]; [1]Shenyang University of Chemical Technology; [2]Northeastern University

In this paper, the producing process of using V2O3 as raw material to obtain vanadium nitride was analyzed through the predominance area diagrams. The analysis shows that VN-phase can be accessed in the conversion process of V2O3 by controlling gas phase composition and temperature of the reaction system and the one-step method preparing for vanadium nitride is feasible. Thermalgravimetric analysis and X-ray diffraction were used to determine the reaction pathways of vanadium carbide, namely the following sequential reaction: V2O3 □V8C7 in higher temperature stage, the rule of vanadium nitride synthesized was established, and defined conditions of temperature for the production of the carbides and nitrides were determined. The nitridation process is simultaneous with the carbonthermal reduction. A one-step mechanism of the carbothermal reduction with simultaneous nitridation leaded to a lower terminal temperature in nitridation process for vanadium nitride produced, compared with that of carbothermal reduction process without nitridation.

Chloride 2011: Practice and Theory of Chloride-Based Metallurgy: Hydrometallurgy

Sponsored by: The Minerals, Metals and Materials Society, Canadian Institute of Metals, TMS Extraction and Processing Division, TMS: Magnesium Committee, TMS: Energy Committee
Program Organizers: Dirk Verhulst, Consultant, Extractive Metallurgy; V.I. (Lucky) Lakshmanan, Process Research Ortech, Inc.

Monday AM — Room: 19
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: V. Ram Ramachandran, Consultant; George Demopoulos, McGill University

8:30 AM

Hydrometallurgy of Chlorides: A Review of Recent Developments: *Dirk Verhulst*[1]; Vaikuntam Lakshmanan[2]; [1]Consultant, Extractive Metallurgy; [2]Process Research ORTECH Inc

Past, existing and developing processes are reviewed, with emphasis on base and precious metals as well as titanium dioxide.

8:55 AM

Development of a Novel High-Chloride Circuit for the Starfield Resources' Ferguson Lake Project: Mike Dry[1]; Niels Verbaan[2]; Ernesto Bourricaudy[2]; Michael Moran[3]; [1]Arithmetek Inc.; [2]SGS Canada Lakefield; [3]Starfield Resources Inc.

Starfield Resources is developing a novel circuit for the recovery of nickel, copper, cobalt, iron oxide and possibly also precious metals from the Ferguson Lake deposit in Nunavit, Canada. This deposit contains massive sulphides in which pentlandite is too finely disseminated in pyrrhotite for the production of a smelter-grade nickel concentrate at anywhere near an acceptable nickel recovery, meaning that commercial exploitation of this orebody is not possible via established technology. The circuit under development contains leaching, oxidation of ferrous chloride, hydrolysis of ferric chloride to saleable hematite and gaseous hydrochloric acid that is recycled and conventional steps for separating and recovering the valuable metals from the solution after hydrolysis. This paper presents a process model of the circuit and the results of experimental work on the main enabling steps, viz. the leaching, the oxidation of ferrous chloride and the hydrolysis of ferric chloride to hematite and gaseous hydrochloric acid.

9:20 AM

Synthesis of TiO2 by an Innovative Atmospheric Mixed Chloride Leach Process: *V.I. Lakshmanan*[1]; Ram Sridhar[1]; Raja Roy[1]; [1]Process Research Ortech Inc.

TiO2 is used in paints, plastics, rubber, paper, inks, textiles and other miscellaneous applications. The demand for the pigment grade TiO2 is growing at 3.0% on average and this invites for the development of innovative and cost effective processes. The current processes are not designed to recycle the reagents and thereby discharge the effluents into the environment causing an environmental concern. During this study, synthesis of TiO2 from an ilmenite concentrate was investigated using an innovative atmospheric mixed chloride leach process. The solution purification was carried out using solvent extraction in two stages. In the first stage iron removal was carried out and in the second stage titanium was extracted. The strip solution obtained from the second stage solvent extraction contained the purified titanium and from this stream pigment grade TiO2 was recovered. The barren solution was used for reagent recovery and the regenerated leach liquor was recycled.

9:45 AM

Solvent Extraction for the Separation into Nickel and Cobalt with Anionic Extractant: *Tomohiko Yokogawa*[1]; Satoshi Matsumoto[1]; Nobuhiro Matsumoto[1]; [1]Sumitomo Metal Mining Co., Ltd.

At Niihama Nickel Refinery (NNR), the plant capacity of electrolytic nickel expanded from 36kt/A to 41kt/A as nickel/cobalt mixed sulfide (MS) feed increased. At the same time, cobalt input from MS also increased. For the separation into nickel and cobalt, NNR has installed a solvent extraction process by using tri-n-octhylamine (TNOA) as anionic extractant to change into the effective purification. As a result, NNR has achieved cost reduction, more stable operation and products of better quality, comparing new process with conventional ones of oxidation and neutralization. This paper describes improvements of the operation conditions such as continuous phase control.

10:10 AM Break

10:25 AM

Integrated Plant to Recover Zinc, Lead and Silver from Crude Zinc Oxides Applying ZINCEX and PLINT Technologies: *Carlos Frias Gomez*[1]; Gustavo Diaz[1]; Daniel Martin[1]; Francisco Sanchez[1]; Ana Mejias[1]; [1]Tecnicas Reunidas

Crude zinc oxides are the major source of secondary zinc and are produced from fuming processes like Waelz kilns. The impurities of crude zinc oxides make difficult their processing in primary zinc refineries due to mainly high chloride tenor. Washing with sodium carbonate is a costly operation and chloride removal efficiency is not good enough, so further purification steps are required. The crude zinc oxides are easily treated using ZINCEXTM technology based on leaching, solvent extraction, and electrowinning. The ZINCEXTM process is commercially available and is designed to be a perfect barrier for impurities and halogens, allowing selective zinc extraction in mixed chloride and sulphate leaching solutions. Other valuable components like lead and silver can be efficiently recovered from the leaching residue using PLINT technology in chloride media. Silver is recovered as silver cement and lead is precipitated as pure lead oxide or carbonate able for smelting at low temperature.

10:45 AM

N,N´-Tetrasubstituted Malonamide Derivatives and Hydrochloric Acid Solutions: An Interfacial Study: Maria Soledade Santos[1]; Marc Font Cornella[1]; João Nuno Reis[1]; Sandra Santos Reis[1]; *Ana Paiva*[1]; [1]FCUL

N,N´-tetrasubstituted malonamides have increasingly been used as extractants for various metal ions, such as Fe(III) and platinum-group metals, from HCl media, hence it is important to understand the involved interfacial phenomena. In this work, the influence of the malonamide structure, acidity of the aqueous phase and diluent on the equilibrium interfacial tension (IT) of N,N'-dimethyl-N,N'-diphenylmalonamide and N,N'-dimethyl-N,N'-dicyclohexylmalonamide in 1,2-dichloroethane, and N,N'-dimethyl-N,N'-dicyclohexylmalonamide in toluene, with aqueous HCl, were investigated.

ITs were determined using the Du Nouy ring method. Densities of the organic phases were also determined, and used to screen solute-diluent interactions. Formation of third phases was never observed; furthermore, ITs increase with the phase separation period, constant values being obtained for separation periods larger than 1000 seconds. Each malonamide interfacial efficiency at the oil/water interface is interpreted in terms of malonamide basicity, diluent polarity and malonamide - hydrochloric acid- diluent interfacial interactions.

11:10 AM

Recovery of Precious Metals from Base Metal Sulfide Ores by a Hydrometallurgical Process: *V.I. Lakshmanan*[1]; Ram Sridhar[1]; Raja Roy[1]; [1]Process Research Ortech Inc.

Base metals and PGMs are currently recovered from sulfide concentrates using pyrometallurgical processes. These processes suffer from several limitations such as being capital intensive, scaling problems, SO2 emissions and long delay in value recognition. Alternative hydrometallurgical technologies have so far not been successful and currently total pressure oxidation and partial pressure oxidation hydrometallurgical technologies are being evaluated. Process Research ORTECH has developed a unique hydrometallurgical process to recover base metals and precious metals using mixed chloride technology. In a first stage leach, base metals are recovered and elemental sulfur is liberated from hydrogen sulfide formed during this leaching stage. During second stage leach, precious metals are recovered using proven separation technologies. Some of the advantages of the process are the recovery of elemental sulfur from sulfide sulfur, recycle of the chloride lixiviant thereby eliminating effluent treatment steps and use of excess energy from the Claus reactor for the pyrohydrolysis step.

11:35 AM

Recovery of Precious Metals from Chloride Media Using Microalgae Waste from Biofuel Extraction: *Katsutoshi Inoue*[1]; Kanjana Khunathai[1]; Keisuke Ohto[1]; Hidetaka Kawakita[1]; Kinya Atsumi[2]; Hisaya Kato[2]; [1]Saga University; [2]Denso Corporation

Crosslinked microalgae were prepared from the residue generated in the production of biofuel, by crosslinking-condensation in concentrated sulfuric acid. These microalgae were tested for adsorption of various metal ions in hydrochloric acid medium. The crosslinked microalgae exhibited high selectivity for Au(III) over other precious and base metal ions. From an isotherm experiment, the maximum adsorption capacity was evaluated to be 3.25 mol/kg of dry gel for Au(III) which was many times higher than that of the original microalgae residue. SEM and X-ray diffraction confirmed the formation of metallic gold particles, suggesting the occurrence of a redox reaction between surface functional groups on the gel surface and trivalent gold ion.

Computational Thermodynamics and Kinetics: Defects: Thermodynamics and Kinetics of Grain Boundaries, Interfaces, Surfaces and Dislocations

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Alloy Phases Committee, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee, ASM: Alloy Phase Diagrams Committee
Program Organizers: Raymundo Arroyave, Texas A & M University; James Morris, Oak Ridge National Laboratory; Mikko Haataja, Princeton University; Jeff Hoyt, McMaster University; Vidvuds Ozolins, University of California, Los Angeles; Xun-Li Wang, Oak Ridge National Laboratory

Monday AM
February 28, 2011

Room: 9
Location: San Diego Conv. Ctr

Session Chairs: Kedarnath Kolluri, MIT; Alexey Dick, Max-Planck-Institut für Eisenforschung GmbH

8:30 AM

Grain Boundary Energy in Binary Alloys: *Jonathan Stolle*[1]; Nikolas Provatas[1]; [1]McMaster University

The microstructure of a metal -- and hence grain boundaries -- determines its macroscopic properties. The structural component of grain boundary energy is analytically described by the Read-Shockley (RS) Law (1951). The RS law can be applied rigorously for the low angle grain boundares, where it is related to elastic coefficients, and empirically for large angles. Recently, Elder and Grant (2004) and Mellenthin et al (2008) have used a phase field crystal method (PFC) to demonstrate that the Read Shockley law can be used to describe grain boundary energy in pure materials. This work extends those investigations by using a binary alloy PFC model in conjunction with the Gibbs absorption theorem to systematically investigate the role of alloying impurities on grain boundary energy, wetting, and pre-melting.

8:45 AM

A First Principles Investigation into Hydrogen-Grain Boundary Binding in bcc Fe: *William Counts*[1]; Ron Gibala[2]; Chris Wolverton[1]; [1]Northwestern University; [2]University of Michigan

Hydrogen embrittlement of iron and steels is a classic but still unresolved problem in metallurgy. While hydrogen can freely move through the Fe lattice, its diffusion is hindered by lattice imperfections. Experimentally quantifying the binding energy of hydrogen to these defects has proven to be difficult. Fortunately, computational tools are ideally suited to study defect trapping because it is possible to isolate individual traps. Density function theory was used to quantify the binding energy of hydrogen to a number of Sigma boundaries in bcc Fe. We find that the H-Sigma boundary binding energies range from 0.1 to 0.5 eV. We observe that the H binding energy to Sigma boundaries with mirror symmetry across the grain boundary plane correlates with grain boundary energy, while the H binding energy for boundaries without the mirror symmetry is related to the grain boundary volume.

9:00 AM

Interaction of Grain Boundaries with Voids: *Stephen Foiles*[1]; [1]Sandia National Laboratories

The Zener-Smith model for the pinning of microstructures by precipitates assumes that the interaction energy between the precipitate and boundary is given by the product of the grain boundary energy times the area of the intersection of the precipitate with the boundary. Here we perform atomic-scale simulations of the interaction energy between a grain boundary and a void in Ni in order to test this assumption. It is found that the interaction energy is close to the Zener-Smith approximation, but that a better estimate is obtained if one includes an additional energy term that dimensionally is equivalent to a line energy associated with the boundary-void junction. Sandia

is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC0494AL85000.

9:15 AM

On the Interaction of Point Defects with Semicoherent Heterophase Interfaces: *Kedarnath Kolluri*[1]; Michael Demkowicz[1]; [1]Massachusetts Institute of Technology

We use atomistic modeling to investigate the relation between atomic structure of semicoherent heterophase interfaces and their interaction with point defects. Using Cu-Nb Kurdjumov-Sachs interfaces as a model system, we show that misfit dislocations present at such interfaces play a governing role in point defect absorption and migration. Interactions between point defects and the interface misfit networks can be described quantitatively within dislocation theory. Implications of these insights for predicting point defect interactions with interfaces in other orientations like Nishyama-Wasserman and in other semicoherent heterophase systems like Cu-V are discussed.This material is based upon work supported as part of the Center for Materials at Irradiation and Mechanical Extremes, an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under Award Number 2008LANL1026.

9:30 AM

First Principles Modeling of Dislocation/Twin Boundary and Oxygen Solute Interactions in Ti: *Maryam Ghazisaeidi*[1]; Dallas Trinkle[1]; [1]university of Illinois at Urbana-Champaign

Interaction of gliding dislocations and twin boundaries influences the plastic deformation of titanium. In addition, oxygen greatly affects both strength and twinning in Ti. Predictive models of strength and twinning rely on understanding the underlying atomic-scale deformation mechanisms. We model a [1-210] screw dislocation interacting with a Ti (10-12) twin boundary using density functional theory that provides more accurate results for dislocation/twin boundary interactions compared to previous classical potential approaches and allows for considering the effects of oxygen. Flexible boundary conditions are used to calculate the accurate geometry. We compute the dislocation core structure and transmission stresses through the boundary and repeat these calculations with an oxygen solute. This is a computationally tractable approach that marks the rst ab initio study of isolated line defects in interfaces. The results show the effect of oxygen on core structure and transmission mechanisms of a screw dislocation interacting with a twin boundary.

9:45 AM

Stacking Fault Energies in Fe-Mn-C: Ab Intio Determination of Thermodynamic and Chemical Trends: *Alexey Dick*[1]; Tilmann Hickel[1]; Afshin Abbasi[1]; Joerg Neugebauer[1]; [1]Max-Planck-Institut fuer Eisenforschung GmbH

High-Mn steels are promising candidates for light-weight high-strength materials. Their excellent mechanical properties are caused by sophisticated deformation mechanisms: the transition induced and the twinning induced plasticity. The preference of a certain mechanism is directly related to the stacking fault energy (SFE) of the material. The latter can be determined using CALPHAD based Gibbs free energies in relations that typically contain empirical, experimentally not accessible parameters. Extending the concept of density functional theory to finite temperatures by taking the effect of lattice vibrations, chemical and magnetic disorder into account, we were able to determine the chemical trends for both, the empirical parameters and the stacking fault energy. A combination of this knowledge with CALPHAD bulk data allows to predict mechanism maps over the entire composition range of Fe-Mn-C alloys.

10:00 AM Break

10:15 AM

Topological Characteristics and Events in 3D Grain Growth: *Burton Patterson*[1]; Robert DeHoff[1]; Alan Sprague[2]; Zhiwei Sun[2]; Veena Tikare[3]; [1]University of Florida; [2]University of Alabama at Birmingham; [3]Sandia National Laboratory

Topological description of the 3D grain structure throughout grain growth provides a novel insight into the process. Characteristics investigated include stability of the grain volume and face class distributions and the tendencies for different grain classes to have preferred contact and interaction among themselves and with different topological events, such as contacting a disappearing grain or making or breaking contact, respectively, to form and lose faces. Characterization of grain shape enables following the path of topological decay of grains to final disappearance. The balance of the different topological rates of the different face classes leads to the eventual stability of the face and volume distributions. Determination of these parameters is available from 3D computer simulation and serial sectioning.

10:30 AM

An Inverse Problem in Nucleation and Growth: *Mark Jhon*[1]; Siu Sin Quek[1]; David Wu[1]; [1]Institute of High Performance Computing

There has been recent interest in constructing realistic, polycrystalline microstructures based on a given grain size distribution. Such configurations may be analyzed using various simulation techniques to help develop property-structure relationships. Methods based on the reverse Monte Carlo algorithm have been shown to be very successful in reproducing size distributions. However, such methods have not yet been related to a physically meaningful growth process. In the present study, reverse Monte Carlo algorithms are developed that are consistent with diffusion-limited and interface-limited growth. These techniques are compared using characteristics of the grain distribution that are not explicitly fitted.

10:45 AM

Application of a Monte Carlo Grain Growth Simulation to Test the Growth Path Envelope Analysis Formalism: *Robert DeHoff*[1]; Burton Patterson[1]; Alan Sprague[2]; Zhiwei Sun[2]; Veena Tikare[3]; [1]University of Florida; [2]University of Alabama at Birmingham; [3]Sandia National Laboratory

During grain growth the volume of a grain varies with time along some path, V = V (V0, t), called its growth path. The collection of growth paths for all the grains in the structure is defined to be the growth path envelope for the process. Analysis of a Monte Carlo simulation of grain growth developed at Sandia National Laboratories permits the full determination of the growth path envelope. This envelope is not directly experimentally accessible for grain growth in real physical grain structures. However, an analysis exists that permits its determination from the time dependence of the grain volume distribution, which is experimentally accessible. At the base of this analysis are two critical assumptions. The simulation provides both the growth path envelope and the time dependence of the grain volume distribution, and therefore an assessment of the applicability of these assumptions in modeling real grain growth.

11:00 AM

Phase Field Simulation of Grain Growth in a System with Dissolving Precipitates: *Sina Shahandeh*[1]; Matthias Militzer[1]; [1]University of British Columbia

Controlling grain boundaries migration with addition of second phase particles has important technological applications. Conventional theories assume inert particles. However, in many processes particles are not stable at high temperature. Dissolution of precipitates changes particles size and volume fraction thus decreasing particle pinning force on the interface. On the other hand, particle dissolution injects solutes to the matrix therefore increasing solute drag effect. Phase field method has been used to simulate grain growth combined with an effective formulation for the interface drag using a friction force. The approach includes interface drag caused by both particles and solutes. Kinetics of grain growth and grain size distributions have been predicted in 3D simulations. Based on these studies conditions can

be quantified where the variable interface drag due to gradual dissolution of particles can trigger abnormal grain growth.

11:15 AM

A Phase-Field Model for Recrystallization Grain Growth: Effects of Grain Boundary Energy Anisotropy and Second–Phase Particles: *Mohsen Asle Zaeem*[1]; Haitham El Kadiri[1]; Paul Wang[1]; Mark Horstemeyer[1]; [1]Mississippi State University

We use a phase-field model to investigate the effects of grain boundary (GB) energy anisotropy and the presence of second-phase inert particles on grain growth (GG) in polycrystalline materials. Different conditions are studied. System of grains with anisotropic GB energies is considered by including models of low and high misorientation angles between adjacent grains. Systems without particles reach a steady state grain growth rate and this rate decreases by including GB anisotropy. The presence of particles significantly alters the microstructures during grain growth. It was shown that for systems with particles the critical average grain size to stop grain growth depends not only on volume fraction and size of particles but also on GB anisotropy.

11:30 AM

Phase-Field Simulation of Segregation to Stacking Fault (Suzuki Effect) in Co-Ni Based Superalloy: *Yuichiro Koizumi*[1]; Sho Suzuki[1]; Takuma Otomo[1]; Shingo Kurosu[1]; Yunping Li[1]; Hiroaki Matsumoto[1]; Akihiko Chiba[1]; [1]Tohoku University

Co-Ni based superalloy exhibits excellent high-temperature mechanical properties[1,2] and peculiar microstructural development such as: good creep resistance with dynamic strain aging, ultra-grain refinement by conventional rolling-and-annealing process and very wide extension of staking faults (SFs). These behaviors have been attributed to the chemical interaction between SFs and solute atoms, i.e. Suzuki effect. However, Suzuki effect has not been verified either experimentally or theoretically. In this study, a phase-field simulation for evaluating Suzuki effect has been developed and applied to Co-33Ni-20Cr-10Mo (wt%) alloy which exhibits the peculiar behaviors remarkably. The simulation suggested that Co-segregation and Ni-depletion can be very significant, and the deviation in concentration can exceed 10wt% at 973 K, for instance. Thus, Suzuki effect has been verified computationally. This approach may be applicable to many alloys for optimizing material properties. [1] Takahashi, Sugawara, Chiba: US Patents 2005/0016645 A1, [2] Chiba et al. Philos Mag A 1999;79:1533.

11:45 AM

Simulating Triple Junction Drag Using a 2D Phase Field Model: *Anthony Johnson*[1]; Peter Voorhees[1]; [1]Northwestern University

Two-dimensional grain growth kinetics have been shown to deviate from the classical Von Neumann-Mullins relation at low temperatures or for small grains due to limited triple junction mobility. A multiorder parameter phase field model was developed to account for triple junction mobility. In a tri-crystal system, decreasing the triple junction mobility causes the triple junction velocity to decrease and the angle to decrease well below its isotropic equilibrium value of 120\176. For a range of triple junction mobilities, this simple geometry remains consistent with the analytical theory developed by Gottstein and Shvindlerman, et al. Triple junction energies have also been measured as a function of the parameters in the phase field model. For larger 2D polycrystalline systems, preliminary simulations using this model show that the triple junction mobility significantly alters (quantitatively and qualitatively) the grain growth kinetics: triple junction angles deviate significantly from 120\176 and the growth rate is slowed.

12:00 PM

Thermal-Activated Buckling Swapping of Topological Defects in Graphene: *Chun-Wei Pao*[1]; Te-Huan Liu[2]; Chien-Cheng Chang[2]; [1]Research Center for Applied Sciences, Academia Sinica; [2]Institute of Applied Mechanics, National Taiwan University

Topological defects in graphene, such as dislocations and grain boundaries, often introduce out-of-plane bucklings. For example, symmetrical tilt grain boundaries in graphene are consisted of regular arrays of dislocations, and some of them can introduce bucklings. In this paper, we present our recent long-time-scale accelerated molecular dynamics simulation results on the swapping dynamics of these out-of-plane bucklings at symmetrical tilt graphene grain boundaries. We demonstrate that these even-distributed out-of-plane bucklings along grain boundaries can have thermal-activated deflections swapping: they can swap from deflecting upward to downward with rates up to thoudsands of times per second at room temperature. Our discovery will shed new lights on the mechanisms of defects migration in graphene, and the potential possibilities of fabricating extremely durable nanoscale actuators.

12:15 PM

Defects in Ferrite: An Ab Initio Based Description of Long-Range Elastic Effects: *Alexander Udyansky*[1]; Johann von Pezold[1]; Alexey Dick[1]; Vladimir Bugaev[2]; Jörg Neugebauer[1]; [1]Max-Planck-Institut für Eisenforschung GmbH; [2]Max-Planck-Institut für Metallforschung

We study interstitial Fe-based solid solutions containing C, N, O, B, C+N and H impurities by combining first principles simulations with the reciprocal space microscopic elasticity theory (MET), which allows us to account for long-ranged elastic interactions. The short-range chemical interactions, as well as the parameters entering the MET are obtained by density functional theory (DFT) in the generalized gradient approximation, using rather modest supercell sizes. This approach provides a direct insight into the formation mechanism of martensite and its stability limits: in particular, tetragonal states are predicted to be preferred even at low impurity concentrations due to a thermodynamically driven orientational ordering of impurities. The temperature of the martensitic cubic-tetragonal transition is found to be in agreement with available experimental data and sensitively depends on the external pressure. Furthermore, the impurity content strongly affects the vacancy concentration within the host matrix.

David Pope Honorary Symposium on Fundamentals of Deformation and Fracture of Advanced Metallic Materials: Intermetallics I

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: E. P. George, Oak Ridge National Laboratory; Haruyuki Inui, Kyoto University; C. T. Liu, The Hong Kong Polytechnic University

Monday AM — Room: 32A
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: John Bassani, University of Pennsylvania; Sharvan Kumar, Brown University

8:30 AM Introductory Comments

8:35 AM Invited

What Controls the Choice of the Slip Systems in Different Compounds with B2 Structure: *Vasek Vitek*[1]; Yi-Shen Lin[1]; Miroslav Cak[1]; Vaclav Paidar[2]; [1]University of Pennsylvania; [2]Academy of Sciences of the Czech Republic

Usual slip systems in B2 compounds are < 111 >{101} and < 010 >{101} but different slip systems dominate in different alloys. The dislocation property that often controls the choice of the slip system is dislocation dissociation and thus available stacking fault-like defects. A common wisdom has been that such fault is the ½<111 > APB but, as shown in 1981 by Pope and co-workers, it may but need not exist. The existence of stacking faults was studied by calculating {101} \947-surfaces in nine B2 compounds using a density functional theory based method. The displacement vectors of stacking fault-like defects differ from ½<111 >. Using this information and anisotropic elasticity theory we analyzed dislocation dissociations and

concluded that in order to decide which slip system dominates it is necessary to consider elastic anisotropy, vectors of stacking faults and their energies. The corresponding analysis is in full agreement with observations.

9:05 AM Invited

Recent Development of Porous Materials Based on Aluminide Intermetallics: *C. T. Liu*[1]; Yuehui He[2]; Baiyun Huang[2]; [1]Hong Kong Polytechnic University; [2]Central South University

Porous metals are of great interest as a potential engineering material in various industrial fields. Aluminide intermetallics based on TiAl, NiAl and FeAl are particularly suitable for use as porous materials for gas and liquid permeation applications because of their good oxidation resistance at elevated temperatures, high corrosion resistance in acid and alkali solutions, good high and room temperature strength, and excellent thermal shock resistance. This paper summarizes the recent work on low-cost manufacturing of aluminide porous materials by self-propagation high-temperature synthesis, where the size, shape and their distribution can be well controlled by exothermal reactions and preheating conditions. The near-net-shape porous tubes have been verified to possess excellent corrosion & oxidation resistance, microstructure stability, and mechanical strength for filtering industrial applications. *This research was supported by Natural Science Foundation and National High-Tech R&D Program of China, and internal funding from Honk Kong Polytechnic University

9:35 AM Invited

Size-Affected Flow and Intermittency in Small Ni3Al Crystals: *Dennis Dimiduk*[1]; Michael Uchic[1]; Ed Nadgorny[2]; Satish Rao[3]; Jaafar El-Awady[4]; Paul Shade[4]; Chris Woodward[1]; [1]Air Force Research Laboratory; [2]Michigan Technological University; [3]UES, Inc.; [4]UTC, Inc.

Experimental studies reveal that nano- and micrometer-scale crystals show strengthening effects, irrespective of the material type. Their flow behavior is characterized by four clear attributes: (i) a rising flow stress with decreasing sample size; (ii) stochastic variation in flow stress that exhibits wider scatter at smaller sample sizes; (iii) intermittent flow as revealed by either repeated load drops or constant-stress flow avalanches; and (iv) an extended elastic–plastic transition. These attributes are known for FCC, BCC and HCP metals, LiF and other metallic alloys. However, studies show that Ni3Al exhibits selected unique characteristics at small scales. The present work examines the microcrystal phenomenology of Ni3Al and, evaluates behavior mechanisms relative to samples size or conditions for this material. The evaluation suggests that limitations exist for understanding Ni3Al behavior from current data and, that some of these limitations pertain for microcrystal deformation behavior more generally.

10:05 AM

Plasticity of $L1_2$ Intermetallics: A New Insight Using AFM Observations: *Joël Bonneville*[1]; Christophe Coupeau[1]; [1]University of Poitiers

Atomic force microscopy (AFM) is an experimental technique that allows for the investigation of surface structures at the atomic scale, which is particularly sensitive to surface relief. When dislocations move on crystallographic planes, they produce steps at the intersection of these planes and the crystal surfaces. The steps appear as lines at the sample surface, whose tracking gives information on dislocation movements. Slip lines are therefore intimately associated with both the crystal structure and plastic deformation, and give useful information concerning the elementary dislocation processes. We intend to present a study of $L1_2$ intermetallic plasticity using AFM by investigating the number, height and length of slip lines that reflect dislocation source activity and dislocation mean free path, which are key parameters for the understanding of crystal plasticity. The contribution of D. Pope to the understanding of the plastic behaviour of $L1_2$ intermetallics will be highlighted.

10:20 AM Break

10:30 AM Invited

Properties of New Cobalt Alloys with High Volume Fractions of Ordered Precipitates: *Tresa Pollock*[1]; Akane Suzuki[2]; [1]University of California Santa Barbara; [2]GE Global Research

New cobalt-base alloys containing ordered L12 precipitates have been investigated. Two phase $\gamma - \gamma'$ microstructures are maintained with additions of Cr, Mo, Ni, Re, Ta and V to the ternary Co-Al-W system. Solidus and liquidus temperatures are 100 – 150°C higher than advanced nickel-base single crystal alloys strengthened with the L12 phase. An anomalous rise in flow stress with temperature is observed. The temperature dependence of deformation mechanisms has been studied. Single crystals have been solidified and partitioning during solidification is limited in the ternary system, suggesting a high resistance to convective instabilities. Directions for alloy design will be discussed.

11:00 AM Invited

Temperature Dependence of Yield Stress and Dislocation Dissociation in L12-Ordered Intermetallic Compounds: *Haruyuki Inui*[1]; [1]Kyoto University

L12 compounds are usually classified into three groups depending on their yield stress versus temperature curves. All these temperature dependences of yield stress have been explained in terms of the planarity of dislocation cores. While the core of 1/2[110] dislocations separated by APB on {111} is planar, those of 1/2[110] dislocations separated by APB on {001} and 1/3[121] dislocations separated by SISF on {111} are believed to be non-planar, giving rise to the rapidly decreasing yield stress at low temperatures for some types of L12 compounds such as Pt3Al and Co3Ti. However, our recent study indicates that the rapidly decreasing yield stress at low temperatures can also be observed for many L12 compounds with dislocations being dissociated into two 1/2[110] partials separated by APB on {111}. The classification of L12 compounds in terms of the temperature dependence of yield stress is discussed in relation to the observed dislocation dissociation modes.

11:30 AM Invited

Microstructure and Mechanical Properties of Dual Two-Phase Intermetallic Alloys Composed of Geometrically Close Packed Ni_3Al and Ni_3V Structures: *Takayuki Takasugi*[1]; Yasuyuki Kaneno[1]; [1]Osaka Prefecture University

The so-called dual two-phase intermetallic alloy, which is composed of $Ni_3Al(L1_2)$ and Ni solid solution (A1) phases at high temperature and is additionally refined by a eutectoid reaction at low temperature aging, according to which the Al phase is decomposed into the $Ni_3Al(L1_2)$+ $Ni_3V(D0_{22})$ phases, was recently developed. The dual two-phase intermetallic alloys showed high tensile strength, accompanied with high tensile elongation and fracture toughness over a broad temperature range, and also superior oxidation/corrosion resistance. Also, wear resistance at high temperature was very excellent and therefore attractive as high temperature tool. The obtained results are thus promising for the development of the dual two-phase intermetallic alloy as a new-type of high-temperature structural material. In the presentation, microstructure, various properties and applications of dual two-phase intermetallic alloys will be presented.

Dynamic Behavior of Materials V: Fundamentals of Dynamic Behavior

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Marc Meyers, UCSD; Naresh Thadhani, Georgia Institute of Technology; George Gray, Los Alamos National Laboratory

Monday AM
February 28, 2011
Room: 5A
Location: San Diego Conv. Ctr

Session Chair: Marc Meyers, UCSD

8:30 AM Introductory Comments

8:40 AM Keynote
Impact Testing and Dynamic Behavior of Materials: *Lothar Meyer*[1]; Norman Herzig[1]; Frank Pursche[1]; Shawky Abdel-Malek[1]; [1]Nordmetall GmbH

An overview about different testing facilities and the mechanical material behavior including monoaxial and multi axial testing under high rate loading is given. Special emphasis is laid on difficult loading conditions and loading states like high temperature and high strain loading (T>1200°C, γ>1) and multiaxial impact tests. Thereby, the impact behavior of selected materials is presented and compared under different loading conditions. A distinction is made between virgin and manufactured material behavior (e.g. welding) or pre-damaged materials . Especially, if the influence of the manufacturing history is investigated, under certain loading states the impact material properties show a dramatic difference compared to virgin material state. This paper shows some examples of different material behavior under the conditions described before.

9:20 AM
Atomistically-Informed Dislocation Dynamics Simulations of High Rate Deformation of Single fcc Crystals: *Zhiqiang Wang*[1]; [1]University of North Texas

Predicting dynamic deformation of single fcc crystals with dislocation dynamics (DD) has been a new computational approach to connecting fundamental dislocation mechanisms and microstructure evolutions to macroscopic behavior of materials. In DD method, some physical parameters are essential in controlling the behavior of individual dislocations, and thereafter macroscopic properties. Dislocation mobility is one of the most important among them, which are hard to be measured with experimental techniques while critical to the fidelity of DD methods. This talk will present some results of determining dislocation mobility under complex loading conditions with atomistic simulations. Large scale DD simulations, integrating atomistic results, are applied to predict stress-strain curves of single crystal materials in high rate deformation. It is shown that accurate dislocation mobility information is of paramount importance to develop a reliable mesoscale DD method.

9:40 AM
Plastic Response of Low- and High-Energy Grain Boundaries in Copper under Shock Loading: *Christian Brandl*[1]; Timothy Germann[1]; [1]Los Alamos National Laboratory

Previous molecular dynamics (MD) simulations have revealed that the preferred nucleation sites for partial dislocations at grain boundaries are related to the local atomic interface structure. Moreover, shock experiments discovered different post-mortem defect structures for low-energy and high-energy grain boundaries. In the present study, MD simulations are conducted to understand the structural origin of the differences in dislocation activity under shock compression, and failure upon unloading. We present MD simulations of shock/spallation loading conditions in copper bicrystals corresponding to grain boundaries studied in recent shock experiments. The defect structures produced in the MD studies are compared with the experimental post-mortem defect analysis, and the differences in the dynamic response are discussed in terms of the local grain boundary structures.

10:00 AM
Void Initiation, Growth and Collapse in BCC Tantalum: Molecular Dynamics Simulations: *Yizhe Tang*[1]; Eduardo Bringa[2]; Bruce Remington[3]; Marc Meyers[1]; [1]University of California, San Diego; [2]Univ. Nac.Cuyo; [3]Lawrence Livermore National Laboratory

The growth and collapse of nanoscale voids in BCC tantalum is investigated under different stress states and strain rates. Three principal mechanisms of deformation were identified and quantitatively evaluated: (a) shear loop emission and subsequent expansion from the surface of the void; (b) cooperative shear loop emission from slip planes that parallel to the same <111> slip direction and their combination, forming prismatic loops; (c) twinning starting at the void surface. The generation and evolution of these defects is found to be a function of stress state and strain rate. Void initiation in Nanocrystalline tantalum was also discussed

10:20 AM Break

10:30 AM Invited
Atomistic Simulations of Shock-Induced Plasticity in Tantalum: *Eduardo Bringa*[1]; J. Hawreliak[2]; N. Park[3]; A. Higginbotham[4]; [1]CONICET-Universidad Nacional de Cuyo; [2]Lawrence Livermore National Laboratory; [3]AWE; [4]University of Oxford

We have carried non-equilibrium atomistic simulations of shocks in tantalum, using 0.5-100 million atoms, with samples nearly 1 micron long and shock rise times of up to 200 ps. Our samples include perfect single crystals, single crystals with dislocation sources, and polycrystals. We find agreement with the experimental Hugoniot of polycrystalline Ta up to ~20% compression. We present an analysis of the resulting plastic behavior as a function of strain rate and shock pressure. There is no homogeneous nucleation of dislocations as in simulations of fcc metals. Dislocations originate only from pre-exsiting dislocations sources. Twinning dominates plastic deformation at high stress and strain rate, as suggested by high pressure recovery experiments from gas-guns. X-ray diffraction simulations are used to mimic future experiments that will take advantage of dynamic diffraction capabilities.

11:00 AM
The Viscosity of Liquid Tantalum*: *James Belak*[1]; [1]Lawrence Livermore National Laboratory

Tantalum is used as a prototype material for multi-scale modeling. Despite this attention, the interatomic force models have been validated with static experimental information only. Dynamic information, e.g. dislocation mobility, has proven a difficult experiment. Here we focus on a different kinetic experiment, the shear viscosity of the liquid. Because of tantalum's high melting point, limited viscosity data is available. In a tour de force experiment, Paradis et al. used electrostatic levitation to measure the relaxation of liquid droplets and extracted viscosity data. Use non-equilibrium molecular dynamics, our simulated the shear viscosity (validated on copper) agrees with the experiment at the melting point. However, the temperature dependence (activation energy) is significantly softer in the simulation, suggesting a possible reinterpretation of the experiment. Results will be presented for both the MGPT and Finnis-Sinclear models for tantalum. *Work performed under the auspices of the U.S. DOE by LLNL under Contract DE-AC52-07NA27344.

11:20 AM
"Driving Forces" for Moving Inclusion and Inhomogeneity Boundaries with Transformation Strains: *Xanthippi Markenscoff*[1]; Luqun Ni[1]; [1]University of California, San Diego

A half-space constrained Eshelby inclusion (in an infinite elastic matrix) with general uniform eigenstrain (or transformation strain) is analyzed when the plane boundary is moving from rest in general subsonic motion. The radiated fields are calculated based on the Willis expression for constrained time dependent inclusions, which involves the three-dimensional dynamic Green's function in an infinite traction-free body, and they constitute the

unique elastodynamic solution, with initial condition the Eshelby static fields obtained as the unique minimum energy solution by a limiting process from the spherical inclusion. The mechanical energy-release –rate and associated "driving force" to create dynamically an incremental region of eigenstrain (due to any physical process) is calculated for general eigenstrain. By extending the Eshelby equivalent inclusion method to dynamics, the solution for a moving inhomogeneity (different elastic constants) is also obtained. In the absence of dissipation, Noether's theorem requires that the total (with external loading) "driving force", or total dynamic J integral (--equal to the energy-release rate--) vanishes, which yields the "kinetic relation", comprising a Peach-Koehler type force and a self-force. This, in turn, results in an evolution equation for a moving plane phase boundary.

11:40 AM

Dynamic-Tensile-Extrusion Response of Polymers: *Eric Brown*[1]; George Gray[1]; [1]Los Alamos National Laboratory

Dynamic-Tensile-Extrusion (Dyn-Ten-Ext) experiments have been utilized to probe the dynamic tensile responses of polytetrafluoroethylene (PTFE) and polychlorotrifluoroethylene (PCTFE). These fluoropolymers exhibit more irregular deformation and stochastic-based damage and failure mechanisms than the stable plastic elongation and shear instabilities observed in metals. Similar to the observed ductile-to-brittle transition for Taylor Impact loading, PCTFE failure occurs at a peak velocity greater than for PTFE. However, for the Dyn-Ten-Ext PCTFE exhibits even greater resistance to failure due to the tensile stress-state. While PTFE generates a large number of small fragments when extruded through the die, PCTFE draws out a smaller number of larger particles that dynamically evolve during the extrusion process through a combination of local necking mechanisms and bulk relaxation.

12:00 PM

The Dynamic Constitutive Response of Four Light Metals: Al 7039, Al 5083, Al 5059, and AZ31B: *Sara Perez-Bergquist*[1]; George Gray III[1]; Ellen Cerreta[1]; Carl Trujillo[1]; Mike Lopez[1]; [1]Los Alamos National Laboratory

While a great number of studies have examined the uni-axial stress behavior of a wide range of light metals and their alloys under quasi-static loading conditions, less is known about the dynamic response of these same metals as a function of stress state. For this reason, the dynamic constitutive response of four light metals has been examined as a function of stress state, temperature, texture, and dynamic strain rate. Specimens of Al 7039, Al 5083, Al 5059, and AZ31B, a magnesium alloy, have been characterized prior to deformation and then dynamically loaded in compression, tension, shear, and torsion. Post mortem characterization of these specimens, using optical microscopy, scanning electron microscopy, and electron back scattered diffraction has been utilized to correlate the observed mechanical response to the microstructural evolution during loading and leading to damage and failure of these materials.

12:20 PM

Structure and Shear Resistance of an Asymmetric Tilt Grain Boundary as Function of Temperature: *Saryu Fensin*[1]; Mark Asta[2]; Richard Hoagland[1]; [1]Los Alamos National Laboratory; [2]University of California, Berkeley

Grain boundaries play an important role in governing the microstructure and deformation evolution in a material. Hence, it is important to understand the structure of grain boundaries that are subjected to high homologous temperatures. Boundaries that are susceptible to extreme structural disorder as the temperature is increased might drastically change the dynamic damage in a material under shock loading conditions. In this talk we will present the results of molecular dynamics simulations studying the temperature dependence of the structure of an asymmetric tilt grain boundary in copper. At high homologous temperatures a grain boundary can either form a disordered structure or can completely premelt. The change in grain boundary structure can be drastic enough to alter its response to an applied external force. The nature of the structural disorder in the grain boundary will be investigated by using the grain boundary width and its resistance to an applied shear strain.

Fatigue and Corrosion Damage in Metallic Materials: Fundamentals, Modeling and Prevention: Fundamentals of Fatigue Damage and Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division

Program Organizers: Tongguang Zhai, University of Kentucky; Zhengdong Long, Kaiser Aluminum; Peter Liaw, University of Tennessee

Monday AM — Room: 31C
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Angus Wilkinson, University of Oxford; Mark Hardy, Rolls Royce

8:30 AM Introductory Comments

8:35 AM Invited

High Resolution EBSD Studies of Fatigued Metals: *Angus Wilkinson*[1]; Philip Littlewood[1]; Benjamin Britton[1]; Phani Karamched[1]; [1]University of Oxford

We have used the relatively new cross-correlation-based analysis of EBSD patterns to map variations of elastic strain and lattice rotation tensors within selected grains of cyclically deformed metals. This method gives improved sensitivity of ~10^{-4} (rads) compared to ~10^{-2} rads for conventional Hough-transform-based analysis. The measured rotation (and elastic strain) gradients have been used within the Nye geometrically necessary dislocation (GND) framework to generate maps of the GND density distribution. Examples will be given for cyclically loaded OFHC Cu and Ti-6Al-4V polycrystal. The method allows the GND density distributions to be correlated with the orientation of a particular grain and those in its neighbourhood. We have also applied the technique to examine the plastic zones associated with fatigue crack tips. Examples will be given for cracks grown in single crystal Ni-based superalloy at elevated temperature and mid-Paris regime loading conditions.

8:55 AM

A Quantitative Crystallographic Model for Fatigue Crack Propagation through Grain Boundaries: *Wei Wen*[1]; Tongguang Zhai[1]; [1]University of Kentucky

A crystallographic model for short fatigue crack propagation through grain boundaries was developed to quantify the resistance of grain boundaries to crack growth. The model was based on the early discovery that the twist and tilt components of crack plane deflection across a grain boundary were the key factors controlling the crack growth in high strength Al alloys. The model developed in this work was a function of twist angle at each grain boundary that interacted with a short crack, and allowed calculation of variation in growth rate at different grain boundaries and prediction of the crack front. Further work needs to be done to incorporate other factors, such as tilt angle and Schmidt factor, into the model to simulate the growth behavior of a short fatigue crack in 3-D.

9:15 AM Invited

Cyclic Deformation and Fatigue Cracking Mechanisms of F.C.C. Crystalline Materials: *Zhefeng Zhang*[1]; Peng Li[1]; Peng Zhang[1]; Shen Qu[1]; Zhenjun Zhang[1]; [1]Institute of Metal Research, Chinese Academy of Sciences

The cyclic dislocation evolution and fatigue cracking behaviors in various f.c.c. metal and alloys, including various single crystals, Cu bicrystals and polycrystals, Cu-Al and Cu-Zn polycrystalline alloys, ultrafine-grained Cu-Al and Cu-Zn alloys, were systematically investigated and reviewed. It is confirmed that the effect of stacking fault energy (SFE) on cyclic deformation behaviors of f.c.c. single crystals is decisive and a new criterion is proposed to judge the formation of regular saturation dislocation patterns or not in various f.c.c. single crystals. In Cu, Cu-Al, Cu-Zn single crystals, fatigue cracking always nucleates along slip bands (SBs) and strain localization becomes weak gradually with decreasing SFE. The large-angle

grain boundaries (GBs) also become preferential sites for crack nucleation in Cu bicrystals and polycrystals. Fatigue cracking can also nucleate along SBs and twin boundaries (TBs) in pure Cu, Cu-Al and Cu-Zn alloys. The fatigue cracking mechanisms along SBs, GBs, TBs were compared and discussed.

9:35 AM Invited
The Effect of Pore Position in Depth on Stress Field around the Pore on Sample Surface: Zhiqiang Xu[1]; Wei Wen[2]; *Tongguang Zhai*[2]; [1]Yanshan University; [2]University of Kentucky

Stress field around a pore was analyzed as a function of the pore position in depth in the surface of a linear elastic solid using FEM. It was found that the pore depth dominated the stress field around the pore on surface and that the maximum stress was increased sharply when the pore intercepted with the surface at its top. Given the applied nominal stress, the magnitude of the maximum main stress only depended on the relative depth of the pore, while the pore size affected the stress distribution in surface. An elastic-plastic model was also used to account for the yielding effect in the region where stress was over the yield strength. The results still indicated a significant maximum stress concentration when the pore was just buried underneath the surface, but with a lowered value than that of the linear elastic model. These results were consistent with experimental observations.

9:55 AM Invited
Fatigue Weak-Link Density and Strength Distribution in High Strength Al Wrought and Cast Alloys: *Tongguang Zhai*[1]; Yuanbin Zhang[2]; [1]University of Kentucky; [2]Shandong Jianzhu University

An experimental methodology was developed to measure fatigue weak-link density and strength distribution by measuring surface crack population as a function of the maximum applied stress. The method was applied to wrought and cast Al alloys such as AA8090 Al-Li, AA2026, AA2524, AA2024 and A713 alloys where fatigue cracks were initiated at either coarse particles or pores on surface. It was found that the surface crack populations were a Weibull function of the applied maximum stress. By fitting this measured curves, fatigue weak-link density could be quantified in these alloys. The derivative of the measured Weibull function was the strength distribution of the fatigue weak-links. Fatigue weak-link density and strength distribution are materials fatigue properties and useful for alloy development and applications.

10:15 AM Break

10:25 AM
Effect of Cooling Rate on the Fatigue Life of a Nickel-Base Superalloy Used for Disc Rotor Applications: *Mark Hardy*[1]; Robert Mitchell[1]; Hang-Yue Li[2]; [1]Rolls-Royce plc; [2]University of Birmingham

It is understood that the strain hardening behaviour of precipitation strengthened nickel alloys is produced from the interactions of dislocations with gamma prime particles. Furthermore, the degree of resistance to inelastic deformation is determined by the volume fraction and size of these particles. Since the particles that are precipitated from quenching after solution heat treatment offer the most effective resistance to inelastic deformation, this study has evaluated the effect of cooling rate on the fatigue endurance of alloy RR1000. It was found from testing at 300ºC that fatigue life at this temperature is sensitive to cooling rate under conditions that produce limited plasticity. Under conditions that give rise to either significant inelastic strain or lives beyond 105 cycles, in which the deformation is predominantly elastic, endurance was found to be relatively insensitive to cooling rates. A rationale for the observed behaviour is proposed.

10:45 AM
An Energy-Based Microstructure Model to Account for Fatigue Scatter in Polycrystals: *Michael Sangid*[1]; Huseyin Sehitoglu[1]; [1]University of Illinois, Urbana-Champaign

Excessive scatter is observed in the fatigue response of a nickel-based superalloy, U720, which is partly attributed to the variability in the microstructure. There is great interest in linking the microstructure to fatigue properties using a multi-scale approach that focuses on integrating the results of atomic simulations to the continuum level. Our approach is to model the energy of a persistent slip band (PSB) structure and use its stability with respect to dislocation motion as our failure criterion for fatigue crack initiation. Through this methodology, the fatigue life is predicted based on the energy of the PSB, which inherently accounts for the microstructure of the material. From this framework, we construct simulated microstructures based on the measured distributions of grain size, orientation, neighbor information, and grain boundary character, which allows us to calculate fatigue scatter using a deterministic approach. Good agreement is shown between the model predictions and experimental data.

11:05 AM
Tail Departure of Log-Normal Grain Size Distribution in 3D Synthetic Microstructures: *Joseph Tucker*[1]; [1]Carnegie Mellon University

Techniques are discussed that have been used to produce statistical volume elements using geometric shape distributions to simulate single-phase polycrystals. Current methods assume log-normal distributions of grain size; this gives rise to an issue with nonphysical large grains that cannot be accommodated in finite simulation volumes. Other distributions which have better control of the upper tails, e.g. truncated distributions, resolves the problem and allows more realistic distributions to be generated. These points are illustrated with an example of generation of a synthetic 3D microstructure to represent a 7075-T651 aluminum alloy.

11:25 AM
Fatigue Crack Initiation Processes in a Polycrystalline Ni-Base Superalloy: *Jiashi Miao*[1]; Tresa M. Pollock[2]; J. Wayne Jones[1]; [1]University of Michigan; [2]University of California, Santa Barbara

The very high cycle fatigue behavior of a polycrystalline nickel-based superalloy René 88 DT was investigated under fully reversed loading at a frequency of 20 KHz at room temperature using an ultrasonic fatigue system. Microstructural features associated with microcrack initiation and failure were identified by orientation imaging microscopy. Focused ion beam sectioning was used to examine selected microcrack initiation sites. Deformation substructures were characterized using transmission electron microscopy. The early stages of fatigue were dominated by localized slip adjacent to twin boundaries in grains at the high end of the grain size distribution. A fatigue crack initiation model is proposed that incorporates local microstructure features in the initiation process as well as the interaction between local microstructure and small crack growth. A novel approach to quantify crack initiation life using in situ nonlinear ultrasonic analysis is described.

11:45 AM
3D Short Fatigue Crack Investigation Using Diffraction and Phase Contrast Tomography: Michael Herbig[1]; *Wolfgang Ludwig*[1]; Henry Proudhon[2]; Peter Reischig[3]; Andrew King[4]; Jean-Yves Buffière[5]; [1]ESRF / INSA Lyon; [2]MINES ParisTech; [3]ESRF / KIT; [4]GKSS-Research Center; [5]INSA Lyon

In order to investigate the influence of the microstructure on the propagation of short fatigue cracks, a combination of two synchrotron imaging techniques has been used. Grain shape and orientation are measured non-destructively in 3D by means of X-ray diffraction contrast tomography. The crack propagation is monitored at regular intervals using high resolution phase contrast tomography, a technique capable of revealing cracks of sub-micrometer thickness. A technique enabling visualization of the fracture surface orientation and, for the first time, quantification of the local crack growth rate in the bulk of the material has been developed. Its application on two beta titanium alloys gives new insight into several aspects of crack - microstructure interactions during the growth of microstructurally short fatigue cracks.

Friction Stir Welding and Processing VI: High Temperature Materials 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; Murray Mahoney, Retired from Rockwell Scientific; Yutaka Sato, Tohoku University; Yuri Hovanski, Pacific Northwest National Laboratory; Ravi Verma, General Motors

Monday AM
February 28, 2011

Room: 5B
Location: San Diego Conv. Ctr

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

8:30 AM Introductory Comments

8:50 AM Keynote

A Decade of Progress in Friction Stirring of High-Temperature Materials: *Carl Sorensen*[1]; [1]Brigham Young University

Friction stir welding and processing of high-temperature materials, such as ferrous alloys, has been reported in the literature for a little more than a decade. The past decade has seen significant improvements in tool materials, changes in tool features, new understanding of recommended parameters, and an increased number of high-temperature materials being welded. This presentation surveys the state of the art in welding of steel, stainless steel, nickel-base alloys, copper alloys, and titanium alloys. Tool materials, tool features, control parameters, tool performance, and material properties are covered. Based on the history of the past decade, prospects for the future are considered. New applications in tool steels and pipeline steels for the oil and gas industry are presented, along with demonstrations for naval applications. The most significant obstacles to commercial implementation are discussed. With continued development, friction stir processing of high temperature materials shows promis for a bright future.

9:20 AM

Development of Co-Based Alloy FSW Tool for High-Softening-Temperature Materials: *Yutaka Sato*[1]; Masahiro Miyake[1]; Hiroyuki Kokawa[1]; Toshihiro Omori[1]; Kiyohito Ishida[1]; Shinya Imano[2]; Seung Hwan Park[2]; Satoshi Hirano[2]; [1]Tohoku University; [2]Hitachi, Ltd.

The authors have developed new friction stir welding (FSW) tool that enables to weld high-softening-temperature materials, such as steels and titanium alloys. The new tool is made of Co-based alloys which are strengthened by dispersing intermetallics, Co3(Al,W), with L12 structure at high temperatures. The Co-based alloy tool can be manufactured at a low cost through a simple production method consisting of casting, heat-treatment and then cutting. They exhibit yield strengths higher than 500 MPa at 1000 degC, so that they might have great potential as FSW tool for high-softening-temperature materials. In this study, feasibility of the Co-based alloy tool to various high-softening-temperature materials, such as carbon steels, Ti-6Al-4V and so on, was examined. Change in tool shape during FSW, and the weld appearances and microstructure produced with the Co-based alloy tool will be briefly shown.

9:40 AM

Friction Stir Welding and Processing of Advanced Materials for Coal and Nuclear Power Applications: *Glenn Grant*[1]; Scott Weil[1]; Yuri Hovanski[1]; Jens Darsell[1]; [1]Pacific Northwest National Laboratory

A wide range of materials are anticipated in the next generation of power plant designs. To increase efficiency and reduce carbon emissions, boilers, heat exchangers, and turbines all will be asked to perform at higher temperature and pressure. New alloys including precipitation hardenable Ni alloys, Ferritic ODS, and high-chrome steels will be employed. A significant barrier to implementing many of these is that joining technologies are either immature or unknown. This is especially true for ODS alloys where conventional fusion welding produces a weld nugget that does not have the creep performance of the parent material. Friction Stir Welding has an opportunity to provide a joining solution to these difficult to weld materials. This presentation will outline efforts to develop the FSW weld process on several materials including Ni-alloys, ferritic ODS, and 9Cr2Mo steels. Data will be presented on high-temperature strength, creep performance, and joint microstructure.

10:00 AM

Friction Stir Welding of Alloy 22: *Bharat Jasthi*[1]; Willaim Arbegast[1]; Stanley Howard[1]; [1]South Dakota School of Mines and Technology

The main objective of this work is to characterize and compare Alloy 22 weld microstructures formed in friction stir (FS), gas tungsten arc (GTA), and electron beam (EB) welding. Topologically closed packed (TCP) phases were identified in all the as-welded condition. TCP phases that precipitated in the FS weld nugget were extremely small (~50 to 300 nm) compared to those in the EB and GTA welds. Compared to the parent material the FS weld nugget microhardness increased from (~206 HV) to (~327 HV). Only a slight increase in microhardness was observed for the EB and GTA welds. The area fraction of the TCP phases in FS weld nugget increased with aging temperature and time. The activation energy calculated for the TCP phase growth in friction stir welds was determined to be ~252.5 kJ/mol.

10:20 AM

Effect of Processing Parameters and Post-Weld Microstructure on Friction Stir Welded HSLA-65 Charpy V-Notch Impact Toughness: *Sam Sanderson*[1]; Tracy Nelson[1]; [1]Brigham Young University

Correlations between post-weld mechanical properties, post-weld microstructure and processing parameters in FSW HSLA-65 were investigated. FSW was conducted at combinations of spindle speed and welding speed to cover a range of heat inputs, from about 1 kJ/mm to 4 kJ/mm. Longitudinal, all-weld tensile tests, and Charpy V-notch (CVN) impact tests of weld centerline and heat-affected zone (HAZ) were evaluated. CVN results of weld centerline showed increased transition temperature over base metal transition temperature, with a large spread in absorbed energies near the transition temperature. Results from CVN testing of weld HAZ showed the transition temperature to be at or below base metal transition temperature. Longitudinal, all-weld tensile tests exhibited ultimate tensile strengths above ASTM A945 requirements for HSLA-65, while yield strengths were slightly below ASTM specification. The results show strong correlations between post-weld microstructure, tensile properties and weld heat input, while CVN results show only weak correlations.

10:40 AM

Friction Stir Processing of Cast Inconel 718: *Bharat Jasthi*[1]; Edward Chen[2]; William Arbegast[1]; Matthew Heringer[1]; Douglas Bice[2]; Stanley Howard[1]; [1]South Dakota School of Mines and Technology; [2]Transition45 Technologies, Inc

The objective of this work was to investigate the effects of friction stir processing (FSP) on the microstructure and mechanical properties of Inconel 718 castings. Polycrystalline cubic BN pin tools were used to process 0.35-in. thick cast Inconel 718 plates. Successful bead-on-plate welds were produced at a rotational speed of 225 RPM and travel speed of 0.5 IPM. FSP created a homogeneous microstructure and produced extremely fine grains (< 10 μm) in the weld nugget while eliminating casting defects. Post-FSP heat treatment cycles increased the strength of the FSP nugget over that of the parent material. Fracture toughness and microhardness of the welds were superior to that of parent material. This paper describes the microstructure and the mechanical properties of friction stir processed Inconel 718 castings.

11:00 AM Break

11:10 AM Invited
Fabrication of Nanostructured Tool Steel Layer by Combination of Laser Cladding and Friction Stir Processing: *Yoshiaki Morisada*[1]; Hidetoshi Fujii[2]; Tadashi Mizuno[3]; Genryu Abe[3]; Toru Nagaoka[1]; Masao Fukusumi[1]; [1]Osaka Municipal Technical Research Institute; [2]Joining and Welding Research Institute, Osaka University; [3]AMC Corporation

A fabrication process of the nanostructured tool steel layer with various carbides by the combination of laser cladding and friction stir processing (FSP) was proposed. The segregated carbide particles on the grain boundary of the matrix formed by the laser cladding with a coaxial cladding head were crushed into the carbide nanoparticles during the FSP, and were uniformly dispersed in the matrix. The carbide nanoparticles assisted in forming the fine recrystallized grains of the matrix by the FSP due to the increment in the induced stress and a pinning effect for the static grain growth. The nanostructured tool steel layer showed a higher hardness than conventional tool steel.

11:30 AM Invited
Friction Stir Welding of Oxide Dispersion Strengthened Alloy MA956: *Michael West*[1]; bharat jasthi[1]; Peter Hosemann[2]; Viswanath Sodesetti[1]; [1]South Dakota School of Mines and Technology; [2]University of California at Berkeley

Ferritic oxide dispersion strengthened MA956 alloys (ODS) were joined using friction stir welding (FSW) as a means of joining these alloys without disturbing the dispersion characteristics. Two MA956 plates of thickness 12.7 mm and 6.4 mm were successfully plasticized by friction stir welding (FSW). Fully consolidated welds were made at 400 RPM (50 mm/min) with Q60 composite pin tools and 600 RPM (13 mm/min) with W-Re pin tool. Post weld heat treatment was performed at 1000°C and 1300°C to observe the effects of heat treatment on the weld properties. Microstructural characterization, grain size analysis, and mechanical property measurements were performed as a function of weld processing parameters. Microhardness in the weld region showed a modest reduction in the strength compared to the as-processed MA956. The oxide dispersion between the bulk and the weld was analyzed by small angle neutron scattering (SANS) and scanning electron microscope (SEM).

11:55 AM
Friction Stir Processing of Ti-6Al-4V for Grain Size Reduction in Fusion Welds: *Jason Livingston*[1]; Jeff Rodelas[1]; John Lippold[1]; [1]The Ohio State University

Friction Stir Processing (FSP) is a solid-state process that produces microstructure modification in a wide range of materials (Al, Ti, Cu, Ni, and steels). In Ti alloys, such as Ti-6Al-4V, fusion welding results in coarse-grained microstructure with reduced mechanical properties (toughness and ductility). In this study, FSP was used to both modify the as-welded and base metal microstructure. FSP was conducted on beta-processed Ti-6Al-4V plate using a tungsten-rhenium tool. The as-received base material exhibited a prior beta grain size in the range from 1.2 to 1.5mm, while FSP produced a fine, equaixed alpha microstructure with grain diameters from 1 to 5 microns. Thermal data collected in situ confirmed processing temperatures were below the beta transus. Single pass GTAW welding on FSP-conditioned base metal resulted in reduced weld metal grain size, based on epitaxial nucleation and growth from the refined microstructure. Optical metallography and EBSD were used to characterize these microstructures.

12:15 PM
Microstructure and Mechanical Properties of Friction Stir Processed Grade 40 Grey Cast Iron: *Michael West*[1]; Bharat Jasthi[1]; Nicholas Smith[1]; Josiah Oduor[2]; Yong-Ching Chen[3]; [1]South Dakota School of Mines and Technology; [2]University of Tennessee, Knoxville; [3]Cummins Technical Center

Friction stir processing (FSP) of Grade 40 grey cast iron was performed using bead-on-plate friction stir welds with a PCBN pin tool. Approaches for FSP focused on the use of induction preheating and the use of a mild steel cover plate. Successful welds were made using the cover plate approach in forge force mode. Microhardness tests show high hardness (~600 HV) in regions of mixed cast iron and steel above the weld nugget. Significant refinement in the graphite structure and increase in microhardness is observed in the nugget region compared to the base material. Results of tensile testing are also presented. Transverse tensile specimens show low strength because of alignment of graphite flakes in the thermo-mechanically affected regions adjacent to the weld nugget region.

Frontiers in Solidification Science: Atomistic Simulations

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS: Solidification Committee
Program Organizers: Jeffrey Hoyt, McMaster University; Daniel Lewis, Rensselaer Polytechnic Institute

Monday AM — Room: 6E
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chair: To Be Announced

8:30 AM Invited
Characterization of the Structure, Thermodynamics and Transport at the Chemically Heterogeneous Cu/Pb Interface by Atomistic Simulation: *Brian Laird*[1]; J. Pablo Palafox-Hernandez[1]; Mark Asta[2]; [1]University of Kansas; [2]University of California-Berkeley

Using MD simulation, we examine the equilibrium interface between crystalline Cu and liquid Pb, modeled. This interface is characterized by the calculation of density, energy, stress and diffusion profiles, and by Fourier analysis of the interfacial layers for two crystal orientations: (100) and (111). At 625 K, we observe significant surface alloying in the first layer of the (100) interface, consistent with earlier investigations. No surface alloying is seen in the (111) orientation; however, a "prefreezing" layer of crystalline Pb, approximately 2-3 lattice layers in thickness, is observed to form at the (111) Cu surface.These layers are hexagonal with a lattice spacing that is 33% larger than the underlying Cu (111) planes and rotated by an angle of ~6\176 relative to the Cu lattice. Further simulations show that the preefreezing layers significantly enhance heterogeneous nucleation in the (111) orientation relative to (100).

9:00 AM Invited
Finite Size Effects in Molecular Dynamics Simulations of Nucleation and Growth: *James Morris*[1]; Trevor Pate[2]; Lujian Peng[2]; [1]Oak Ridge National Laboratory; [2]University of Tennessee

Molecular dynamics provides a direct, unbiased approach for simulating the processes associated with phase transformations. However, the small size and time scales can limit the applicability of these methods. In this talk, recently observed finite-size effects in simulations of both crystal nucleation times and growth are reviewed: both nucleation dynamics and initial grain structures have been shown to have important size effects, sometimes requiring millions of atoms to converge. Both size effects are statistical in nature. We examine the connection between these two effects, and demonstrate that simple arguments can provide estimates of the relevant size scale and how this depends on undercooling conditions. Moreover, the nucleation times are well predicted by nucleation theory, without fitting parameters, in contrast to experimental work in both colloids and glasses. This research was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, Office of Basic Energy Sciences.

9:30 AM Invited
Heterogeneous Nucleation of Solid Al from the Melt: Molecular Dynamics Studies: *Junsheng Wang*[1]; Peter D. Lee[1]; Andrew Horsfield[1]; Peter Brommer[2]; Udo Schwingenschloegl[3]; [1]Imperial College London; [2]Universität Stuttgart; [3]King Abdullah University of Science and Technology

Nucleation of solid Al from the melt by TiB2 is well established, but the atomic scale mechanisms are not fully understood. It is believed to be a two step process: Al3Ti is formed which then nucleates solid Al. Using molecular dynamics (MD) simulations with embedded atom method potentials we simulated the nucleation and early stages of growth of Al on Al3Ti. A strong anisotropy in the growth depending on the crystal orientation was found and explained in terms of interfacial energies. Using Density Functional Theory MD the early stages of Al nucleation on TiB2 were simulated. We find no signs of incipient growth of solid Al at the B-terminated surface, but see fcc-like ordering at the Ti-terminated surface. For the Al3Ti substrate, fcc-like structures are observed which extend rapidly into the melt.

10:00 AM Break

10:15 AM Invited
Molecular Dynamics Simulations of Alloy Rapid Solidification: *Mark Asta*[1]; Harith Humadi[2]; Yang Yang[3]; Brian Laird[4]; Deyan Sun[3]; Jeff Hoyt[2]; [1]University of California, Berkeley; [2]McMaster University; [3]East China Normal University; [4]University of Kansas

Molecular dynamics (MD) simulations are employed to investigate the kinetic properties of alloy crystal-melt interfaces under rapid-solidification conditions. The MD simulations yield results for velocity dependent partition coefficients, and the relationship between interface velocity and thermodynamic driving force. Both sets of results are compared to available sharp-interface models for solute trapping and solute drag. The MD results for fcc-based alloy systems can be characterized as follows. For interface mobilities, the MD results are best described by models that account for some degree of solute drag. Results for solute trapping are best described by models that account for an abrupt transition to partitionless growth at a critical velocity. A key finding in the MD simulations is the presence of a significant degree of crystalline anisotropy and composition dependence of interface mobility. Preliminary results for bcc alloys will also be described.

10:45 AM Invited
Heterogeneous Nucleation of Liquid at Grain Boundaries: T. Frolov[1]; *Y. Mishin*[1]; [1]George Mason University

At temperatures approaching the bulk melting point, many grain boundaries exhibit premelting by developing liquid-like layers. Some boundaries can be overheated above the melting point until the material suddenly melts. Both premelting and melting processes can involve nucleation of liquid droplets existing in unstable equilibrium with the boundary. We report on atomistic simulations of grain boundary premelting, melting and formation of critical liquid nuclei at grain boundaries in copper. We show, in particular, that nuclei can be effectively stabilized using the NVE ensemble of molecular dynamics, allowing systematic studies of the nucleus size and shape depending on temperature. By varying the system size, different morphologies of the nuclei are observed, including the cylindrical (quasi-2D) and the lens (3D) shapes. We demonstrate how a cylindrical nucleus can break up into several lens-shape nuclei when the system size is increased. The results are compared with homogeneous liquid nucleation inside the crystal.

11:15 AM Invited
Molecular Dynamics Study of Solid-Liquid Interface Migration in FCC Metals: *Mikhail Mendelev*[1]; [1]Ames Laboratory

The recent MD simulation results for the solid-liquid interface (SLI) migration in fcc metals will be discussed. First, it will be shown that the generation of latent heat at the moving SLI during MD simulations can lead to significant underestimations of the interface mobility. Second, the kinetic coefficient for all systems will be compared to theoretical predictions. It will be shown that near the melting temperature the magnitude of the kinetic coefficient correlates well with the value of the diffusion coefficient in the liquid. Finally, the results of the MD simulation of the SLI migration at very low temperatures will be presented. It will be shown that the results of such simulations are extremely sensitive to the way how the liquid part of the simulation cell was created.Work at the Ames Laboratory was supported by the Department of Energy, Office of Basic Energy Sciences, under Contract No. DE-AC02-07CH1135.

Geek Speak on the Hill: Communicating Science to Policy Makers

Sponsored by: The Minerals, Metals and Materials Society, TMS: Public and Governmental Affairs Committee
Program Organizers: Jud Ready, Georgia Tech; Marlit Hayslett, Georgia Tech

Monday AM — Room: 17B
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chair: Marlit Hayslett, Georgia Tech

8:30 AM Introductory Comments by Kevin Hemker, Chair, Public & Governmental Affairs Committee

8:35 AM Invited
Recap of Congressional Visit Days: Iver Anderson[1]; [1]Iowa State University

TMS, MRS, ASM, FMS, IEEE, and numerous other societies visit congressional leaders each year to provide them with scientific information (typically in the spring: Next for TMS is April 6-7, 2011). This talk will include an overview of that "CVD" program as well as details for upcoming events.

8:55 AM Invited
Legislative Process 101: Arnie Thomas[1]; [1]Senior Vice President, Client Relationships, CQ Rollcall Group

The many stakeholders, nuances and steps involved in legislative public policy will be covered. The role of committees, special interests, lobbyists and other aspects of the legislative process are included.

9:15 AM Break

9:30 AM Invited
Case Studies in Reality Colliding with Science-Policy: *Jim Treglio*[1]; [1]Consultant, Technology and Marketing, Molecular Metallurgy

The speaker will share real-world examples where scientific interchange with policy makers could have resulted in a different outcome.

9:50 AM Invited
Communicating Science to Policy Makers: Marlit Hayslett[1]; [1]Director, Office of Policy Analysis and Research

This talk will encompass how to translate technical jargon into a language understandable by federal, state and local public policy makers.

10:10 AM Invited
Tools and Techniques for Being an Effective Communicator: Gina Schatteman[1]; [1]University of Iowa

This session will review tactics and approaches for effective delivery of scientific information to public policy makers. Oral and written testimony, letters to representatives, constituent visits and other types of interactions will be discussed.

10:30 AM Break

10:45 AM Panel Discussion
"The Congress shall promote the progress of science..." US Constitution; Article 1, Section 8

Each speaker will participate in a strategic discussion of how members of the materials community can effectively interact with federal, state and local legislatures to influence public policy for the betterment of society.

11:45 AM Concluding Comments

General Abstracts: Electronic, Magnetic and Photonic Materials Division: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS: Electronic Materials Committee, TMS: Electronic Packaging and Interconnection Materials Committee, TMS: Energy Conversion and Storage Committee, TMS: Magnetic Materials Committee, TMS: Nanomaterials Committee, TMS: Thin Films and Interfaces Committee

Program Organizers: Long Qing Chen, Pennsylvania State University; Sung Kang, IBM Corporation; Mark Palmer, Kettering University

Monday AM
February 28, 2011

Room: 16B
Location: San Diego Conv. Ctr

Session Chair: To Be Announced

8:30 AM

Charged Point Defect Configurations, Domain Stabilization Effects, and Ferroelectric Aging: *Tianle Cheng*[1]; Yu Wang[1]; [1]Michigan Technological University

Configurations of charged point defects and resultant internal electric field are investigated based on point ion model to understand ferroelectric aging phenomenon. Analysis of the energetics of various defect configurations reveals that the interactions among point defect dipoles play a most important role, which leads to a formation of asymmetric L-shaped defect configuration, generating internal electric field sufficient to stabilize ferroelectric domains. Such short-range ordering of charged point defects is believed to be the origin of ferroelectric aging. To study domain stabilization effect in aged ferroelectrics, domain-dependent internal electric field associated with the short-range ordering of charged point defects is incorporated into phase field ferroelectric model. Computer simulation is performed to estimate the internal electric field strength. Clausius-Clapeyron-type thermodynamic analysis of field-induced ferroelectric phase transition is used to evaluate aging-associated internal electric field magnitude from available experimental data, which is in agreement with the theoretical analysis and computer simulation.

8:50 AM

Correlated Nucleation and Self-Accommodating Kinetic Pathway of Ferroelectric Phase Transformation: *Jie Zhou*[1]; Tian-Le Cheng[1]; Yu Wang[1]; [1]Michigan Technological University

Mechanisms of nucleation and growth of domains during ferroelectric phase transformation are investigated by using theoretical and computational approaches. It is shown that ferroelectric phase transformation exhibits some interesting behaviors due to strong long-range dipole-dipole-like interactions involved in the system. Incorporation of electrostatic and elastostatic energies into the classical Landau-Ginzburg-Devonshire theory effectively modifies the coefficients of the polynomial free energy function and introduces extra energy barrier for ferroelectric phase transformation. It is found that independent nucleation of ferroelectric phase in the context of classical nucleation theory is impossible, because electrostatic interaction generates an insurmountable energy barrier to isolated nucleus. Phase field simulation reveals that, in order to circumvent such an energy barrier, nucleation exhibits strong spatial correlation and self-organization behaviors from the very beginning, and ferroelectric phase transformation proceeds via spatial and temporal evolution of self-accommodating domains that provide a low-energy kinetic pathway throughout the entire phase transformation process.

9:10 AM

Bipolar Resistive Switching Behavior in Ti/MnO2/Pt Structure for Nonvolatile Memory Devices: Min Kyu Yang[1]; Sun Young Choi[1]; *Jeon Kook Lee*[1]; [1]Korea Institute of Science and Technology

We examined the electrical properties of Ti/MnO2/Pt devices with stable and reproducible bipolar resistive switching behavior. The dependency of the memory behavior on the cell area and operating temperature suggest that the conducting mechanism in the low resistance states is due to the locally conducting filaments formed. X-ray photoelectron spectroscopy showed that non-lattice oxygen ions form at the MnO2 surface. The mechanism of resistance switching in the system examined involves the generation and recovery of oxygen vacancies with the non-lattice oxygen ions. The Ti/MnO2/Pt device has potential applications in nonvolatile resistive switching memory devices due to the reasonable switching endurance over the course of 105 cycles, long term retention at 85 ºC and a uniform distribution of both resistance states and operation voltage. A high content of non-lattice oxygen ions exists on the MnO2 film surface. The switching behavior can be explained by the generation and recovery of oxygen vacancies with non-lattice oxygen ions.

9:30 AM

Preparation and Characterization of Oxide Thin Films for the Resistance Random Access Memory (RRAM) Application: *W. Z. Chang*[1]; J. Chu[1]; S. F. Wang[2]; [1]National Taiwan University of Science and Technology; [2]National Taipei University of Technology

RRAM is considered as a promising candidate for next-generation memory application because of high-density, fast write/read access, low energy operation, and high performance of endurance and retention. Many oxide systems such as NiO, TiO2, and SrZrO3 have been proposed for this application. In this study, sputtered HoScO3 (HSO) and TiOx thin films with working atmospheres of different Ar/ O2 flow ratios have been characterized for the RRAM resistance switching (RS) behavior. After deposition, HSO and TiOx thin films with a thickness of approximately 50 nm are post-annealed in O2 and N2 atmospheres at various temperatures. As-deposited HSO films exhibit RS behavior, except at an Ar/O2 ratio of 10/10, while as-deposited TiOx films exhibit RS behavior only with high O2 pressures. HSO samples annealed in N2 atmosphere exhibit a better RS behavior than those annealed in O2. However, an opposite trend is observed in the case of TiOx films.

9:50 AM

Investigation of Electronic Properties for Nano-Titania/Metal-Ion-Doped Titania Semiconductor Prepared by Sol-Gel Methods: *Leo Chau-Kuang Liau*[1]; [1]Yuan Ze University

Semiconductor homojunction devices were designed and fabricated by coating a metal-ion-doped TiO2 film on top of a TiO2 nanoparticle film. The sample films were prepared with synthesized sol-gel TiO2, verified to be nano-size particles with anatase structure. The semiconductor characteristics of the ion-doped and undoped films were analyzed by current-voltage (I-V). Results showed that the semiconductor properties of TiO2 were greatly influenced by doping with different types and amounts of metal ions, i.e. Fe2+, Cr3+, Zn2+, and Ag+. The semiconductor characteristics of the TiO2/metal-ion-doped TiO2 devices, such as onset voltage and rectifying curves, were affected by the fabrication of the ion-doped layer. The fabricating conditions of the ion-doped layer, i.e. thickness, and types and amounts of metal ions, can change the electronic properties of the device.

10:10 AM Break

10:30 AM

Effects of Structural Heterogeneities on Magnetization Processes in FePt Crystals: Yan Yang[1]; *Jianyang Li*[1]; Yongmei Jin[1]; [1]Michigan Technological University

Magnetic domain evolutions in FePt crystals of different structural heterogeneities are studied by micromagnetic modeling. In particular, {110} transformation twins and {111} annealing twins in $L1_0$ phase, and $A1+L1_0$ two phase systems are considered. Computer simulations reveal detailed

magnetization processes through domain wall motion and magnetization rotation, and show strong interactions of magnetic domain walls with twin boundaries and inter-phase interfaces. It is found that magnetostatic interaction between magnetic domains across interfaces generates local magnetic charges, which produce heterogeneous internal magnetic field. The competition between the internal and external magnetic fields under the constraint of high magnetocrystalline anisotropy of $L1_0$ phase determines magnetic domain structures and their evolution paths, which explains the different characteristics of magnetic properties of FePt crystals with different structural heterogeneities. The influences of size and morphology of heterogeneities on magnetic processes are discussed.

10:50 AM

Phase Formation, Microstructure and Magnetic Properties of Rapidly Solidified SmCo Alloys Modified with Hf and C: *Shampa Aich*[1]; Jeffrey Shield[2]; [1]Indian Institute of Technology; [2]University of Nebraska-Lincoln

A series of $(Sm_{0.12}Co_{0.88})_{95}Hf_{5-x}C_x$ (x = 0, 1, 2, 3, 4 and 5) alloys were arc-melted in a TIG arc melting furnace and then melt-spun on a copper wheel surface at a wheel speed of 30 m/s. X-ray diffraction indicated that the primary phase was $SmCo_7$ with the metastable $TbCu_7$-type structure. While rapid solidification of binary Sm-Co alloys results in coarse grains (grain size ~ 1 μm) and a coercivity of 0.8 kOe, combined additions of Hf and C resulted in effective grain refinement (grain size ~ 100-200 nm). The reduction in grain size resulted in dramatic increase in magnetic properties dependent on the Hf/C ratios. The coercivity (H_{ci}) increased systematically from approximately 6 kOe to 17 kOe and the magnetization (M_s) decreased from 92 emu/g to 55.3 emu/g with a maximum reduced remanence (M_r/M_s) value of 0.78 as the Hf/C ratio varied from 1/4, 2/3, 3/2, 4/1 to 5/0.

11:10 AM

Structure and Magnetic Properties Characterization of Electro-Deposited Co37Fe63 Containg Oxygen for Magnetic Recording Applications: *Shereen Elhalawaty*[1]; Ray Carpenter[1]; Jinnie George[2]; Stanko Brankovic[2]; [1]Arizona State University; [2]University of Houston

Soft and high magnetic moment Co37Fe63 alloys have been electro-deposited with variable additives on Cu/Ti/Si substrate. The correlation between structure and magnetic properties has been investigated. TEM showed that the crystal structure of the deposits was BCC with a <111> texture, and 10 to 20 nm grain size. Oxygen in the deposited film has been identified by High Annular Dark Field STEM combined with EDS and EELS. The oxygen content in the deposits has also been analyzed using RBS. SIMS showed that both oxygen and hydrogen are present in the deposited CoFe films. The magnetic properties of the deposits have been measured by magnetometer quantifying the impact of oxygen and hydrogen on the saturation magnetic flux density BS. Analytical results showed that the oxygen was mainly distributed along the grain boundaries in the CoFe film. Where oxygen was present in the film, the Fe content was enhanced relative to Co.

11:30 AM

High Permeability Co-Hf-Ta Thin Films: *Shu-Wen Huang*[1]; Yuan-Tai Lai[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University

High permeability Co-Hf-Ta thin films were fabricated by adjusting the Hf content from 1 at.% to 8 at.%. It was revealed that appropriate Hf addition can effectively reduce the Co grain size and improve magnetic properties. Magnetic properties were shown to strongly depend on the microstructure of Co-Hf-Ta films. X-ray diffraction and transmission electron microscope diffraction patterns revealed that appropriate Hf addition would promote the formation of nanocrystalline structure. Such microstructure could be beneficial to obtain soft magnetic properties and a superior permeability (μ'). In this study, by optimizing the Hf content, the film with Hf concentration of 2.81 at.% exhibits excellent soft magnetic properties: high saturation magnetization (4pMS ~ 13.6 kG), and low coercivity (HC ~ 0.6 Oe). The effective permeability of the film reaches 800 and remains nearly unchanged up to 1 GHz.

11:50 AM

Tungsten Doping Effect on Thermoelectric Properties of Heusler Fe_2VAl Alloy: *Haruka Morishita*[1]; Masashi Mikami[1]; Kimihiro Ozaki[1]; Keizo Kobayashi[1]; [1]National Institute of Advanced Industrial Science and Technology

A Heusler alloy, Fe_2VAl, is a promising candidate for thermoelectric power generation near room temperature because of its high thermoelectric power factor *PF* (=S^2\963;, where *S* is the Seebeck coefficient, \963 is the electrical conductivity). However, the thermoelectric figure of merit *Z* (=$S^2\sigma/\kappa$ where κ is the thermal conductivity) of this alloy is poor by reason of its high thermal conductivity (κ =28 W/mK). We had reported that microstructural refinement using powder metallurgy technique enhances phonon scattering at grain boundaries, resulting in the reduction of κ : 16 W/mK. In this study, we investigated the effect of substitution of W, which has the larger atomic mass, for V to achieve thermal conductivity reduction within grains by the mass-difference scattering. The κ of 6 W/mK was then obtained at 300 K for $Fe_2V_{0.9}W_{0.1}Al$. W doping effect on electric properties will also be presented.

12:10 PM

The Introduction of Nano-Scale Inclusions in to Bulk MgB2 via Infiltration and Growth Process: *Hari babu Nadendla*[1]; Anthony Dennis[2]; Yunhua Shi[2]; David Cardwell[2]; [1]Brunel University; [2]University of Cambridge

It is well established that magnetic flux pinning in MgB2 bulk samples can be enhanced significantly by the introduction of nano-sized, non-superconducting second phase particles into the MgB2 phase matrix, and this has been achieved primarily by co-sintering a mixture of MgB2 and nano-scale second phases. The density of material fabricated by this technique, however, is low and bulk samples are typically porous, with a relatively low critical current density (Jc). In this paper, we report an extension of the infiltration and growth process to the fabrication of high density (90-95% of theoretical), bulk MgB2 that contains nano-scale inclusions (SiC, Y2O3, and ZrO2). This process also allows to fabricate complex shaped bulk MgB2 superconductor. The influence of the presence of these inclusions on Jc as a function of external field in the temperature range of 5 K - 38 K is investigated in detail for various nano composites.

General Abstracts: Light Metals Division: Primary Production and General Issues

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee, TMS: Energy Committee, TMS: Magnesium Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Alan Luo, General Motors Corporation; Eric Nyberg, Pacific Northwest National Laboratory

Monday AM — Room: 17A
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chair: Eric Nyberg, Pacific Northwest National Laboratory

8:30 AM

A Model for the Collapse of the World Trade Center: *Christian Simensen*[1]; [1]SINTEF

Two airplanes of Boeing 767 flew into the Twin Towers in the morning of September 11th 2001. The crashes caused petrol to come on fire, and the airplanes to be fragmented. They were subsequently heated to a high temperature. Aluminium alloys in the airplanes were melted at 660°C while the surrounding building was only heated to a much lower temperature due to suitable insulation. When the temperature reached 750-800°C, the approximately 30 ton melt managed to stream down to the lower floors. There it came in contact with water from the sprinkler devices and other sources. The encounter resulted in a series of fierce explosions by generation of hydrogen gas and local heating to high temperatures well above 1000°C. A section of the buildings was torn apart, and the upper part fell on the bottom part so the whole building collapsed.

9:00 AM

Analysis on the Market Developing Conditions of Prebaked Carbon Anodes for the Aluminium Industries: Guanghui Lang[1]; Chunyu Fu[1]; *Ronald Logan*[1]; Yan Li[1]; [1]Sunstone

In recent years, with the sustained and rapid development of the aluminium electrolytic industries worldwide, an unprecedented growth of Chinese prebaked carbon anode production has been achieved. The high-quality and abundant sources of raw materials for anode production, unique production technologies, experienced operators, governmental support of research, and efficient logistics have made the further development of large-scale and integrated anode production facilities possible. China is positioned to become the production base of prebaked carbon anodes for the electrolytic business all over the world in the future.

9:20 AM

Qatalum – Organizational Challenges in Starting up a Modern Smelter: Roald Holten[1]; Lene Solli[1]; *Jan Arve Haugan*[2]; [1]Norsk Hydro; [2]Qatalum

The Qatalum smelter in Qatar started its operation in December 2009, with target production of 585 kt by the end of 2010. Two experienced industrial partners teamed up to create this modern aluminium smelter: Hydro with its strong position in reduction technology and Qatalum Petroleum as a key provider of industrial infrastructure in Qatar. Bringing this new large plant up to speed demands an efficient organization with competent and well-trained employees as well as well-functioning organizational structures and processes. The diversity in having thousand employees of 32 nationalities and from 35 company cultures represents a challenge, as does serving demanding customers with customized, high-quality casthouse products. The Qatalum Production System, a local adaptation of Hydro's own production system, has been devised as the main mechanism to align the organization and ensure quality in all processes. This presentation summarizes experiences and main learning points from the first year of operation.

9:40 AM

Building Qatalum – A Large, Modern Smelter: *Tom Rotjer*[1]; Erik Smith[1]; Anton Husøy[1]; [1]Norsk Hydro

On December 19 2009 production started at the Qatalum aluminium smelter, a joint venture between Hydro and Qatar Petroleum. With this the main milestone of the USD 5,7 billion project was successfully achieved, and a modern smelter based on the HAL300 reduction technology was ready for ramp-up of production within cost, on time and according to specifications. A construction village was built to accommodate the huge number of workers, providing welfare standards that Hydro are proud of. Likewise, environmental considerations, safety precautions and working conditions were all along on top of the priority list of the project team. The project was managed by Hydro Projects and was divided into 21 main contracts based on an EPC procurement strategy. This presentation provides detailed insights into how the project team handled the technical and organizational complexities during its work to construct one of the largest aluminium smelter ever built in one step.

10:00 AM

Removal of Fluoride from Waste Water of Aluminium Smelter by Aluminium Ion Loaded Ion Exchange Resin Method: *Balakrushna Padhi*[1]; Arun Kumar Sharma[1]; [1]National Aluminum Company Limited

Strong Acid Cation resin has sulphonic acid functional group (H+ form) possesses appreciable defluoridation capacity and it has been enhanced by chemical modification into Al3+ form by loading of Aluminium ions in H+ form of resin. The defluoridation Al3+ forms was found to be 480 mg F-/kg, at 15 mg/L initial fluoride concentration. The nature and morphology of sorbents are characterized using XRD,FTIR and SEM analysis. The fluoride sorption was explained using the Freundlich, Langmuir and Redlich–Peterson isotherms and kinetic models. The calculated thermodynamic parameters such as □G°, □H°, □S° and sticking probability (S*) explains the nature of sorption. A Defluoridation Plant is in service at aluminium smelter of National Aluminium Company to treatment the waste water for removing Fluoride and reutilise the water to minimise the water loss.

10:20 AM Break

10:40 AM

Theortical Study of Light Weight Materials on Replacement of Traditional Materials: *Pradeep Raja*[1]; [1]Government College of Technology

Materials innovation and application are increasingly vital to sustain advanced manufacturing and modern methods of construction.The demand for lighter designs is growing for a broad spectrum of products. In this juncture, the paper aims at the study in development of cost-effective, high-strength, lightweight materials that reduces the weight of a product without compromising cost, performance or safety that is necessary to compete in today's global market. As a result of more stringent requirements for improved fuel economy and emissions, there is a growing trend to substitute Al and Carbon-fiber for conventional steel and cast irons in vehicles. However, because of their different tribology, strength, and ductility, light-metal castings require improved foundry procedures and more sophisticated design rules before product engineers will use them in larger quantities. A major challenge for lightweight materials is the ability to produce a functional component at an acceptable price.

11:00 AM

Corrosion Behavior of Cermet Anodes in Na_3AlF_6-K_3AlF_6-based Baths for Low-Temperature Aluminum Electrolysis Cells: *Guihua Wang*[1]; Xiaofei Sun[1]; Wenshan Wang[1]; Deren Wang[1]; Yedong He[1]; [1]University of Science and Technology Beijing

5Cu/ (10NiO-90$NiFe_2O_4$) cermet anodes were tested in different Na_3AlF_6-K_3AlF_6-based electrolytes at three temperature regimes: 740-780, 820-840, 940-960 °C. Post-electrolysis microscopic examination was carried out with SEM-EDS and XRD. The Fe, Ni and Cu contents in the bath and aluminum were measured with ICP-AES, according to which the corrosion rate was calculated. The results indicate that low temperature electrolysis can reduce the corrosion rate effectively. However, as temperature decreases too near the liquidus line, solid cryolite can form on the cathode, and high current density can be caused locally, which is detrimental to the anodes. The cermet anodes show best anti-corrosion properties when electrolysis temperature is controlled at 830-840 °C, and the corrosion resistance can be further enhanced by increasing superheat degree.

11:20 AM

Quantifying Casting Processing Variability Due to Ingot Source: Primary vs. Secondary: *Darius Singh*[1]; Ian Paine[2]; [1]AUT University; [2]Glucina Alloys Ltd

A fundamental issue of processing variability still exists in industry in terms of casting products using ingots that have been produced from either primary, secondary, or directly internally recycled. Downstream problems of oxide and other non-metallic inclusion borne defects are usually attributed to a myriad of visible and directly controllable processing parameters, however the alloy source, its manufacturing technique, quality rating and testing is often overlooked or taken for granted as a "workable" baseline to operate from. This work investigates the quality of CC601/A356 ingots produced from a primary smelter vs. a secondary producer. Best practices in manufacturing each ingot are described from each plant and measurements of melt cleanliness are made using the following foundry techniques: Prefil Footprinter, Spiral mold, Tensile, K mold, plus a modified K+ mold technique. The sensitivities of each testing method are discussed and differences between the ingot quality are revealed.

11:40 AM

Grain Refiner Characteristics of a Novel Chemical Grain Refiner for Al Alloys: *Hari babu Nadendla*[1]; M Nowak[1]; [1]Brunel University

We have recently developed an effective chemical grain refiner for Al alloys. Addition of this grain refiner to commercial pure Al and Al-Si alloy is observed to decrease the undercooling of the melt. As a result, fine grain structure is obtained for Al alloys cast at various cooling rates. Microstructural investigations of newly developed master alloy reveals presence of fine sized second phases. Their chemical composition has been investigated using high

resolution electron microscopy. It is found that there is a good lattice match between the second phase and Al, suggesting that these particles enhances heterogeneous nucleation sites in the Al melt. The paper also discusses optimisation of chemical composition of newly grain refiner, microstructures of various casting alloys and their mechanical properties.

12:00 PM

Modern Trends and Methods for Debottlenecking Primary Aluminum Smelters: *Joe Petrolito*[1]; [1]Hatch

At the advent of the global financial crisis, aluminum smelters around the world are faced with similar challenges: generate more revenue with minimum capital investments. Accordingly, smelters are paying close attention to their existing operations in order to increase production via amperage creep to improve the bottom line. However, this cannot be accomplished solely by operations, finance or engineering working in isolation. Determining the best practical approach which delivers the highest returns with the least technical risk requires the continuous collaboration of all three parties. This paper will examine the interface between these parties: what are the recent trends in terms of debottlenecking projects and what methods are being used to ensure that both capex and opex are properly analyzed to ensure the operational, financial and technical success of the project.

12:20 PM

Preparation of Al-Mg Alloys from MgO in KCl-MgF2-LiF Electrolyte by Molten Salt Electrolysis Method: Fengli Yang[1]; *Sh Yang*[1]; Xianwei Hu[2]; Zhaowei Wang[2]; Zhongning Shi[2]; Bingliang Gao[2]; [1]Jiangxi University of Science and Technology; [2]School of Materials and Metallurgy117#, Northeastern University

Aluminum-magnesium alloys were prepared from magnesium oxide by molten salt electrolysis method. 30w%KCl-40w%MgF2-30w%LiF was taken as electrolysis. Effect of temperature on electrolysis was great. Back electromotor force(BEMF) was reduced 0.2-0.4V with electrolytic temperature from 800° to 840°. The process of electrolysis was controlled together by electrochemical polarization and concentration polarization. Content of magnesium in alloys was 6-8w%, and to be controlled by electrolytic time. Prepared aluminum-magnesium alloys were even. The results showed it was feasible to prepare aluminum-magnesium alloys from magnesium oxide in KCl-MgF2-LiF electrolyte by molten salt electrolysis method.

Hume-Rothery Symposium Thermodynamics and Diffusion Coupling in Alloys - Application Driven Science: Thermodynamics and Diffusion

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

Program Organizers: Zi-Kui Liu, The Pennsylvania State University; Larry Kaufman, CALPHAD, Inc.; Annika Borgenstam, Royal Institute of Technology; Carelyn Campbell, NIST

Monday AM — Room: 31A
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Gary Purdy, McMaster University; Staffan Hertzman, Outokumpu Stainless Research Foundation

8:30 AM Introductory Comments

8:40 AM Keynote

WILLIAM HUME-ROTHERY AWARD LECTURE: Thermodynamics and Diffusion Coupling in Alloys - Application Driven Science: *John Agren*[1]; [1]Royal Institute of Technology

As emphasized by Stokes (1997) the common assumption of a linear progression from basic research (science), via applied research, to technological innovations (engineering), should be questioned. In fact society would gain much by supporting long-term research that stems from practical problems and has usefulness as a key word. Such research may be quite fundamental and often it cannot be distinguished from "basic" research if it were not for its different motivation. The development of the Calphad method and the more recent development of accompanying kinetic approaches for diffusion, serve as excellent examples and are the themes of this symposium. The drivers are, e.g., development of new materials, processes, and life-time predictions. Many challenges of utmost practical importance require long-term fundamental research. This presentation will address some of them, e.g. effect of various ordering phenomena on activation barriers, the strength and practical importance of correlation effects, etc.

9:20 AM Invited

Application of Thermodynamic Equilibria and Kinetic Calculations to Phase Transformations in High Performance Stainless Steels: *Staffan Hertzman*[1]; [1]Outokumpu Stainless Research Foundation

In the development and processing of modern stainless steels the phase evolution during thermal cycles plays a crucial role since the microstructure strongly influences the material performance. In order to facilitate the understanding and control of the transformations, computational thermodynamics is a prerequisite. Alloy development, weldability, processability and service performance require however different approaches and examples are given to illustrate the importance of relevant thermodynamic and kinetic modelling tools.

9:50 AM Invited

The Implications of Thermodynamic Models on Diffusion Simulations: *Ursula Kattner*[1]; Carelyn Campbell[1]; [1]National Institute of Standards and Technology

The importance of appropriate models for phase descriptions in thermodynamic modeling of systems employing the Calphad method is well known. Models should take into account crystal structure and preferred site occupations on deviation from stoichiometry in multicomponent systems and reflect whether an order/disorder transformation is present. Models must also use a minimum number of independent parameters to describe the thermochemistry of phases. These requirements necessitate that compromises be made in the description of many phases, resulting in so-called simplified model descriptions. Improper simplification of a phase may not necessarily manifest itself during the thermodynamic calculations, but may become present during diffusion simulations. For instance, describing order/disorder phases with an order/disorder model or different sublattice models may not result in noticeable differences in the calculated phase diagram but may produce unreliable results if thermodynamic and diffusion mobility descriptions do not agree. Examples from calculations of superalloys and photovoltaics will be presented.

10:20 AM Break

10:40 AM Invited

Modeling of Diffusion-Controlled Phase Transformations in Steel: *Joakim Odqvist*[1]; [1]Royal Institute of Technology (KTH)

The local equilibrium hypothesis is usually adopted when modeling diffusion-controlled phase transformations. However, for finite interface velocities there will be a deviation from local equilibrium at the moving phase interface. Different approaches to model this deviation for the austenite-ferrite transformation are reviewed and compared with recent experimental findings. Furthermore, Larsson's model for modeling diffusion in the lattice-fixed frame of reference is modified to account for a cooperative mechanism for the interface migration.

11:10 AM Invited

A Computationally Based Approach to Homogenizing Advanced Alloys: *Paul Jablonski*[1]; Christopher Cowen[1]; [1]US Department of Energy

We have developed a computationally based approach to optimizing the homogenization heat treatment of complex alloys. The Scheil module within the Thermo-Calc software is used to predict the as-cast segregation present within alloys, and DICTRA (Diffusion Controlled TRAnsformations) is used to model the homogenization kinetics as a function of time, temperature and microstructural scale. We will discuss this approach as it is applied to both Ni based superalloys as well as the more complex (computationally)

case of alloys that solidify with more than one matrix phase as a result of segregation. Such is the case typically observed in martensitic steels. With these alloys it is doubly important to homogenize them correctly, especially at the laboratory scale, since they are austenitic at high temperature and thus constituent elements will diffuse slowly. The computationally designed heat treatment and the subsequent verification real castings are presented.

11:40 AM Invited
Kinetic Transitions in the Growth of Ferrite during the Decarburization of Alloyed Austenite: *Gary Purdy*[1]; Hatem Zurob[1]; Damon Panahi[1]; Christopher Hutchinson[2]; Yves Brechet[3]; [1]McMaster University; [2]Monash University; [3]Instutute National Polytechnique de Grenoble

The growth of ferrite from alloyed austenite under conditions of controlled decarburization has proven a rich area for study, permitting the inference of the contact conditions for carbon at a migrating planar interface. In this contribution, we summarize recent findings with emphasis on transitions between full local equilibrium in the absence of alloying element partitioning (LE-NP) and the constrained local equilibrium state, paraequilibrium (PE). We have utilized Agren's evaluations of the diffusion coefficients of carbon in ferrite and austenite, first to validate the binary (Fe-C) results, then for the evaluation of the interfacial carbon concentrations for several cases of ternary iron alloys (Fe-C-X, where X is a substitutional solute). The implications of these observations are discussed in terms of new models for the migrating interfaces.

Hydrogen Storage in Materials: Theory and Experiment: Session I

Sponsored by: The Minerals, Metals and Materials Society, ASM Materials Science Critical Technology Sector, TMS: Energy Conversion and Storage Committee
Program Organizer: Louis Hector Jr, GM R&D Center

Monday AM
February 28, 2011
Room: 13
Location: San Diego Conv. Ctr

Session Chair: Eric Majzoub, University of Missouri - St. Louis

8:30 AM
A Consistent Thermodynamic Description of Hydrogen Diffusion and Defect Interaction in a Material Containing Dislocations, Voids and Micro-cracks: *Kiran Solanki*[1]; Douglas Bammann[1]; [1]Mississippi State University

A finite deformation, rate and temperature dependent model of inelasticity is developed within the thermodynamic framework of Gurtin to describe the hydrogen effect on material deformation and material deformation on hydrogen transport. Internal state variables associated with statistically stored dislocations, the concentration of hydrogen and associated gradients and a consistent damage-elastic strain metric are introduced into the Helmholtz free energy, in addition to the standard variable of temperature. The theory is developed for large strains, whereby the total deformation gradient is multiplicatively decomposed into contributions from elasticity, damage, plasticity and hydrogen effects. Thermodynamic restrictions from the dissipation inequality result in the restriction that the stress and microforces are defined as derivatives of the free energy with respect to elastic-damage metric, and H concentration and its gradient, respectively. Furthermore, a general form for the chemical potential is constructed, based upon thermodynamic restrictions. Simplified models are examined and compared with experimental examples.

8:50 AM Invited
Formation of Hydrogen Cottrell Atmosphere in Palladium: Theory and Measurement from Inelastic Neutron Scattering: *Dallas Trinkle*[1]; Hyunsu Ju[1]; Brent Heuser[1]; [1]University of Illinois, Urbana-Champaign

Palladium has a high hydrogen solubility and diffusivity. H can be stored both in octahedral sites and in dislocation cores, which act as nanoscale H traps--forming Cottrell atmospheres that are metal-hydride-like at low temperatures. The formation of a Cottrell atmosphere can be measured in situ with inelastic neutron scattering. Ab initio density-functional theory computes the potential energy for a hydrogen in the core of a partial dislocation and with volumetric strains. We model changes in the hydrogen potential from neighboring Pd vibrations to predict the vibrational density of states for hydrogen from 0K to room temperature. The predicted inelastic neutron scattering intensity compare with new measurements, which show a shoulder at 0K from the core and hydride formation, widening of the peak at 200K from spreading of the Cottrell atmosphere, and a shift in the peak at 300K as the atmosphere dissolves

9:30 AM
Effects of Gaseous Impurities in Hydrogen on the Long Term Cycling Stability and Storage Capacity of Li3N: *Joshua Lamb*[1]; Dhanesh Chandra[1]; Wen-Ming Chien[1]; Delphine Phanon[2]; Nicolas Penin[2]; Radovan Černý[2]; Klaus Yvon[2]; [1]University of Nevada, Reno; [2]University of Geneva

We have studied the effect of gaseous impurities in hydrogen on Li-N-H. The gaseous contamination effects were studied using 100 ppm levels of impurity gases such as O_2, H_2O, CH_4, CO and NH_3 mixed with UHP hydrogen. In addition, commercial-grade industrial hydrogen was also used. In the case of industrial hydrogen we found a ~50% loss (~2.6wt% out of ~5.6wt%H_2) after 1100 pressure cycles. X-ray diffraction studies revealed formation of Li_2O. The addition of H_2O, CH_4, NH_3, and CO showed varying degrees capacity loss. We observed significant improvement in the reversible capacity up to ~10wt.%H after 516 cycles with 80/20 H_2/N_2 mixtures, an improvement of ~7.1wt.H as compared to using hydrogen without nitrogen additives. We attribute this enhancement to the reaction of nitrogen with liquid lithium during cycling as the Gibbs energies of formation of Li_3N (ΔG_o=-100.16 kJ/mol) are more negative than that of LiH phase (ΔG_o=-50.74 kJ/mol).

9:50 AM
Analysis of Deformation Twins and the Partially Dehydrogenated Microstructure in Nanocrystalline Magnesium Hydride (MgH2): *David Mitlin*[1]; Mohsen Danaie[1]; Shu Xia Tao[2]; Peter Kalisvaart[1]; [1]University of Alberta and NINT NRC; [2]Eindhoven University of Technology

We employed cryo-stage transmission electron microscopy (TEM), supported by density functional theory (DFT), to explore the microstructure of magnesium hydride (MgH2) powders. Mechanical milling results in deformation twinning of the hydride. The crystallography of the twins is established. DFT analysis shows that the twin unit cell is just as thermodynamically stable as the un-deformed a-MgH2 matrix. We hypothesize that the twins contribute significantly to the observed milling-induced kinetic enhancement, by acting as high diffusivity paths for hydrogen. Energy-Filtered TEM (EFTEM) analysis on partially desorbed MgH2 demonstrates that nucleation and growth of metallic magnesium occurs non-uniformly. Larger powder particles are a composite of isolated magnesium grains heterogeneously nucleated on the remaining hydride. Smaller particles are either fully transformed to magnesium or remain entirely a hydride. There is little evidence for any "core-shell" structure. We also show that in-situ hydrogen desorption in the TEM is not representative of the elevated-temperature ex-situ sequence.

10:10 AM Break

10:30 AM
Mechanical Properties and Hydrogen Sorption in Mg/Nb Multilayer Films: Byoungsoo Ham[1]; *Xinghang Zhang*[1]; [1]Texas A&M University

Mg/Nb multilayers with layer thickness varying from a few to 200 nm were prepared by the magnetron sputtering technique. The as-deposited films have Mg (0002) and Nb (110) textures. The hardness of films is greater at smaller individual layer thickness, and approaches a maximum when h is a few nm. The strengthening mechanisms in the multilayer are discussed. Hydrogen sorption studies were performed in some of the sputtered Mg/Nb multilayers. Sputtered multilayer films were loaded in hydrogen for 24 hr

at elevated temperature. The hydrogen desorption characteristic of textured Mg/Nb multilayer films will be reported.

10:50 AM

Mg-Based Nanocomposites for Room Temperature Hydrogen Storage: *Mieczyslaw Jurczyk*[1]; Marek Nowak[1]; Leslaw Smardz[2]; Andrzej Szajek[2]; [1]Poznan University of Technology; [2]Polish Academy of Sciences

In the last decade a great interest has been observed in the field of nanoscale materials. In this work, we study experimentally the electrochemical and electronic properties of Mg2M/A (M = Ni, Cu; A= C, Pd) and Mg2Ni/LaNi5/A nanocomposite hydrides. The nanocomposites were prepared by mechanical alloying followed by annealing. Results showed that the XPS valence bands measured for MA nanocrystalline alloys and composites showed a significant broadening compared to those obtained for microcrystalline materials. Furthermore, the surface segregation process of La (Mg) atoms in LaNi5/A (Mg2M/A) nanocomposites is stronger compared to that observed for the MA nanocrystalline LaNi5 (Mg2M) alloys. Additionally, the nanocomposite structure reduced hydriding temperature and enhanced hydrogen storage capacity of Mg-based materials. The nanocomposite Mg2Ni (50 wt%)-LaNi5 (50 wt%) material releases 1.6 wt% hydrogen at 25oC. The strong modifications of the electronic structure and surface segregation effect in the nanocomposites could significantly influence their hydrogenation properties.

11:10 AM

Desorption Kinetics of the Alkali Hexahydride Alanates (M2M'AlH6) at Constant Pressure Thermodynamic Driving Forces: *Hongwei Yang*[1]; Andrew Goudy[1]; [1]Delaware State University

A study was done to compare the desorption kinetics of the first decomposition step in a series of the hexahydride alanates (Na3AlH6, Na2LiAlH6 and K2NaAlH6). This comparison was made using a novel procedure in which the ratio of the equilibrium plateau pressure (Pm) to the opposing pressure (Pop), or the N-value, was the same. This represents the first time that such a comparison has been made in a complex hydride displaying two decomposition steps. Since the Gibbs free energy change is proportional to Ln(Pm/Pop), it was concluded that these experiments were carried out under constant thermodynamic driving forces. The results demonstrate that appropriate cation substitutions can be adopted to stabilize or destabilize the complex hydride. Modeling studies reveal the mechanisms controlling the kinetics for each alanate.

11:30 AM

Ammonia Borane at High Pressures: *Jiuhua Chen*[1]; Helene Couvy[1]; Vadym Drozd[1]; Haozhe Liu[2]; Yongzhou Sun[1]; Shah Najiba[1]; [1]Florida International University; [2]Harbin Institute of Technology

In situ synchrotron x-ray diffraction and Raman spectroscopy experiments of ammonia borane were performed at high pressures up to 23 GPa and elevated temperatures (300K – 505K). At ambient temperature, one first-order phase transition (I4mm to Cmc21) was observed at 1.3 GPa and one second-order phase transition were observed at about 5 GPa. The experiments reveal a bulk modulus of K = 9.3 ± 0.4 GPa (K'= 4.8) for the I4mm phase, K = 11.9 ± 0.5 GPa (K' = 4.6) for the Cmc21 phase below 5 GPa, and K = 37 ± 4 GPa (K'= 4.6) for pressure above 5 GPa. There is a 6% volume drop at the first order phase transition. Transition pressures from I4mm to Cmc21 phases were determined at elevated temperatures. The phase boundary has a negative claperon slope of -1.67MPa/K. Rehydrogenation of decomposed ammonia borane was observed above 6 GPa.

11:50 AM

Hydrogen Interactions with Li_3N and Formation of Intermediate Complex Hydride Phases: *Joshua Lamb*[1]; Anjali Talekar[1]; Wen-Ming Chien[1]; Dhanesh Chandra[1]; Delphine Phanon[2]; Nicolas Penin[2]; Radovan Černý[2]; Klaus Yvon[2]; [1]University of Nevada, Reno; [2]University of Geneva

Complex of Lithium nitride are important for hydrogen storage. In this study we found evidence of solid solution phases $Li_{2-x}NH_{1+x}$, particularly at x=1/2 ($Li_{1.5}NH_{1.5}$) during the progression of hydrogenation of Li_2NH. In-situ x-ray diffraction data shows the progression of Li_2NH to $Li_{1.5}NH_{1.5}$ to $LiNH_2$ transitioning through single and two phase regions as the concentration of hydrogen increases. In-situ x-ray diffraction, thermodynamic data, such as heat capacities of Li_3N and Li-N-H complex hydrides, differential scanning calorimetric and thermogravimetric data will be presented.

12:10 PM

Aluminoboranes and Boron Compounds for Hydrogen Storage: *Ji-Cheng Zhao*[1]; Xuenian Chen[1]; Zhenguo Huang[1]; Teshome Yisgedu[1]; Hima Lingman[1]; Beau Billet[1]; Sheldon Shore[1]; [1]The Ohio State University

Boron-containing compounds have been studied extensively around the world in recent years for hydrogen storage due to their high gravimetric hydrogen density. We are investigating many boron hydrides including aluminoboranes and boron-cage compounds in addition to borohydrides. The materials we are synthesizing and studying include AlB4H11, (NH4)2B10H10, and (NH4)2B12H12. Results we obtained on their syntheses, structures and desorption processes will be presented.

ICME: Overcoming Barriers and Streamlining the Transition of Advanced Technologies to Engineering Practice -- The 12th MPMD Global Innovations Symposium: Plenary Session and the Integration of ICME

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division
Program Organizers: Paul Mason, Thermo-Calc Software Inc; Mei Li, Ford Motor Company; James Warren, National Institute of Standards and Technology; Jeff Simmons, AFRL

Monday AM
February 28, 2011

Room: 7A
Location: San Diego Conv. Ctr

Session Chair: Paul Mason, Thermo-Calc Software

8:30 AM Plenary

Integrated Computational Materials Engineering: Current Status and Future Challenges and Opportunities: *John Allison*[1]; [1]Department of Materials Science and Engineering, The University of Michigan

ICME is now a recognized field of study within some sectors of the materials community and industry. ICME has lead to substantial increases in the efficiency with which new components are developed and promises to improve the efficiency and robustness of the development of new materials. When it is fully embraced as a discipline within the materials profession, ICME has the potential to revolutionize the way the materials community provides input to the engineering and scientific communities. This talk will attempt to define the current status of the field by drawing from a National Academies report on ICME and reflecting on what has transpired since that report. It will also draw from the presenter's prior experience at Ford Motor Company in development and implementation of a successful ICME tools. The author will attempt to identify future challenges and opportunities for realization of the full potential offered by ICME.

9:05 AM Plenary

Recent Progress of Multi-Scale Modeling of Solidification Process of Shape Casting: *Baicheng Liu*[1]; Tao Jing[1]; Qinyan Xu[1]; Zhiqiang Han[1]; [1]Tsinghua University

The development of manufacturing industries requires an advanced and comprehensive system which integrates and optimizes the product design and manufacturing process and makes the product development efficient, economic and environment friendly. Integrated computational materials engineering (ICME) could be considered as a promising approach to fulfill this requirement. This paper presents recent progress of ICME, especially multi-scale modeling of solidification process of shape casting in China, including mathematical models and engineering application as well. Some case studies, including microstructure simulation of unidirectionally solidified turbine blade castings for aviation industry, microstructure simulation of aluminum and magnesium alloy castings based on cellular

automaton and phase field methods for automobile industry and simulation of casting and heat-treatment processes of turbine blade casting of water-turbine are discussed.

9:40 AM Plenary
Microstructure-Based Descriptions of the Deformation of Metals: *Peter Gumbsch*[1]; Daniel Weygand[2]; Stefan Sandfeld[2]; Dirk Helm[1]; Alexander Butz[1]; [1]Fraunhofer IWM; [2]izbs, KIT

Metal forming simulations today are based on classical constitutive descriptions of yield behavior and hardening. Microstructural materials characteristics like texture, grain size or dislocation microstructure are rarely considered and never systematically evolved. This is due to a lack of available methodology. There is therefore a need for mechanistic and physically-consistent descriptions of plastic deformation on all levels of structural complexity. I will address three levels. First, single crystalline microcomponents are assessed using discrete dislocation dynamics simulations, which are inherently able to represent size effects. Second, progress in the development of a dislocation density based continuum theory of plasticity is demonstrated. Third, advanced homogenization techniques are used to bridge from single crystal plasticity to the deformation behavior of polycrystals. In a "virtual lab" one thereby computes texture evolution and associated changes in the yield locus. Such computed data can then be used to adjust the parameters of classical plasticity models.

10:15 AM Break

10:30 AM Invited
The Development of the ICME Supply-Chain: Route to ICME Implementation and Sustainment: *David Furrer*[1]; [1]Rolls-Royce

Integrated computational materials engineering (ICME) has been an evolving field for many years. Models which simulate materials-related phenomena have been developed and are being validated for industrial application. The integrating computational methods into material, process and component design has been a challenge, however, in part due to the development of a supply-chain that supports this emerging technology area. ICME touches many disciplines, which results in a requirement for many levels of computational-based technology organizations to be involved to provide tools that can be rapidly developed, validated, deployed and maintained for industrial applications. This talk will assess the need and current state of ICME supply-chain development and future requirements for the continued pace of introduction of ICME into industrial design practices.

10:55 AM Invited
On the Competitive and Pre-Competitive Aspects of ICME in New Technology Insertion - A Material Supplier Perspective: *Robert Bucci*[1]; Mark James[1]; Markus Heinimann[1]; Michael Kulak[1]; [1]Alcoa Technical Center

Analytical consensus between supplier and OEM is key to new product acceptance - particularly so when design/certification path changes are needed to capture full advantage of promising approaches. The underpinnings of ICME facilitate new technology insertion with benefits to all. This in turn enables competitive advantage via product differentiation and knowledge transfer. This presentation describes how Alcoa is deploying ICME and related best practices to accelerate new product development. Past examples of material solutions that did and did not transition are reviewed with key lessons learned. The presentation main focus is on Alcoa's current application of ICME to: a) Bulk residual stress management in large single-piece airframe components machined from Alcoa Signature Stress Relief™ aluminum die-forgings; b) Mature advanced hybrid structural concepts utilizing optimized combinations of aluminum alloys and fiber metal laminates. Finally, pre-competitive and competitive aspects of ICME are discussed in context of the preceding.

11:20 AM Invited
Air Force Adoption of ICME for Materials and Manufacturing R&D: *Katherine Stevens*[1]; Chuck Ward[2]; [1]Air Force Research Laboratory; [2]US Air Force

Numerous challenges and opportunities face the Air Force with regard to materials and manufacturing R&D. An aging Air Force aircraft fleet, a smaller defense industrial base, fewer major weapon systems produced, and a need for a more rapid and agile development cycle are all current challenges. Advances in physical understanding of materials and computational capability, when combined with a holistic systems engineering approach toward design of and with materials, provide a compelling case for adopting ICME as the necessary path to take for conducting materials and manufacturing R&D. The emerging ICME paradigm for materials engineering holds promise for reducing development time, cost, and risk. The transition to that paradigm presents needs for improved data structures, model verification and validation standards, and growth in quantitative descriptions of material behavior and manufacturing methods. Efforts by the Materials and Manufacturing Directorate to adopt ICME throughout its research and development program will be described.

11:45 AM
Cyberinfrastructure Support for Integrated Computational Material Engineering: *Tomasz Haupt*[1]; [1]Mississippi State University

The goal of this effort is to develop a cyberinfrastructure to exploit the recent transformative research in material science involving multiscale physics-based predictive modeling, multiscale experiments and design. The cyberinfrastructure comprises a collection of materials databases as well as model, model calibration, and simulation tools at multiple size scales. It streamlines the process of gathering experimental results and deriving material properties and linking processes and properties at different length scales enabling one to design and develop the next generation materials and structural components exploiting the integrative nature of ICME. Moreover, through an Open Source process, it serves as a platform for collaborative research and transitioning research tools and codes to more robust tools ready for industrial use. The cyberinfrastructure is implemented using the Web 2.0 technologies including Wiki, AJAX-based rich user interfaces, Service Oriented Architectures (SOA), Web Services and Grid computing.

12:05 PM Invited
Building Knowledge Systems for the Design and Processing of Materials with Improved Performance Characteristics: *Surya Kalidindi*[1]; Stephen Niezgoda[1]; Tony Fast[1]; Giacomo Landi[1]; [1]Drexel University

There exists a critical need to couple simulations over several length scales to predict macroscale performance characteristics, which tantamounts to executing complex numerical models within other sophisticated numerical models in a hierarchical manner. High performance data-mining tools are critically needed for harvesting efficiently the essential knowledge contained in the very large experimental and modeling datasets being produced by experts in the materials related fields. We have developed a novel framework called Materials Knowledge Systems that aims to establish highly accurate local interaction laws at each length scale by data-mining results from numerical models. Once established these local influence laws can be exercised with minimal computational effort as part of a scale-bridging framework for multi-scale materials modeling and design problems.

Intelligent Materials and Structural Health Monitoring: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, ASM Materials Science Critical Technology Sector, TMS/ASM: Composite Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Corrosion and Environmental Effects Committee, METSOC-CIM: Metal Processing and Fabrication Committee
Program Organizer: Subu Nayak, ScienceTomorrow

Monday AM
February 28, 2011

Room: 33C
Location: San Diego Conv. Ctr

Session Chair: To Be Announced

8:30 AM
Robustness of Ultrasonic Nonlinearity Measurements: *Aurora Zinck*[1]; Krishnan Balasubramanian[2]; Sridhar Krishnaswamy[1]; [1]Northwestern University; [2]IIT Madras

Ultrasonic nonlinearity has been shown to be highly sensitive to microstructural changes compared to conventional ultrasonic techniques for structural health monitoring. Changes in the ultrasonic nonlinearity parameter, \946, have been qualitatively linked to changes in dislocation structures of mechanically loaded metals. In this work the limitations of current experimental techniques for measuring the ultrasonic nonlinearity by second harmonic generation are studied. Polycrystalline copper samples are monotonically loaded and the varied nonlinearities are measured by longitudinal waves traversing through the samples. The effect of experimental variables is explored though the use of multiple laboratory setups and procedures are outlined for optimized experimentation. Finally, the non-destructive measurements are compared to corresponding destructive measurements for an evaluation of the change in \946 with respect to plastic deformation. These results suggest a comparison of ultrasonic nonlinearity testing methods that could be applicable for future use of ultrasonic nonlinearity as a structural health monitoring tool.

8:55 AM
Impact Monitoring for Composite and Ceramic: *Jyoti Agrawal*[1]; Subu Nayak[1]; [1]ScienceTomorrow

A structural health monitoring technology for composite and ceramic structures based on differential sensors is developed. While vibration-based frequency response function (FRF) used for structural health monitoring, the threshold of damage detection is too high and in case of an externally imposed vibration (such as that of an engine in an aircraft). The differential sensor in ScienceTomorrow's technology eliminate the effect of temperature and external vibration. Differential strain gauges amplify signal and reduce noise, for example, by subtracting a compressive stain signal from a tensile signal. A neural network algorithm calibrates the signal against temperature, external signal and noise. Tunable amplitude modulation use the externally imposed vibration as the carrier vibration and detect any transient change in vibration such as an impact loading. The SHM system record impact loading (the composite and ceramics are most susceptible to) and correlate any damages as indicated by shift in natural frequency.

9:20 AM
Nanocomposite Sensing Skins for Damage Identification and Localization: Kenneth Loh[1]; *Bryan Loyola*[1]; [1]University of California, Davis

Structural systems are susceptible to damage resulting from yielding, cracking, impact, and corrosion during operation or service. In this study, structural damage identification and localization is performed via layer-by-layer assembled carbon nanotube-polyelectrolyte "sensing skins." Previous studies have verified that these conformable films exhibit highly sensitive electromechanical and electrochemical responses to applied strain and corrosion processes, respectively. Instead of using these thin films as point sensors, they are coupled with an electrical impedance tomographic (EIT) spatial conductivity mapping technique. EIT relies on boundary electrical measurements to reconstruct the spatial conductivity map of the nanocomposite. Since electrical conductivity is calibrated to applied external stimuli, the conductivity maps directly correspond to spatial damage maps; one can then use the maps to precisely identify damage location and severity. A series of experiments are conducted to characterize the damage identification and localization performance of the sensing skins, namely non-uniform strains, impact, and corrosion.

9:45 AM
Recent Progress on Processing of Amorphous Coatings: *Sandip Harimkar*[1]; [1]Oklahoma State University

Amorphous materials or bulk metallic glasses represent a new class of advanced materials exhibiting attractive combinations of properties such high strength/hardness and excellent wear/corrosion resistance. Even though the rapid solidification (casting) methods for processing amorphous alloys are well established, the need for simultaneous mold filling and rapid cooling rate limits the range of geometries that can be formed. These processing difficulties in combination with low tensile ductility and toughness are likely to limit the applications of amorphous materials as bulk structural materials. However, the amorphous materials can be good candidates for wear/corrosion resistant coatings on the crystalline substrates. In this presentation, an overview of the newest processing approaches, including thermal spraying, cold spraying, electrodeposition, and laser surface treatments (surface amorphization and cladding) for the fabrication of amorphous coatings will be highlighted. Also, the results of our recent research on spark plasma sintering of iron-based amorphous coatings will be presented.

10:10 AM
Thermography Detection of Both Crystalline and Amorphous Materials during Cyclic Loading: *P. Liaw*[1]; Gongyao Wang[1]; B. Yang[2]; L. Jiang[3]; Y. Yokoyama[4]; A. Inoue[4]; [1]University of Tennessee; [2]Shell Company; [3]General Electric Global Research Center; [4]Institute for Materials Research

It is of critical importance to find new ways to in-situ monitor the structural material fatigue damage so that in-time reparations will be possible, and failures or losses can be controlled. Usually temperature patterns serve as fingerprints during loading. In our lab, a state-of-art infrared (IR) thermography camera is employed to monitor the temperature evolutions of both crystalline and amorphous materials during fatigue experiments. With the understanding of the temperature evolutions during fatigue, thermography could provide the direct information and evidence of the stress-strain distribution, crack initiation and propagation, shear-band formation and growth, and plastic-zone evolution, which will open up wide applications in studying the structural integrity of engineering components in service. In-situ visualizations as well as qualitative and quantitative analyses of fatigue-damage processes have been performed using thermography results. Theoretical models combining thermodynamics and heat-conduction theory are developed to understand the fatigue behavior of both crystalline and amorphous alloys.

10:35 AM
Differential Signal vs. Differential Sensor in Structural Health Monitoring: *Subu Nayak*[1]; [1]ScienceTomorrow

Structural health monitoring circuit design are typically based on differential signaling. While noise and distortion are reduced in differential signal, the resolution is limited by the sensitivity of the sensor. Also, temporal and spatial information is not captured or processed. In this work, a novel differential sensor approach being proposed. In lieu of or in addition to differential signal circuit, differential pair of sensors being used to improve structural health monitoring. For example, in a mechanical system sensors strategically located to capture tensile as well as compressive stress are most effective in determining stress and health of structures. In corrosion, sensors monitoring local anodes and cathodes work as differential sensor. In dynamic systems, additional information in form of time stamping can provide temporal resolution for more accurate monitoring such as an impact

loading. This new approach is also fault tolerant when used in a distributed sensor network.

Magnesium Technology 2011: Opening Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Wim Sillekens, TNO Science and Industry; Sean Agnew, University of Virginia; Suveen Mathaudhu, US Army Research Laboratory; Neale Neelameggham, US Magnesium LLC

Monday AM
February 28, 2011
Room: 6F
Location: San Diego Conv. Ctr

Session Chairs: Wim Sillekens, TNO Science and Industry; Suveen Mathaudhu, US Army Research Office

8:30 AM Opening of the Symposium and Presentation of the Best Paper Awards 2010

8:50 AM Keynote
Magnesium in North America: A Changing Landscape: *Susan Slade*[1]; [1]US Magnesium LLC

The changing landscape of North American manufacturing in the context of global competition is impacting the market of all raw materials, including magnesium. Current automotive fuel economy legislation and pending legislation on the emissions of greenhouse gases are impacting magnesium's largest consuming industries, such as aluminum, automotive components, steel and transition metals. These industries are all considering innovative ways to efficiently incorporate the needed raw materials into their processes. The North American magnesium market differs from other regions based on maturity, supply streams, changing manufacturing capabilities and trade cases, combined with the transformation of North American manufacturing. The impact of these factors on the supply/demand dynamics of the North American magnesium market in both the short and long-term will be reviewed. The influence of new applications, products, and legislative changes are considered in the equation.

9:20 AM Keynote
Global Magnesium Research: State-of-the-Art and What's Next?: *Karl Kainer*[1]; [1]GKSS Research Centre Geesthacht

In recent years magnesium and its alloys have been successfully introduced into weight-saving applications in the transportation industries in order to reduce fuel consumption and greenhouse gas emissions as well as to increase the performance of modern cars. Besides advantages, e.g. superior specific strength and excellent processability, applications of magnesium alloys are limited due to their inferior properties at elevated temperatures, e.g. low creep resistance and reduced corrosion behavior, especially when in galvanic contact with other metallic materials. Current developments are revealing possibilities to improve these properties by using modern alloys and processing routes. While the majority of industrial applications utilize cast products, the use of wrought magnesium alloys is still at an early stage. Within the framework of ongoing research and development, the corrosion behavior of both cast and wrought magnesium materials in standalone uses or in galvanic couples with other metallic materials is gaining increasing attention. New coating systems tailored to selected applications will have to be developed in order to increase the usage of magnesium alloys in the transportation industries in the future. This work also needs to be coordinated with new processes for joining magnesium alloys with similar and dissimilar metals and alloys, to achieve a broad spectrum of materials that fulfill the requirements given by the applications. This presentation will first address these issues and challenges, then discuss new developments and finally show some examples of new applications. In the conclusions, gaps and challenges will be analyzed and recommendations for sustainable research and development will be given.

9:50 AM Keynote
Environmental Challenges for the Magnesium Industry: *Robert E. Brown*[1]; [1]Magnesium Assistance Group Inc.

The subject of environmental concerns with magnesium production and magnesium processing first started showing up in technical analysis of problems about the time of Life Cycle Analysis articles. Magnesium is produced and processed in relatively small quantities throughout the world. Annual magnesium production has been around 500-700,000 metric tons per year. This compares to aluminum which is produced in annual amounts up to 35 million metric tons. There have been some excellent review papers done, but a great amount of work related to electrolytic magnesium production which was the predominant method of production. That situation has changed totally in the past 10 years and now 85% of the world's magnesium is produced by thermal processes and most of that is in China. Comparison papers have been written comparing the environmental impacts of the two main magnesium production processes. As the measurement technology improves and as the total information references are better understood the environmental challenges can be more clearly identified. This paper reviews the situation and suggests some forward looking steps that might need to be taken.

10:20 AM Break

10:40 AM Keynote
Predicting Mg Strength from First-Principles: Solid-Solution Strengthening, Softening, and Cross-Slip: *Dallas Trinkle*[1]; Joseph Yasi[1]; Louis Hector[2]; [1]University of Illinois, Urbana-Champaign; [2]General Motors R&D Center

Predictive modeling of strength from first-principles electronic structure methods offers great promise to inform Mg alloy design. Simulating the mechanical behavior for new alloys requires an understanding of mechanisms for deformation at atomic-length scales, with accurate chemistry, extended to larger length- and time-scales. To design ductile Mg alloys, we identify solutes that strengthen basal slip and increase cross-slip. We use first-principles modeling of dislocations to predict dislocation motion under stress through a field of solutes at a finite temperature. First-principles flexible boundary conditions compute accurate core structures of basal and prismatic dislocations, and dislocation/solute interactions. We develop new models to predict the solute-strengthening for basal dislocations; cross-slip from basal- to prismatic-slip for a-type screw dislocations; and cross-slip stress with solutes. The first-principles data provides insight into the response of dislocations to solutes and the quantitative data to build new predictive models.

11:10 AM Keynote
Biodegradable Magnesium Implants - How do They Corrode In-Vivo?: *Frank Witte*[1]; Norbert Hort[2]; Frank Feyerabend[2]; [1]Hannover Medical School; [2]GKSS Research Centre

Biodegradable magnesium implants are currently breaking the paradim of designing and producing only corrosion resistant metallic biomaterials. The academic and industrial interest in this novel class of biomaterials is increasing dramatically in the recent years. First biodegradable metal implants have been realized as vascular stents and bone screws. However, the knowledge of the underlying degradation mechanism of these metal implants remains mainly undiscovered. This lecture will summarize the current published knowledge and recent advances in elucidating the in-vivo corrosion processes of these novel biodegradable magnesium implants.

11:40 AM Keynote
The Next Generation of Magnesium Based Material to Sustain the Intergovernmental Panel on Climate Change Policy: *Fabrizio D'Errico*[1]; Gerardo Garces[2]; Stefano Fare[1]; [1]Politecnico di Milano; [2]Consejo Superior de Investigaciones Científicas (CSIC)

Current Mg alloys have several drawbacks that limit wide and profitable utilization in the industrial sector. From an environmental point of view, lighter metals like magnesium are currently considered unclean products as they are energy-intensive. But they have been proven to be "clean" in

the transport sector, as they can reduce fuel consumption. Here the potential of magnesium based materials is addressed through double-tasking: a) establish innovative lean-manufacturing processes, avoid the classic melting step to substantially reduce carbon footprint of the magnesium products; b) encourage the using of no-melt processes, realizing high-resistant ultra-fined microstructures. In September the European Union started the "Green Metallurgy 2020" project through the LIFE+ 2009 Programme. The program, which is coordinated by the Politecnico di Milano (ITA), was started in September 2010. And by using the no-melting route CENIM (SPA) has achieved up to 400 MPa UTS and an elongation capability of about 13% for some ultrafine bi-phase Mg –Zn-Y.

12:10 PM Keynote
JIM INTERNATIONAL SCHOLAR AWARD WINNER: Fracture Mechanism and Toughness in Fine- and Coarse-Grained Magnesium Alloys: *Hidetoshi Somekawa*[1]; Alok Singh[1]; Toshiji Mukai[1]; [1]National Institute for Materials Science

The fracture mechanisms in the extruded magnesium alloys with two different grain sizes, 2 and 50 μm, were investigated by SEM, TEM and EBSD microstrctural observations. The coarse-grained alloy showed that the {10-12} type deformation twins formed at the beginning of test, and the crack was propagated into the boundaries between twins and matrix. On the other hand, the fine-grained alloy showed that the sub-grain boundaries formed instead of the deformation twins. No formation of twins at the early deformation stage causes a crack-tip blunting, and thus, the fracture toughness is a high value.

Massively Parallel Simulations of Materials Response: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: High Temperature Alloys Committee
Program Organizers: Diana Farkas, Virginia Tech; Susan Sinnott, University of Florida

Monday AM
February 28, 2011

Room: 1A
Location: San Diego Conv. Ctr

Session Chair: To Be Announced

8:30 AM Introductory Comments

8:40 AM Keynote
Mesoscale Molecular Dynamics with LAMMPS: *Steve Plimpton*[1]; [1]Sandia National Labs

I'll briefly describe our LAMMPS molecular dynamics (MD) code and highlight some of its design features that have made it easy for users to modify and extend. One area of recent development in the code has been for modeling solvated nanoparticle or colloidal systems. In an MD code this requires coarse-graining to achieve meaningful simulation times for the study of rheological and other manufacturing properties. This involves treating the nanoparticles as single point particles,moving from explicit to coarse-grained to implicit solvent, and capturing hydrodynamic effects. I'll describe efficient techniques we've developed for several models. The first is nanoparticles in a Lennard-Jones solvent, which involves multiple length scales. The second is the stochastic rotation dynamics (SRD) formalism, where solvent particles interact with nanoparticles via collisions but not with each other. Together these algorithms can enable speed-ups of several orders of magnitude, making solvated nanoparticle systems more accessible to modeling.

9:20 AM Invited
Implementation of Multi-level Parallelism in LAMMPS for Improved Scaling on PetaFLOP Supercomputers: *Axel Kohlmeyer*[1]; [1]Temple University

Understanding the properties of vesicles with diameters between a few tens and hundreds of nanometers has many applications from transport mechanism in cells to the formulation of consumer products. Mechanism that guide fusion of vesicles or stabilize them are to specific for continuum level theory and too costly for all-atom molecular dynamics simulations. Coarse grain potentials for MD can help to overcome this dilemma. Still, petaflop range supercomputers are required and scaling and throughput of LAMMPS need to be improved to make those kind of studies possible. This has motivated the implementation of a hybrid OpenMP/MPI parallelization strategy into many modules of LAMMPS that span nearly the full range of applications. We will discuss the results of benchmark calculations using a variety of test systems and supercomputers and demonstrate how to scale inputs with long-range electrostatic and tens of millions of particlesto over 100,000 processor cores.

9:45 AM Invited
Atoms-to-Continuum (AtC): A User Package for LAMMPS: *Jonathan Zimmerman*[1]; Reese Jones[1]; Jeremy Templeton[1]; Gregory Wagner[1]; [1]Sandia National Laboratories

To enhance the capabilities of LAMMPS and expand its usefulness in the modeling and simulation of nanostructures, we have developed the user package and associated "fix", atc (atoms-to-continuum). This multi-purpose toolset can perform coupled finite element-molecular dynamics simulations, as well as calculate on-the-fly estimates of continuum mechanical fields (e.g. stress, temperature, displacement gradient) based on atomistic simulation results. In this talk, we discuss objectives, capabilities, and use-details for this user package. We also present several examples of how it's been used to perform multi-scale analysis of phenomena in physics, materials science and applied mechanics. 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.

10:10 AM Break

10:25 AM Invited
Large-scale Excited Electron Molecular Mechanics/Dynamics: *Andres Jaramillo-Botero*[1]; Julius Su[1]; William Goddard[1]; [1]Caltech

To simulate large electronically excited systems, we have developed [at the Materials and Process Simulation Center at Caltech] a molecular dynamics (MD) method that considers electrons explicitly - called the Electron Force Field (eFF). In eFF, the electrons interact with the nuclei and each other via pairwise effective potentials, and their motions are propagated independently, making it possible to go beyond adiabatic dynamics. Unlike other fermionic MD methods, our approach achieves a balanced description of both ground-state condensed systems and highly excited systems containing ionized electrons. eFF is thus uniquely suited to simulate materials in extreme conditions, where many electronically excited states of matter can occur and coexist, and overcomes salient limitations of quantum mechanics methods, restricted to tens of atoms and sub-picosecond time scales. I will summarize recent progress in developing the method, as well as results using the extended capabilities of our parallel implementation under LAMMPS, code-named pEFF.

10:50 AM Invited
Interface Free Energy of CuNb Multilayers Using Massively Parallel Metropolis Monte Carlo Simulations: *Alfredo Caro*[1]; Enrique Martinez[1]; [1]LANL

Recent experimental discoveries and computational modeling of CuNb multilayers under irradiation suggest a particularly high resistance of these nanostructures to extreme environments. In this work we investigate the connection between interface stability under extremes and interface free energy. We use a recently developed algorithm (B. Sadigh et al. to be

published) called variance constrained semi-grand canonical Metropolis Monte Carlo, implemented into Lammps, to predict microstruture and free energy of several CuNb nanostructures.

11:15 AM Invited
Simulation of Nanofoams under Irradiation: *Eduardo Bringa*[1]; J. Rodriguez-Nieva[2]; J. Monk[3]; D. Farkas[3]; A. Caro[4]; R. Johnson[5]; [1]CONICET-Universidad Nacional de Cuyo; [2]Instituto Balseiro; [3]Virginia Polytechnic Institute and State University; [4]Los Alamos National Laboratory; [5]University of Virginia

High-porosity materials can be found in a number of situations, from reactor materials to grains in space. Using LAMMPS for molecular dynamics (MD) simulations, we analyze the case of high porosity nano-scale foams, where experimental techniques are difficult to use and interpret. We consider two irradiation scenarios: (a) irradiation with ions with energies in the range 1-25 keV, of interest for fusion and fission energy applications; (b) swift heavy ion irradiation, with energies up to 5 GeV, relevant for track formation and interstellar grain evolution. Irradiation effects have larger spatial extent than for compact, full-density solids, and include the production of point-defects and twins which change the foam mechanical properties. In addition, we analyze the swift-heavy ion induced sputtering of these nanofoams.

11:40 AM
A Two Temperature Model of Radiation Damage in α-Quartz: *Carolyn Phillips*[1]; Rudolph Magyar[2]; Paul Crozier[2]; [1]University of Michigan; [2]Sandia National Laboratories

Two temperature models are used to model the interaction between the electronic subsystem and the atomic subsystem during thermal transients such as radiation damage, laser heating, and cascade simulations. By coarse-graining the electronic subsystem, its impact on thermal transients can be modeled in longer and larger classical molecular dynamics simulations. Building on the LAMMPS code, we introduce a new version of an inhomogeneous finite reservoir two-temperature model applied to an insulator, α-quartz, to model heat deposition in a SiO_2 lattice. Our model of the SiO_2 electronic subsystem is based on quantum simulations of the electronic response in a SiO_2 repeat cell. The parameterization of the electronic subsystem has a significant impact on the degree of permanent amorphization of the lattice. We argue that the inclusion of a simple electronic subsystem substantially improves the realism of such radiation damage simulations.

12:00 PM
Parallel Molecular Dynamics for Radiation Damage Modeling in Structural Materials: *Christophe Domain*[1]; Charlotte Becquart[2]; Ghiath Monnet[1]; Dmitry Terentyev[3]; [1]EDF R&D; [2]UMET, UMR 8207; [3]SCK-CEN

To understand the microstructure evolution under irradiation, the defect formation mechanisms and interaction with the microstructure have to be investigated. Neutron or ion irradiation leads to displacement cascades, phenomena occurring at nanometer scale for few tens of picoseconds. The cascades are simulated using millions of atoms simulation boxes. The study of the dislocation interaction with irradiation defects requires also very large simulation boxes and long simulations to achieve the level of dislocation velocity comparable with the experimental one. Large parallel MD simulations are thus also required. Parallel MD simulations done using the DYMOKA code in Fe will be discussed for the modeling of pressure vessel steels. The importance of ab initio calculations will be underlined to assess the validity of the interatomic potentials used [Phil. Mag. 89 (2009) 3215]. The use of these results in multiscale modeling will be presented emphasizing their integration in upper scale models.

Materials and Society: Linking Science and Technology for Global Energy Solutions: Plenary Session

Sponsored by: The Minerals, Metals and Materials Society, TMS: Materials and Society Committee
Program Organizer: Christina Meskers, Umicore

Monday AM
February 28, 2011
Room: 11A
Location: San Diego Conv. Ctr

Session Chairs: Iver Anderson, Ames Laboratory; James Foley, Los Alamos National Laboratory

8:30 AM Plenary
A Plan for a Sustainable Future Using Wind, Water, and Sun: *Mark Z. Jacobson*[1]; [1]Stanford University

In this talk, I will lay out a plan for the world to supply 100% of its energy for all purposes from wind, water, and sunlight (WWS) within the next 20-40 years and how materials will play a role in this plan. The study starts by reviewing and ranking major proposed energy-related solutions to global warming, air pollution mortality, and energy security while considering other impacts of the proposed solutions, such as on water supply, land use, resource availability, reliability, wildlife, and catastrophic risk. It then evaluates a scenario for powering the world on the energy options determined to be the best while considering materials, transmission infrastructure, costs, and politics. The study concludes that powering the world with wind, water, and solar technologies, which are found to be the best when all factors are considered, is technically feasible but politically challenging.

8:55 AM Plenary
Metallurgical Considerations in the Photovoltaic Module: *Funsho Ojebuoboh*[1]; [1]First Solar, Inc.

The incipience of thin film photovoltaic as a commercial power generator has occurred even as the device continues to be developed. At the same time, the commercial deployment of photovoltaic module manufacturing and the deployment of photovoltaic power plants have together emerged as a business sector on its own. In particular, since its incipiency, CdTe PV has been on the cutting edge of a drive to achieve grid parity. Materials are invariably critical to all aspects of power generation and photovoltaic power generation is no exception. In fact, photovoltaic power generation is an increasingly active research and development thrust in Materials Engineering. One aspect of the thrust is investigating the sources, synthesis and characterization of input materials (raw materials) and the management of output materials, including spent process materials. In this presentation, we consider materials important to the production of the photovoltaic module; invariably, our focus is on thin film modules and particularly CdTe PV. In accord with the theme of the session, "Linking science and technology for global energy solutions," the presentation includes a discussion of a world powered by clean, affordable solar electricity and the materials challenges.

9:20 AM Plenary
Electrical Energy Storage for Renewable Integration and Grid Applications: Status, Challenges and Opportunities: *Zhenguo "Gary" Yang*[1]; [1]Pacific Northwest National Laboratory

Growing concerns over the environmental consequences of burning fossil fuels and their resource constrains, along with the increasing world energy consumption, have spurred great interests in renewable energy from sources such as wind and solar. However, the power from these intermittent sources is constantly varied, making quite challenging for its use and dispatch through the aging electrical grid. One effective way to smooth out the intermittency is to employ electrical energy storage. There are potential technologies for the applications, including batteries, super-capacitors, fly-wheels, compress air, pump hydro, etc. But these technologies either cannot meet the performance and cost matrices for broad market penetration or are simply limited by site selection or environmental constrains. As such there have been growing

interests and R&D activities in advancing the storage technologies. This paper offers an overview on varied technologies, in particular batteries, and discusses the status, challenges and research needs.

9:45 AM Plenary
The Rare Earth Contributions to Global Energy Solutions: *Karl Gschneidner*[1]; [1]Iowa State University

The rare earths have many unique physical and chemical properties which make them important, if not critical, components in a variety of energy technologies. In the transportation sector: La is used in batteries; Ce in gasoline cracking catalysts and in three-way catalytic converters; Nd in the electric motors; Y as an oxygen sensor to control lean/rich fuel mixtures and as an oxidation resistant coating in aircraft turbine engines; and Y, Gd, Lu as the hosts and Eu, Tb, Dy and Er as the activators in phosphors for display units. In the energy generating and transmission sectors: Nd in NdFeB permanent magnets for wind generators; and Y in YBa2Cu3O7 superconductors in both wind generators and electrical transmission lines. While in the energy efficiency sector: phosphors (as noted above) in lighting – compact fluorescent lamps (CFL) and LED devices; La, Nd and Gd in magnetic refrigeration.

10:10 AM Break

10:25 AM Plenary
Materials R&D to Enable a Nuclear Energy Renaissance: *Steven Zinkle*[1]; [1]Nuclear Science and Engineering Directorate Oak Ridge National Laboratory

As part of a balanced portfolio of sustainable energy options, nuclear energy offers significant promise as a proven, cost-effective, and reliable baseline power option. In order to explore the potential to enhance the nuclear power contribution beyond its current level of 20% of US electricity production, three key initiatives are under investigation. The first initiative is examining the potential to safely and reliably extend the operational lifetime of existing nuclear power plants by quantifying the key materials degradation mechanisms and identifying materials replacement or damage mitigation solutions. The second initiative is exploring a broad range of new fission reactor concepts, encompassing Small (<300 MW) Modular Reactor designs that may be attractive for replacement of aging fossil energy plants as well as so-called "Generation IV" reactor concepts that offer further improvements in fuel sustainability, economics and safety. Finally, materials challenges for realizing practical fusion energy systems will be briefly reviewed.

10:50 AM Plenary
Energy Efficiency Studies: *Ken Somers*[1]; [1]McKinsey
Abstract not available.

11:15 AM Plenary
Materials Challenges for Solid Oxide Fuel Cells: Application of Metallic Materials and Analysis of Oxide Ionic Diffusion at the Component Interfaces: *Teruhisa Horita*[1]; [1]AIST

Solid Oxide Fuel Cells (SOFCs) can convert the chemical energy to the electricity directly with high efficiency. Because of their high operation temperatures around 873-1273 K, the component materials should be high temperature resistance materials such as alloy and ceramics. In this presentation, technological issues and challenges will be shown for improving the SOFC performance. Our recent activities related to the SOFC materials will be presented: 1) high temperature oxidation of alloy and compositional modification of alloy, 2) oxygen ionization and oxide ionic diffusion of cell components, and 3) degradation and durability of SOFC component materials with small amount of impurities. A short technological review will be made on the SOFC demonstration project in Japan as well as PEFC commercialized system.

11:40 AM Plenary
The Pivotal Role of Materials Science and Engineering for an Energy Efficient and Low Carbon Economy: *Diran Apelian*[1]; [1]Worcester Polytechnic Institute

The most critical issue we face as a global society for a sustainable 21st century on the planet earth is energy. During the last 10 years, world population increased by 1billion - from 6 billion to 7 billion in one decade. Annual projections for population growth hover a bit over 1.4% whereas energy consumption is growing at a faster rate ~1.7%; certainly not a sustainable scenario. Innovation in materials and material processing technologies is critical to achieving the longer term objectives of an energy-efficient and low-carbon world. While significant efforts have been made to identify breakthrough materials and their benefits, less attention has been given to the integration with materials manufacturing, including synthesis science, needed to propel promising materials candidates across the "valley of death" into cost-effective application at scale. This presentation will provide an overview of a study commissioned by the U.S. Department of Energy focused on identifying those areas where materials science and engineering can have the most significant impacts on energy efficiency and carbon reduction.

Materials for the Nuclear Renaissance II: Materials and Welding

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS/ASM: Nuclear Materials Committee
Program Organizers: Raul Rebak, GE Global Research; Brian Cockeram, Bechtel-Bettis; Peter Chou, Electric Power Research Institute; Micah Hackett, TerraPower, LLC

Monday AM — Room: 4
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chair: Raul Rebak, GE Global Research

8:30 AM Introductory Comments

8:35 AM Invited
Nickel Alloys Used in Nuclear Power Systems: Julie Tucker[1]; *George Young*[1]; Micah Hackett[1]; [1]Knolls Atomic Power Laboratory

Nickel based alloys are used extensively in nuclear power systems due to their excellent combination of corrosion resistance, strength, and toughness. However, primary water stress corrosion cracking (PWSCC) of Alloy 600-type materials has been a significant issue in the commercial nuclear industry. Attempts to mitigate PWSCC via weld repair using high chromium filler metals is hindered by their susceptibility to both subsolidus (e.g. ductility dip cracking) and supersolidus (e.g. solidification cracking) defects. New construction utilizing PWSCC resistant alloys (e.g. A690) face similar challenges; high chromium nickel based alloys are susceptible to welding-induced cracking. This talk presents an overview of weldability and environmentally assisted cracking in nuclear power systems; presents work to quantify and predict primary water stress corrosion crack growth rates; and summarizes alloy 690 weld metal development research. This research has resulted in a new nickel alloy filler metal with a desirable combination of PWSCC resistance and weldability.

9:15 AM
Effect of Residual Stresses on SCC Crack Growth Specimens Fabricated from Weld Metal: *Matthew Kerr*[1]; Mike Hill[2]; Alexandreanu Bogdan[3]; Darrell Dunn[1]; [1]US Nuclear Regulatory Commission; [2]Hill Engineering, LLC; [3]Argonne National Laboritory

Pressurized water reactor piping system dissimilar metal welds are susceptible to primary water stress corrosion cracking (PWSCC) as an active degradation mechanism. PWSCC is highly influenced by the state of stress within susceptible material with tensile residual stresses in welds an

established driving force for PWSCC. Proper predictions or measurements of these residual stresses are essential to accurate crack growth assessment. The US Nuclear Regulatory Commission Office of Research is currently undertaking several programs aimed PWSCC mitigation and weld residual stress characterization. This talk focuses on the effect of residual stresses on welded SCC crack growth specimens fabricated from weld metal typical of nuclear reactor piping systems.

9:35 AM

Enhancement of Intergranular Corrosion Resistance of TIG Welded and Laser-surface Melted SUS 304 for Nuclear Power Plants: *Joung Soo Kim*[1]; Chin-Man Chung[1]; Sung-Hoon Baik[1]; Sang-Bae Lee[2]; [1]Korea Atomic Energy Research Institute; [2]Korea Institute of Science and Technology

In order to improve intergranular corrosion including intergranular stress corrosion cracking resistance of Type 304 stainless steel weld, its surface was melted using an pulse Nd:YAG laser beam. The thickness of surface melted by the laser beam was obtained to be around 390 μm at a scan speed of 600 mm/min. and a laser power density of 20 J/mm2. The microstructures of the TIG welded region and the region laser-surface melted on the TIG weld were observed and analyzed using OM, SEM, and TEM attaching EDS. Then intergranular corrosion resistance of the two different specimens were characterized using a double loop electrochemical potentiodynamic reactivation(DL-EPR) polarization method at the same test condition. Intergranular corrosion resistance of the laser-surface melted SUS 304 weld was observed to be much higher than that of only TIG welded SUS 304. In this presentation, the mechanism of intergranular corrosion improvement by laser-surface melting will be discussed.

9:55 AM

Mechanical Properties and Microstructural Evolution of ODS Alloys Joined by Solid State Welding: *Evan Young*[1]; James Carillo[1]; Brian Jaques[1]; Jatu Burns[1]; Larry Zirker[2]; Indrajit Charit[3]; Darryl Butt[1]; Megan Frary[1]; [1]Boise State University; [2]Idaho National Laboratory; [3]University of Idaho

Oxide dispersion strengthened (ODS) alloys are used in nuclear reactors for their excellent high temperature strength, corrosion resistance and radiation damage resistance. Although the properties of ODS alloys make them ideal for nuclear reactor applications, conventional joining techniques degrade material properties. This study evaluates the mechanical properties and microstructural evolution that result from joining a ferritic alloy (HT-9) to an ODS alloy (Fe-based MA956 or Ni-based MA754) using either friction stir welding or pressure resistance welding. The mechanical properties of the welds are determined using both hardness mapping across the weld interface and crack growth experiments. Microstructural evolution is evaluated using electron backscatter diffraction and energy dispersive spectroscopy. Results show discrete regions with differing grain size and orientation across the weld affected zone. The goal of the work is to develop better welding procedures for ODS alloys such that these materials could be used more widely in nuclear applications.

10:15 AM Break

10:25 AM

Laser Welding for Nuclear Power Systems: *Julie Tucker*[1]; Terrance Nolan[1]; George Young[1]; [1]Knolls Atomic Power Laboratory

Advances in laser welding technology are increasing the applicability of this joining technique to nuclear system welds. Advantages of laser welding include fiber optic delivery, high power density, small heat affected zones, minimal distortion and limited susceptibility to solidification or liquation cracking. These advantages are of special interest to next generation nuclear power systems where dissimilar metal welds are likely. Laser welds can be prone to defects, especially porosity and oxide inclusions. This work uses the corrosion resistant, nuclear structural material Alloy 690 to illustrate the effects of shielding, travel speed, focal point, and joint type on the penetration, defect type, and defect density in autogenous welds. The test welds were characterized by light optical microscopy, scanning electron microscopy, and x-ray computed tomography to quantify welding defects. Results show that defect density decreases with increasing defocus. Additionally shielding gas can have a large effect on defect type and weld penetration.

10:45 AM

Pressure Resistance Welding for Advanced Reactor Applications: *Nathan Jerred*[1]; Larry Zirker[2]; Indrajit Charit[1]; Jim Cole[2]; Brian Jaques[3]; Troy Bradshaw[4]; James Carrillo[3]; Evan Young[3]; Megan Frary[3]; Darryl Butt[3]; Mitch Meyer[2]; K. Murty[4]; [1]University of Idaho; [2]Idaho National Laboratory; [3]Boise State University; [4]North Carolina State University

Pressure resistance welding (PRW) has been used to produce solid-state joints of high temperature materials, such as oxide dispersion strengthened (ODS) alloys, ferritic/martensitic steels and tungsten. The key emphasis of this study is to optimize the PRW parameters and characterize the welds of these alloys joined in similar and dissimilar fashions, and establish appropriate processing-microstructure-property relationships to facilitate their use in various nuclear applications. Initial studies have suggested that high quality welds can be made between MA957 cladding tubes and HT9 end-plugs. Initial bend and burst tests have shown encouraging results. Current efforts also include joining similar ODS alloys (MA956 and MA754) and tungsten coupons. A multi-physics program (COMSOL) has been employed to model the PRW process and predict initial input parameters prior to welding. Weld characterization experiments include micro-hardness, three-point bend, burst and tensile tests for mechanical integrity, and optical microscopy and scanning electron microscopy for microstructural characteristics.

11:05 AM

Surface Modification of 316L Stainless Steel by a Low Temperature Severe Plastic Deformation Linear Raking Process: *Giovanni Facco*[1]; Shashank Shekhar[1]; Andreas Kulovits[1]; Ravi Shankar[1]; Jorg Wiezorek[1]; [1]University of Pittsburgh

The plane-strain machining process, linear racking, is a novel technique to modify microstructures of complex multi component alloy systems, which can be easily up-scaled. Linear raking offers access to severe plastic straining at high rate, modifies heat treatment alloy responses, often improving properties. Here we apply linear raking to 316L stainless steel to produce nano-structured and grain-boundary-engineered sub-surface layers, which could provide for enhanced mechanical and corrosion performance and improve irradiation tolerance. We use electron backscatter diffraction in the scanning electron microscope and automated acquisition and indexing of transmission electron microscope diffraction patterns to monitor the microstructure changes imposed by linear raking and annealing based processing. We correlate them with mechanical properties and stress corrosion susceptibility studies. We acknowledge using facilities of the Materials Micro-Characterization Laboratory, Department of Mechanical Engineering and Materials Science, University of Pittsburgh, and support by a grant from the Nuclear Regulatory Commission, NRC-38-09-935.

11:25 AM

Serrations in Austenitic Fe-Cr-Ni Alloys: *Young Suk Kim*[1]; Sung Soo Kim[1]; Dae Whan Kim[1]; [1]Korea Atomic Energy Research Institute

Tensile tests were conducted on 316L stainless steel with nitrogen over a temperature range of RT to 750oC at different strain rates of 1x10-2/s to 2x10-4/s. The 316L stainless steel showed serrated flow, a linear increase of strain hardening from 400 to 600oC and a negative strain rate sensitivity of strain hardening due to dynamic strain aging (DSA). Using neutron diffraction, the lattice contraction due to short range ordering (SRO) was seen to occur in the 40% cold-worked 316 stainless steel on aging at 400oC corresponding to a minimum of the tensile elongations. We first suggest that SRO is the cause of shear localization and serrated flow in austenitic Fe-Cr-Ni alloys. Using TEM, planar dislocations were confirmed in the 316L stainless steel after tensile deformation at 400oC where SRO was operating. This study definitively demonstrates that SRO is the cause of serrations and DSA in austenitic Fe-Cr-Ni alloys.

11:45 AM
Precipitation and Spinodal Decomposition in a Lean Grade of Duplex Stainless Steel: *Julie Tucker*[1]; George Young[1]; Daniel Eno[1]; [1]Knolls Atomic Power Laboratory

Duplex stainless steels are desirable for use in power generation systems due to their attractive combination of strength, corrosion resistance, and cost. However, thermal embrittlement at high temperature (~750°C), due to the precipitation of undesirable phases, can complicate fabrication. Similar embrittlement at low temperatures (~475°C), via spinodal decomposition, limits upper service temperatures for many applications. The recent development of lean grade alloys may improve the manufacturing margin and potentially increase the upper service temperature of these alloys. The present work assesses the thermal stability of lean grade of duplex stainless steel, AL2003 through a series of isothermal agings between 280°C and 815°C for times between 1 and 10,000 hours. Aged samples were characterized by changes in microhardness and Charpy-impact toughness. Microstructural characterization of the aged samples via transmission electron microscopy was also performed. This analysis has determined the rates and the temperatures at which these phase transformations occur.

12:05 PM
High Temperature Fracture Toughness of Thermally Aged Inconel 617: Mikhail A. Sokolov[1]; *Randy Nanstad*[1]; [1]Oak Ridge National Laboratory

Nickel-base alloy Inconel 617 is being considered as a structural material for application in the secondary heat exchanger of the New Generation Nuclear Plant, a very high temperature gas-cooled reactor. Inconel 617 has previously been shown to exhibit degraded toughness following thermal aging at elevated temperatures. Thermal aging of Inconel 617 plate and welds is being performed with tensile, Charpy impact, and fracture toughness tests conducted at temperatures to 950°C. Results of testing for thermal aging to 2,000 h have been obtained and are presented; varying effects of thermal aging temperature and time on fracture toughness are observed.

Materials Processing Fundamentals: Solidification, Deformation, and Heat Treatment

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Technology and Modeling Committee
Program Organizers: Prince Anyalebechi, Grand Valley State University; Srikanth Bontha, Temple University

Monday AM
February 28, 2011

Room: 12
Location: San Diego Conv. Ctr

Session Chair: Prince Anyalebechi, Grand Valley State University

8:30 AM
The Influence of Solidification Rates on Hot Workability and Mechanical Properties of AM60 Magnesium Alloy: *Goran Kugler*[1]; Milan Tercelj[1]; [1]University of Ljubljana, NTF-OMM

The use of the up-to date light alloys and their development are in the sharp ascent especially in transportation industry. In the last years the use of the magnesium alloys for the various purposes was strongly increased. The aim of present research was to study the influence of solidification rate on hot workability and on obtained mechanical properties of AM60 magnesium alloy. The study was also focused on possibility to omit prior homogenisation process which would result in cost reduction. The solidification of gravity cast alloy has been investigated with various methods of the thermal analysis: "in situ" simple thermal analysis and simultaneous thermal analysis. In order to reveal optimal hot working conditions the deformation behavior was studied in the temperature range 200-450 °C and strain rates between 0.001-10 s^{-1}. Mechanical properties have been studied with tensile and impact toughness testing.

8:45 AM
Interplay of Flow and Solidification in a Horizontal Centrifugal Casting: *Autumn Fjeld*[1]; [1]University of Leoben

A large-scale industrial centrifugal casting is simulated to investigate the interplay of flow and solidification during filling. In the casting under study the behavior of the flow is not fully understood; the relative motion of molten metal in the axial and circumferential directions is examined as well as the existence of a layered structure as the liquid contacts the mold and begins to solidify. Pick-up speed of the incoming metal is determined and influences of casting temperature, heat extraction, pouring rate, and rotation are considered. A single alloy and dual-alloy casting are simulated. In the case of a dual-alloy casting the mixing of the two alloys as the second alloy is poured onto the existing first alloy layer is characterized.

9:00 AM
On the Homogenization of Cobalt Modified 17-4 PH Stainless Steel: *Arpana Murthy*[1]; Simon Lekakh[1]; Von Richards[1]; David Van Aken[1]; [1]Missouri S&T

Three heats of 17-4PH stainless steel: with zero, three percent, and seven percent cobalt addition were melted in a 45kg (100lb) induction furnace under argon; and cast into no-bake molds and investment shell molds. Specimens were sealed into quartz tubes under vacuum, and homogenized at 1200°C from 2h to 72h. Computational thermodynamics and optical microscopy were used to predict and characterize the microstructures. A compromise between decrease in segregation and increase in lath martensite packet size was chosen for homogenization time. The specimens were austenitized, quenched, and characterized for retained austenite using X-Ray diffraction. Age hardening kinetics were studied at 454°C(850°F), 468°C(875°F) 482°C(900°F), and the activation energies for precipitation of copper were calculated. Tensile properties and Charpy V-notch toughness were measured in the peak aged condition. Fracture surfaces were observed using Scanning Electron Microscope. The addition of cobalt was found beneficial for the improvement of strength and Charpy V-notch toughness.

9:15 AM
Investigation of the Effects of Solidification Rate and Melt Hydrogen Concentration on Porosity Formation in Aluminum Alloy 2024: *Prince Anyalebechi*[1]; [1]Grand Valley State University

The effects of solidification rate, melt hydrogen concentration, and grain refining on porosity formation in aluminum alloy 2024 have been investigated with unidirectionally cooled laboratory-size ingots. The ingots were prepared from melts containing hydrogen concentration of 0.067, 0.19 cm3/100 g, and 0.27 cm3/100 g and solidified at 0.2-10 K/s. As expected, the amount of porosity and average pore size increased with increase in melt hydrogen concentration and decrease in solidification rate. However, the effect of solidification rate was greater at the relatively very low melt hydrogen concentration (0.067 cm3/100 g). Interestingly, addition of grain refiner decreased the amount of porosity and the average pore size. These results are consistent with reported effects of solidification rate, melt hydrogen content, and grain refining on porosity formation in other aluminum alloys.

9:30 AM
Characterization of the Microstructure of Commercial-Size Ingots of Aluminum Alloy 3004: *Prince Anyalebechi*[1]; [1]Grand Valley State University

The as-cast microstructure of two commercial-size electromagnetic cast ingots of aluminum alloy 3004 was quantitatively characterized. This involved the quantitative determination of across-width and through-thickness variations in dendrite cell size, grain size, macrosegregation of alloying elements, interparticle spacing, and the types, volume fractions, and sizes of the constituent or second phase particles. A significant microstructural heterogeneity across the width and through the thickness of both ingots was observed. For example, macrosegregation, and the sizes of the constituent phase particles, dendrite cells, and grains increased concomitantly from edge to mid-width and from surface to center of the ingots. There was a -20% depletion of Mg and a +54-87% enrichment of Ti in the center of the ingots. With the exception of grain size, there was no significant microstructural difference between the two ingots examined. The causes and practical

implications of the observed microstructural heterogeneity in the ingots are discussed.

9:45 AM

Expansion and Collapse of Liquid Aluminum Foams: *Zhuokun Cao*[1]; Chuan Li[1]; Hongjie Luo[1]; Guangchun Yao[1]; [1]Northeastern University, China

The expansion rate and collapse time of liquid aluminum foams are essential for the determination of operating time and quality of the resulting foam. In the present paper, liquid aluminum foams was fabricated by adding in TiH2 as blowing agent, and the variation of expansion rate with foaming time was non-contact detected by a laser distance measurer. The influence of different stabilizing agent and alloying elements was studied. The results show the fact that the fraction of stabilizing agent only perform little effects on the maximum expansion rate of liquid aluminum foams, while the influence of alloy elements are remarkable. The maximum expansion rate would increase much when Mg is added, and the liquid foam also collapse at a shorter time. Comparison was made between experimental results and mathematic cancelations, which reveal the fact that it is the change of TiH2 decomposition responsible for the change.

10:00 AM Break

10:15 AM

Review of Classical Design Methods as Applied to Aluminium Billet Heating with Induction Coils: *Mark Kennedy*[1]; Shahid Akhtar[1]; Jon Bakken[1]; Ragnhild Aune[1]; [1]Norwegian University of Science and Technology

In the present study classical induction design tools are applied to the problem of heating non-magnetic metal billets, using 50 Hz AC. As an example of great practical industrial interest, the induction heating of aluminum billets is addressed specifically. The predicted work piece power is compared with the measured work piece power for a long and a short coil, using well established methods, such as those of Burch and Davis, introduced in 1926/28, Dwight and Bagai in 1935, Baker in 1944/57, Vaughan and Williamson in 1945, and by Tudbury in 1960. A calculation methodology based on a combination of the available tools is also introduced and discussed. The method has proven to give an error of <10% of the actual work piece power. An equation for Tudbury's work piece shortness correction factor is disclosed for the first time.

10:30 AM

Interactions of Non-metallic Inclusions with Steel and Slag: Thermodynamic Modeling, Experiments and Metallographic Analyses: *Susanne Michelic*[1]; Mario Hartl[1]; Christian Bernhard[1]; [1]University of Leoben

One aspect of an efficient process and product optimization of the liquid steelmaking process is the understanding of reactions and interactions between the components steel, slag and non-metallic inclusions. The present paper focuses on a fundamental study of this topic by combining the powerful methods of thermodynamic modeling with systematic experiments on a laboratory scale and subsequent SEM/EDS analyses. The modeling aspect is addressed by the commercial software FactSage 6.1; laboratory experiments are performed for a low-alloyed, Ca-treated carbon steel combined with varying slag compositions. The consequent SEM/EDS analyses provide a detailed insight into the inclusion landscape which offers a strong tool for characterizing the modification of in¬clusions. In the present study the combination of these methods is mainly applied to oxides, due to their crucial influence on the final product quality, thus giving important indications for further optimizing the secondary metallurgy processes.

10:45 AM

Liquid Metal Flows under Non-Homogeneous Magnetic Field: Lorentz Force Flowmeters in Metallurgy: *Jurijs Kolesnikov*[1]; Christian Karcher[1]; André Thess[1]; [1]Ilmenau University of Technology

During the motion of molten metal under external non-homogeneous magnetic field the induced electric currents produce Lorentz body forces in the opposite direction to the flow, causing deformation of velocity profile, vortical structures, and adding a pressure drop across the magnetic field. The same Lorentz force acts on the magnet system. We analyse these phenomena in metallurgy, namely, at molten metal pumping, stirring, continuous casting, and in contactless Lorentz force flowmeter (LFF). Experiments with liquid metal alloy, including direct measurements of Lorentz forces, velocities, electrical potentials, and flow structure visualization were conducted. Based on this study the industrial prototypes of LFF systems were designed, built, and tested in non-ferrous metallurgy and secondary aluminium production. A molten metal flow at high temperature (700°C–1200°C) can be effectively measured. The LFF technique method allows measuring precisely the volumetric and mass flow rates linearly linked with Lorentz force, and the mass accumulated within production process.

11:00 AM

Study of Bake Hardening Effect on Laser Welded Hot Rolled Bainitic Steel: *Mehdi Asadi*[1]; Heinz Palkowski[1]; Nicole Schlosser[1]; [1]TU Clausthal

The influence of bake hardening (BH) effect on laser welded hot rolled bainitic steel (BS) with respect to the process condition was investigated. Two process conditions were developed for this study. At the first one the samples were pre-strained and then laser welded. At the second one the samples were firstly laser welded and then pre-strained. Pre-straining the samples with defined degrees of deformation and a subsequent aging treatment leads to enhanced hardness and strengthening for the both conditions. The microstructure of the laser track zone (LTZ) and heat affected zone (HAZ) was studied. A high volume fraction of martensite could be observed in the LTZ as well as HAZ. The BS steel exhibited a clear BH effect in both, the as-received and the laser welded conditions. The BH effect is more pronounced in the pre-strained laser welded condition.

11:15 AM

Research on the Combination of Microwave and Heat Pump Drying of Silica Sand: Hao Niu[1]; Yu Li[1]; Ying Lei[1]; Libo Zhang[1]; *Jinhui Peng*[1]; Huilong Luo[1]; Shenghui Guo[1]; [1]Key Laboratory of Unconventional metallurgy, Ministry of Education

In this work, the combination of microwave drying (MWD) and heat pump drying (HPD) drying experiments were designed by response surface methodology (RSM) based on central composite design (CCD) using design expert software (Version 7.15), and the process parameters were optimized. The conditions given by software are: the MWD time is 4.6 or 4.4 min; the HPD time is 9.0 or 8.6 min; and the sample mass is 620 or 600 g. Under those conditions the moisture content is 1.0 10-2kg water/kg wet basis, and the effectiveness ratio is 0.70 kg water/kwh. The results of confirmatory experiments conducted under the optimized conditions show the combined drying have superiority of decrease of energy consumption and increase of handling capacity over the single MWD or HPD.

11:30 AM

Surface Modification by Burnishing and Shot Peening Processes: *Syed Hasan*[1]; M.A. Sadiq[1]; G. Rangajanardhan[2]; V Murti[3]; [1]Deccan College of Engg & Tech; [2]J N T U Vijayanagaram; [3]S V I T S

Burnishing and shot peening are surface modification processes involving cold working of the cutaneous layer of the part surface. Their commonality extends to the generation of compressive residual stresses and improvement of fatigue strength. Burnishing employs a spherical ball to press and shot peening employs spherical shots to impinge on the target surface. Their relative characteristics form the subject of this study. Burnishing reduces surface roughness and produces highly improved surface texture. Whereas peening produces a coarse surface texture with increased surface roughness thus clearly demarcating their suitable applications for surface modification. Burnished surfaces showed furrows from penetration and feed of the ball along the surface. Shot peening produces a matty surface with overlapping dents from impinging shots. The residual stresses and microhardness were more in shot peening compared to burnishing. Stainless steel with higher yield strength had higher induced stresses as well as microhardness compared to aluminium.

Microstructural Processes in Irradiated Materials: Defects and Defect Processes

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee

Program Organizers: Gary Was, University of Michigan; Thak Sang Byun, Oak Ridge National Laboratory; Shenyang Hu, Pacific Northwest National Laboratory; Dane Morgan, UW Madison; Yasuyoshi Nagai, Tohoku University

Monday AM
February 28, 2011

Room: 3
Location: San Diego Conv. Ctr

Session Chairs: Gary Was, University of Michigan; Roger Stoller, Oak Ridge National Laboratory

8:30 AM Introductory Comments

8:40 AM Invited

Modeling Point Defect Cluster Behavior and Their Impact on RPV Embrittlement: *Roger Stoller*[1]; [1]Oak Ridge National Laboratory

Almost all radiation damage accumulation is the result of incomplete recombination of the vacancies and interstitials produced by the primary damage events. Thus, understanding the behavior of these point defects is essential to any predictive radiation damage model. The role os point defect clusters is particularly important in the case of cascade-producing neutron irradiation. A previously developed kinetic embrittlement model has been revised to account for recent advances in cluster dynamics modeling and insights obtained from molecular dynamics simulations. The impact of the these revisions will be presented and assessed in terms of available experimental data, including data from commercial reactor surveillance programs. The new model will provide an improved basis for evaluating the effects of neutron flux (displacement rate) which is critical for determining how high flux data is being used to obtain high fluence data for reactor life extension.

9:20 AM

Evolution Kinetics and Sink Strength of Interstitial Loops in Irradiated Materials: A Phase-field Model: *Shenyang Hu*[1]; Chuck Henager Jr.[1]; Yulan Li[1]; Fei Gao[1]; Xin Sun[1]; Mohammad Khaleel[1]; [1]Pacific Northwest National Laboratory

Interstitial loops are one of evolving defects in irradiated alloys. Their evolution, including spatial and size distributions, affects both vacancy and interstitial accumulation in the matrix, hence, the void formation and volume swelling. In this talk, we present a phase-field model to simulate the growth kinetics of interstitial loops under irradiation. The model takes into account the generation and reaction of vacancies and interstitials, and elastic interaction between interstitial loops and point defects. The local defect flux around the interstitial loop, the effect of defect generation rate, defect mobility and elastic interaction on growth kinetics and the sink strength, and the stability of interstitial loops are investigated. We will compare and discuss the results obtained from simulations and calculated by an empirical formula that is derived with a number of assumptions.

9:40 AM

Void Ordering and Swelling Saturation as a "Chicken and Egg Problem": *Stanislav Golubov*[1]; Alexander Barashev[2]; Roger Stoller[1]; [1]ORNL; [2]The University of Liverpool

Formation of void lattices in metallic crystals exposed to irradiation with energetic particles is a most intriguing phenomenon, which has drawn the attention of both experimentalists and theoreticians for about 40 years. Two striking features of the void lattices are: (1) coincidence of their type and orientation with the crystal lattice and (2) the saturation of swelling. It is commonly believed that (1) is due to one-dimensional migration of self-interstitial clusters produced in displacement cascades, while (2) is due to the resistance of voids to growth when arranged in a lattice. However, there is a contradiction between these two explanations since the swelling rate would increase due to free channel formation. In this paper the situation is critically analyzed, so as to determine whether either phenomenon can be implicated as the cause of the other, or whether an underlying mechanism is responsible for both the void lattice and swelling saturation.

10:00 AM

Non-Saturable Sinks at Grain Boundaries in Nanostructured Mo-Molecular Dynamics Simulations: *Yongfeng Zhang*[1]; Hanchen Huang[2]; Paul Millett[1]; Michael Tonks[1]; Dieter Wolf[1]; Simon Phillpot[3]; [1]Idaho National Lab; [2]University of Connecticut; [3]University of Florida

The defect accumulation in nanocrystalline Mo under electron irradiation is studied using molecular dynamics (MD) simulations. The nanostructured Mo is simulated using a bi-crystal model with <100> □29 twist boundaries as representation of high angle/energy grain boundaries. By assigning a kinetic energy of 40 eV to randomly selected Primary Knock-on Atoms (PKA), electron irradiation is simulated under 2000K until the defect concentration reaches steady state (in < 100 ns). The development of defect concentration obtained from MD simulations agrees well with the prediction of rate theory with parameters derived from MD simulations. With regard to the constant sink strength used in rate theory, this result suggests that high angle/energy grain boundaries are non-saturable sinks for point defects under electron irradiation. In supporting, structural analysis of both short range order by pair correlation function and long range order by planar structure factor shows no change in the grain boundary structure.

10:20 AM Break

10:40 AM

Modelling of Displacement Cascades in Thin Foils of Iron: *Andy Calder*[1]; Yuri Osetsky[2]; David Bacon[1]; Alexander Barashev[1]; [1]University of Liverpool; [2]ORNL

Recent MD simulations of cascades in bulk iron have revealed a sub-picosecond shock-front mechanism for the creation of large interstitial clusters and that the nature of this damage is determined before the thermal spike phase of the cascade. A decelerating supersonic front from the primary recoil event produces an expanding volume of destroyed lattice. Large self-interstitial clusters form on the transonic boundary of this zone if a peripheral hypersonic recoil had created a secondary zone just ahead of this boundary. Where the two zones meet, one injects atoms into the low-density core of the other and they become interstitial clusters. Modelling has now been extended to study free-surface effects in thin samples of iron, allowing direct comparison with experimental results on ion-irradiated films from earlier TEM studies and current picosecond-to-microsecond time-resolved x-ray scattering measurements of displacement cascade dynamics.

11:00 AM

Stoichiometry Dependence of the Evolution of Irradiated-Induced Defect Clusters in $Ce_xLa_{1-x}O_2$: *Wei-Ying Chen*[1]; Di Yun[2]; Aaron Oaks[1]; Bei Ye[1]; Mark Kirk[2]; James Stubbins[1]; Yinbin Miao[1]; [1]U of Illinois at Champaign-Urbana; [2]Argonne National Lab

To study the stoichiometry dependence of irradiation effects in fluorite-type mixed oxide nuclear fuel ($UPuO_2$), the technique of ion implantation in La doped ceria ($Ce_xLa_{1-x}O_2$) is used. Xe ions were implanted into cerium dioxide single crystals with 0%, 5% and 25% La concentration at 600°C. In-situ transmission electron microscope (TEM) was utilized to observe the the damage process and defects created by the ion beam irradiation. A substantial difference in dislocation loop density for 5% and 25% cases was observed at the same dose. For example, loop density is $2x10^{11}$ cm^{-2} on CeO_2 doped with 5% La and $2x10^{10}$ cm^{-2} with 25% La when the dose reaches $1x10^{14}$ ions/cm^2. Kinetic Monte Carlo calculations were also performed to study the oxygen diffusivity in $Ce_xLa_{1-x}O_2$ at different stoichiometric conditions. Calculation results show the oxygen diffusivity dependence on La concentration, which compliments the experiment results.

11:20 AM

Relevance of fcc-bcc Interface Structure to Defect Properties at Interfaces in Irradiation Environment: *Xiang-Yang Liu*[1]; Richard Hoagland[1]; Michael Demkowicz[2]; Xiang-Ming Bai[1]; Blas Uberuaga[1]; Michael Nastasi[1]; Amit Misra[1]; John Hirth[1]; [1]Los Alamos National Lab; [2]Massachusetts Institute of Technology

Nanolayered Cu-Nb composites exhibit high strength and enhanced radiation damage tolerance. To understand the relevance of interface structure to interface properties in fcc-bcc systems, tunable potentials offer a fairly simple way to selectively vary parameters independently. In this work, the parameterization of the EAM interatomic potential in fcc-bcc system is modified to understand the interface properties. We first change the dilute heats of mixing between Cu and Nb and investigate the effect on interface structure and defect formation energies near interface. To understand the interface behavior in different lattice misfit environment, the relative lattice constants between Cu and the bcc crystal are varied. Interface dislocation analysis based on Frank-Bilby formulation is to be presented, together with atomistic simulation result. Defect-interface interactions are studied with molecular dynamics (MD) and accelerated MD method, to predict the radiation damage tolerance of these interface systems.

11:40 AM

Towards a Unified Framework for Interatomic Potential Development: Application to the Fe-He System: *Mark Tschopp*[1]; Kiran Solanki[1]; Mike Baskes[2]; Mark Horstemeyer[1]; Fei Gao[3]; Xin Sun[3]; Moe Khaleel[3]; [1]Mississippi State University; [2]LANL; [3]PNNL

Atomistics simulations require interatomic potentials that are able to reproduce electronic structures information while predicting properties outside of the values they were fit to. However, the design cycle for new interatomic potential development is often a lengthy process. The objective of this research is to outline a methodology for efficiently probing potential parameter space to locate optimal interatomic potentials. Here, we use a combination of random sampling techniques, response surface methodology, and optimization methods to develop an Fe-He modified embedded atom method (MEAM) interatomic potential. This methodology will be used to develop multiple Fe-He potentials, which will then be used to calculate the variability in atomistic properties based on the interatomic potential. Moreover, this methodology will be applied to other interatomic potentials to show the robustness of the methodology. Atomistic simulations rely upon good interatomic potentials and shortening this design cycle can encourage tailoring interatomic potentials for specific applications.

12:00 PM

A New Directional Model for the Electronic Frictional Forces in Molecular Dynamics Simulations of Radiation Damage in Metals: *Christopher Race*[1]; Daniel Mason[2]; Adrian Sutton[2]; [1]Max-Plank-Institute for Iron Research; [2]Department of Physics, Imperial College London

The damage caused by collision cascades in irradiated materials forms the initial conditions for longer term microstructural evolution. The exchange of energy between electrons and ions during cascades can significantly affect this damage. Models for incorporating this exchange within classical molecular dynamics (MD) simulations exist, but all approximate the ion-electron energy transfer via a damping force, opposed to ionic motion. Although such forces predict the total energy transfer over the duration of cascades, they do not capture the complex dependence of the electronic friction force on the speed, direction and atomic environment of individual ions.Here we present a new model for the electronic friction force, derived from quantum-classical Ehrenfest dynamics, which captures this complexity and is suitable for inclusion in existing MD codes at near zero computational cost. We show that our model reproduces the atomic level detail of the electronic friction force in time-dependent tight-binding simulations of cascades.

Neutron and X-Ray Studies of Advanced Materials IV: Interfaces, Surfaces, Nanostructures

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Xun-Li Wang, Oak Ridge National Laboratory; Jaimie Tiley, Air Force Research Laboratory; Peter Liaw, The University of Tennessee; Erica Lilleodden, GKSS Research Center; Brent Fultz, California Institute of Technology; Y-D Wang, Northeastern University

Monday AM — Room: 10
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Dean Haeffner , Argonne National Laboratory; Rozaliya Barabash, Oak Ridge National Laboratory

8:30 AM Keynote

Study of Advanced Materials and Devices Using High-Resolution Hard X-Ray Microscopy: *Jörg Maser*[1]; Martin Holt[1]; Robert Winarski[1]; Volker Rose[1]; Peter Fuesz[1]; Brian Stephenson[1]; [1]Argonne National Laboratory

The Center for Nanoscale Materials' Hard X-ray Nanoprobe (HXN) provides characterization of composition and structure of nanoscale materials and devices with high spatial resolution. A major contrast mode is nanodiffraction, which allows mapping of local phase and strain with a spatial resolution of 40 nm. This provides a powerful tool for investigation of advanced thin film materials and devices, where strain can play an important role in materials properties such as phase transition temperature and electrical conductivity. Our high spatial resolution allows us to study materials systems at technologically relevant length scales. We will present studies of structural changes in resistive RAM structures, strained nanoscale silicon-on-insulator channels, and ferroelectric domains in nanocapacitors, as well as discuss a coherence-based techniques for the high resolution study of crystalline thin films.

8:55 AM

Depth-Dependent Strain Gradients and Interface Strength of Submicron Single Crystalline Mo in Brittle or Ductile Environment from 3D Micro-Laue Diffraction: *Rozaliya Barabash*[1]; Hongbin Bei[1]; Yanfei Gao[1]; Oleg Barabash[1]; Gene Ice[1]; [1]Oak Ridge National Laboratory

Advanced composites with metal or ceramic constituents offer high strength and have a number of advantages with respect to individual phases. As the size of reinforcing fibers/whiskers/lamellae decreases, the importance of defects, boundaries and interfaces increase. Using 3D X-ray microdiffraction we nondestructively probe the depth-dependent strain gradients and interface strength in model systems of submicrom single crystalline Mo in Nial or Ni matrix (JMR 25, 2, 2010). Coupled with micromechanical analysis, our study shows that the relaxation of the residual thermal strains in the NiAl/Mo or Ni/Mo composites results in the formation of the near-surface "slip zone" with large strain gradients in both the reinforcing Mo fibers and the matrix. Based on these results we suggest a new approach to determine the fiber/matrix interface strength for nano- and micro-composites. Research was supported by the Materials Sciences and Engineering Division, Office of Basic Energy Science, U.S. Department of Energy.

9:10 AM Invited

Strain Screening by Oxygen Vacancies in $SrTiO_3$: *Joel Brock*[1]; [1]Cornell University

Recently, Freedman et al. [Phys. Rev. B**80**, 064108, 2009] calculated the elastic dipole tensor for several types of point defects in $SrTiO_3$ and showed that it is nearly traceless for oxygen vacancies. Thus, oxygen vacancies are able to screen elastic strain fields. Here, we report detailed diffuse x-ray scattering measurements of bulk $SrTiO_3$ crystals prepared with controlled oxygen vacancy distributions. We verify the traceless nature of the elastic

dipole tensor of an oxygen vacancy and demonstrate both correlations between oxygen vacancies and elastic strain screening by oxygen vacancies.

9:30 AM Invited

Structure and Thermal Evolution of a Metallic Glass That Grows from the Melt through a First-Order Transition: *Gabrielle Long*[1]; Karena Chapman[1]; Peter Chupas[1]; Leonid Bendersky[2]; Lyle Levine[2]; Judith Stalick[2]; Frederic Mompiou[3]; John Cahn[4]; [1]Argonne National Laboratory; [2]NIST; [3]CNRS; [4]University of Washington

In recent decades, metallic glass structures have been documented with increasing precision, but our understanding is still at best incomplete. One direct proof of this is the proliferation of partially successful models attempting to classify the underlying generalized structure. To approach a fundamental understanding of metallic glasses, it can be illuminating to investigate extreme examples. We investigated an unusual metallic glass that grows from the melt by a first order phase transition in the manner of a crystal, but scatters as a metallic glass. Well-defined nano-domains that closely resemble atomic arrangements within alpha-AlFeSi were identified. Under an isothermal anneal, the glass rejects aluminum and relaxes into a more stable and persistent configuration. As the anneal continues, there is a peritectic reaction in which the rejected aluminum-plus-glass undergoes a first order phase transition to a crystalline phase unrelated to the nano-ordered domains in the original glass, as if no nano-ordering existed.

9:50 AM Invited

Nanosescond Piezoresponse Measurements on Thin Epitaxial Ferroelectric Films at the Hard X-Ray Nanoprobe Beamline: Matthew Highland[1]; Martin Holt[1]; Robert Winarksi[1]; John Pearson[1]; G. Brian Stephenson[1]; Jorg Maser[1]; Stephen Streiffer[1]; Ralu Divan[1]; *Carol Thompson*[2]; [1]Argonne National Laboratory; [2]Northern Illinois University

Our goal is to study polarization dynamics in nanoscale ferroelectric heterostructures as the size of the structure approaches the domain size, and in ultrathin films where interfacial effects dominate. In this talk, we describe the development of stroboscopic scattering techniques using the Hard X-ray Nanoprobe at the Advanced Photon Source by implementing a chopper to isolate individual 100 ps x-ray pulses and synchronize them to electrical stimulation of the sample. We also describe our progress in developing protocols to pattern and integrate working devices from ultrathin epitaxial $PbTiO_3$ films on $SrRuO_3$/$SrTiO_3$ substrates. These devices retain the ferroelectric properties and crystalline quality of the virgin film, and allow electrical access within the constraints imposed by nanoprobe focusing optics. Results are presented showing nanosecond piezoelectric response of thin ferroelectric films using these devices. Work supported by US-DOE under Contract No. DE-AC02-06CH11357.

10:10 AM Invited

Kinetic Studies of Microstructure Evolution in Nanostructured Materials: *Matteo Leoni*[1]; Paolo Scardi[1]; Mirco D'Incau[1]; [1]University of Trento

The knowledge of the evolution in temperature of a nanocrystalline material is the key to its optimization for industrial applications. Synchrotron radiation X-ray diffraction and modern line profile analysis (e.g. the Whole Powder Pattern Modeling) are ideal tools to follow in situ kinetics on the nanoscale. The microstructure evolution data obtained from the analysis of diffraction patterns collected in ramp at several heating rates, show how X-ray diffraction can be the ideal complementary tool to thermal analysis. Basics and examples will be discussed.

10:30 AM Keynote

Advances in Neutron and X-ray Micro/Nano Diffraction: *Gene Ice*[1]; [1]Oak Ridge National Laboratory

Both X-ray and neutron microdiffraction methods are rapidly evolving due to the availability of powerful new sources and advanced focusing optics and detectors. Achromatic optics and methods are particularly interesting for studies of materials with unknown crystallographic orientation, and are essential to efficiently use spallation neutron sources. We describe recent spatially-resolved studies with X-ray and neutron probes that illustrate the future of these methods. We also describe how these techniques can be optimized to exploit a coming generation of new ultra-high-brilliance third generation X-ray sources and the new spallation neutron sources. Work sponsored by the U.S. Department of Energy (DOE), Office of Basic Energy Sciences, Division of Materials Sciences and Engineering. Experiments performed on Beamline 34-ID at the Advanced Photon Source Argonne Il, and the SNAP beamline at the SNS. Both 34-ID and SNAP are supported by the DOE Office of Basic Energy Science, Division of User Facilities.

10:55 AM Break

11:05 AM Invited

Adhesion, Cohesion and Plasticity of Thin Metal Films: *Ralph Spolenak*[1]; [1]ETH Zurich

Thin metal films as conductor materials for wearable microelectronics are usually deposited on polymeric substrates. Thus in addition to their electrical resistivity, the adhesion to the substrate, their plastic deformation modes and their resistance against fracture are of relevance for the application. In the current paper comprehensive studies of fracture patterns by direct observation (optical and scanning electron microscopy) as well as synchrotron based X-ray diffraction are presented. The materials range from brittle Ta and Ti films, where the effects of phase and patterning on fracture are discussed, to the precursors of plastic failure in the Au-Cu, Al-Mg and Al-Li systems. X-ray peak shifts as well as peak broadening is related to microstructural changes in the thin film materials and pathways for optimal ductility are elucidated.

11:25 AM Invited

X-Ray Diffraction Studies of Structural Transitions in Some Pnictide Superconductors: *Zahirul Islam*[1]; Jiun-Haw Chu[2]; Ian Fisher[2]; [1]Argonne National Laboratory; [2]Stanford University

In FeAs-based superconductors a structural transition coincides with or precede the antiferromagnetic transition for compositions in the underdoped regime. This transition lowers the rotational symmetry (e.g. from 4-fold to 2-fold in the case of tetragonal to orthorhombic transition) of the lattice. In these materials a strong anisotropy in electronic resistivity is observed, which increases as the superconducting phase is approached with compositional variation. Although a resistivity anisotropy is expected to arise from electronic order of some kind, its origin is made difficult to determine by the onset of a structural transition. In order to clarify the role of the lattice symmetry in this anisotropy a set of single-crystal diffraction studies of structural transitions was carried out. Results of these studies reveal that the degree of structural anisotropy is inadequate to account for resistivity anisotropy indicating an electronic origin.

11:45 AM Invited

X-Ray Diffraction in Short-Period Superlattices: *Emil Zolotoyabko*[1]; [1]Technion

Short-period superlattices, based on semiconductor or oxide layers, have wide range of applications ranging from infrared imaging to giant magneto-resistance. Some applications require deposition of sub-layers with thicknesses one monolayer and below. Under such conditions, the interface quality becomes crucial for device functioning. Most common imperfections are related to atomic intermixing during growth, which on this scale is difficult to characterize by electron microscopy methods. In this paper, we show how atomic intermixing levels can be extracted from high-resolution X-ray diffraction (HRXRD) data. For this purpose, we develop fast algorithm, which is based on analyzing the positions of diffraction peaks originated from the substrate and superlattice layers. High-resolution transmission electron microscopy (HRTEM) and scanning tunneling microscopy (STM) are applied as complementary characterization methods in order to estimate the number of atomic planes constituting different sub-layers. HRTEM and STM data are then used as an input for HRXRD analysis.

12:05 PM Invited
Wafer Curvature and Stress Measured in-situ for Sputtered WSi2/Si Multilayer Thin Films on Silicon Wafers: K. MacArthur[1]; B. Shi[1]; R. Conley[1]; *Albert Macrander*[1]; A. Genis[2]; L. Zhou[3]; Y-P, Wang[3]; H. Zhou[3]; M. Li[3]; R. Headrick[3]; [1]Argonne National Laboratory; [2]Northern Illinois University; [3]University of Vermont

Wafer curvature was measured in-situ in a magnetron sputtering chamber at Argonne National Laboratory as a function of the pressure of the Ar plasma. Pressures between 2.3 mTorr and 18 mTorr were studied to investigate the trade-off between interface roughness [Y-P Wang et al., J. Appl. Phys. 101, 023503 (2007)] and stress in choosing the optimal pressure for growing thick multilayer stacks. The results support conclusions of in-situ x-ray diffuse scattering experiments at beamline X21 at NSLS at Brookhaven National Lab. The x-ray diffuse scattering measurements lead to the conclusion that there is a transition from deposition of atoms at low pressure to deposition of nanoparticles at high pressure [L. Zhou et al., Phys. Rev. B 82, 075408 (2010)]. Work supported by U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under contracts DE-AC02-06CH11357 and DE-AC02-98CH10886, and under Department of Energy Grant DE-FG02-07ER46380.

12:25 PM Invited
Understanding Structural Effects upon Macroscopic Phenomena in Strained Ordered Oxide Films: *Philip Ryan*[1]; Jong Woo Kim[1]; Steve May[2]; Evguenia Karapetrova[1]; [1]Argonne National Laboratory; [2]Drexel University

The plethora of rapidly emerging high quality epitaxial, strained complex ordered oxide films have demonstrated several macroscopic phenomena including magnetism, metal-insulator transitions and multi-ferroicity whereby strain induces or changes the respective phenomenon. Understanding the coupling between the structure, including spin and charge, and the macro-characteristic is examined using synchrotron based resonant x-ray diffraction. In this talk I will present examples of recent measurements on strained nickelate(M-I transition), manganate (Enhanced TN) and titinate (Multiferoicity) films. The results will demonstrate the invaluable information that standardized albeit advanced x-ray scattering techniques provide for emerging novel ordered oxide film systems.

12:45 PM Invited
Correlation Phenomena in Wurtzite-type GaN-based and ZnO Epilayers: *Alois Krost*[1]; Juergen Blaesing[1]; [1]Otto-von-Guericke University Magdeburg

In this contribution we report on extremely sharp x-ray peaks which very often appear in transverse omega scans of, e.g. InAlN, AlGaN, or ZnO thin films. Such correlation peaks are still visible in (0002) omega scans of 500 nm GaN which might mislead an observer to conclude incorrectly that there exists an extremely high structural quality. As one source of correlation peaks a high density of spatially correlated edge-type dislocations with in-plane Burgers vector and associated stacking faults could be identified. Another source might be surface steps on the sapphire substrate.

Pb-Free Solders and Other Materials for Emerging Interconnect and Packaging Technologies: Next Generation Packaging

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Indranath Dutta, Washington State University; Darrel Frear, Freescale Semiconductor; Sung Kang, IBM; Eric Cotts, SUNY Binghamton; Laura Turbini, Research in Motion; Rajen Sidhu, Intel Corporation; John Osenbach, LSI Corporation; Albert Wu, National Central Univ, Taiwan; Tae-Kyu Lee, Cisco Systems

Monday AM
February 28, 2011
Room: 7B
Location: San Diego Conv. Ctr

Session Chairs: Indranath Dutta, Washington State University; Darrel Frear, Freescale Semiconductor

8:30 AM Introductory Comments

8:35 AM Keynote
Creation and Manipulation of Aligned Nanowires for Packaging and Circuit Integration: *Sungho Jin*[1]; [1]University of California San Diego

Control of the nanowire structure is essential for their successful electronics applications including advanced solder connections and other packaging. Some electronic consumer products require protection against water-related damages through packaged structures, for which a superhydrophobic surface can be useful. Ceramic or metallic superhydrophobic coatings are more desirable than polymer coatings for long term, wear-resistant performances. In this talk, novel fabrication approaches to create vertically aligned nanowire configurations will be discussed, some of which exhibit superhydrophobic, non-wetting surface properties. Well controlled and processed vertically aligned semiconductor nanowire arrays can also be useful for solar cells and vertical nanotransistor arrays. Planarization of such periodically-arranged nanowire structures will be discussed in relation to circuit interconnection and routing of hundreds of millions of vertical nanodevices as well as for construction of three-dimensional multilayer device configurations.

9:15 AM Keynote
All Copper Flip-Chip Interconnect: *Paul Kohl*[1]; Hyo-Chol Koo[1]; Ping Nicole An[1]; Rajarshi Saha[1]; [1]Georgia Institute of Technology

A novel flip-chip technology using electroless copper deposition has been used to produce all-copper, chip-to-substrate interconnect. This process replaces solder in flip-chip connections by electrolessly joining copper pillars or pads between the chip and substrate. Solder has many weaknesses such as the formation of brittle intermetallics and poor electromigration resistance. The electroless copper joints formed in this project are annealed after plating to improve the copper bond strength. The process parameters and bond strength were investigated as a function of pillar diameter, and offset distance between the chip and its substrate. The composition of the plating bath determines the bond strength of the copper joints. Plating bath additives, such as surfactants, accelerators, and suppressors influence the quality of electroless joint. A finite element model was developed to understand the stress distribution within the copper pillar and joined components.

9:55 AM Invited

Plasticity and Reliability: From Unexpected Plasticity-Induced Damages in Advanced Cu Interconnects to Novel Reliability Phenomena in 3-D Interconnect Schemes Using Through-Silicon Vias (TSV) Technology: *Arief Budiman*[1]; Rao Morusupalli[2]; Tae-Kyu Lee[2]; Yu-Lin Shen[3]; Sung-Hwan Hwang[4]; Byoung-Joon Kim[4]; Ho-Young Son[5]; Min-Suk Suh[5]; Qwan-Ho Chung[5]; Kwang-Yoo Byun[5]; Martin Kunz[6]; Nobumichi Tamura[6]; Young-Chang Joo[4]; [1]Los Alamos National Laboratory; [2]Cisco Systems, Inc.; [3]University of New Mexico; [4]Seoul National University; [5]Hynix Semiconductor Inc.; [6]Lawrence Berkeley National Laboratory

Plastic deformation plays an important role in the control of mechanical properties and reliability of advanced microelectronics devices. Studying how materials evolve during the service of the devices will be crucial in understanding the degradation mechanisms leading to the eventual failures of the devices. In an effort to shed light on these topics, experiments using in situ synchrotron-based white-beam (Laue) x-ray microdiffraction technique to measure plasticity and mechanical stresses in the devices during their real and/or accelerated service conditions were conducted. In this talk, I will highlight two cases – advanced Cu interconnects and a 3-D interconnect scheme using TSV technology, in which this technique has been utilized to reveal degradation mechanisms that could lead to eventual failures. Our findings in these two cases further underline the importance of both measuring and controlling plasticity and stresses in the technology development as well as reliability improvement of advanced interconnect schemes.

10:20 AM Invited

Residual Stress of Si near Through-Silicon-Via Structure for 3-Dimensional Packaging: *Young-Chang Joo*[1]; Arief Budiman[2]; Sung-Hwan Hwang[1]; Byoung-Joon Kim[1]; Ho-Young Son[3]; Min-Suk Suh[3]; Kwang-Yoo Byun[3]; Nobumichi Tamura[4]; Martin Kunz[4]; [1]Seoul National University; [2]Los Alamos National Laboratory; [3]Hynix Semiconductor Inc.; [4]Lawrence Berkeley National Laboratory

Through-silicon via technology is widely used for 3-dimensional integration. However, stress arise in Si near TSV due to the difference in the coefficient of thermal expansion of Si and Cu. Devices cannot be located in the strained Si region (keep-away zone) due to mobility change. The plastic deformation of Cu during thermal load is the cause of the residual stress of Si, so comparing the stresses of Si and Cu is important in understanding the origin and size of keep-away zone. We used the synchrotron white beam X-ray to measure stresses of Cu and Si in TSV. Finite element method simulation was also conducted to analyze the keep-away zone. Keep-away zone was about 30 μm away from edge of TSV. By the comparison of the evolution of Cu and Si stresses and Cu grain structure during annealing, the cause of Si residual stress is discussed.

10:45 AM Break

10:55 AM Invited

The Effect of Filler-Network Heterogeneity on the Thermal Resistance of Polymeric Thermal Bondlines: *David Rae*[1]; Peter Borgesen[2]; Eric Cotts[2]; [1]Universal Instruments; [2]Binghamton University

The influence of processing on filled-polymeric bondline thermal performance was examined, with a focus on factors that promote local filler entrapment and compaction. The materials studied were viscously applied, adhesive thermal interface materials with dispersed filler particles. All stages of bondline assembly were controlled in an attempt to create high density regions of compacted filler particles. These included material deposition (initial material location andthickness), bondline formation (normal forces, assembly speeds, assembly temperatures), and post-formation bondline stabilization (force during cure). Following cure, bondline thermal resistances and microstructures were characterized. For one high-performing, commercial thermal interface material, a four-fold decrease in thermal resistance was achieved. The general response of filled-polymeric thermal interface materials to such processing is under investigation.

11:20 AM Invited

Recent Development of Lead-Free Nano-Solders for Nanowire and Nanoelectronics Assembly: *Zhiyong Gu*[1]; [1]University of Massachusetts Lowell

Many new nanomaterials and nanostructures have been fabricated in the past twenty years. With the dramatic development of nanotechnology in the past decade, promise has been shown in many potential applications of nanomaterials, including nanoelectronics, nanosensors, nanophotonics, and nanomedicine. However, in order to realize the manufacturing of nanoelectronic devices and systems, efficient and effective integration strategies have to be developed. In this talk, I will present our recent effort in the synthesis of lead-free nano-solders and the development of "nano-soldering" techniques. We have used the electrodeposition method in nanoporous templates to synthesize multisegmented nanowires with nano-solders, with diameter of 15-200 nm and length up 20 μm. Self-assembly method, such as magnetic assembly or dielectrophoretic (DEP) assembly, has been utilized to integrate nanowires into large-scale 2D or 3D network structures with conductive solder joints. These nanowires and nano-soldering techniques have great potential in the assembly and integration of functional nanoelectronics devices.

11:45 AM

Deformation and Interfacial Effects during Thermal Cycling of Cu Filled Through-Silicon Vias (TSV): *Praveen Kumar*[1]; Indranath Dutta[1]; Muhannad Bakir[2]; [1]Washington State University; [2]Georgia Institute of Technology

Large shear stresses develop at interfaces between dissimilar materials during thermal excursions when there is a significant difference in the coefficient of thermal expansion between them. In TSVs, these interfacial shear stresses prevail over a larger fraction of via-length as the chip thickness becomes smaller. Under thermal cycling conditions, the interface may slide via diffusional process, thereby accommodating relative dimensional changes between via-fill(Cu) and Si. This phenomenon can become a significant reliability issue in 3-D interconnect architectures involving TSVs. Here we present experimental evidence of interfacial sliding between Cu and Si in Cu filled TSV. TSV samples were thermally cycled under different conditions (temperature ranges and heating/cooling rates). Under rapid cycling conditions, evidence of interfacial fracture was found, whereas significant diffusional sliding was found to occur at the Cu-Si interface during slow cycling. A simple 1-D analytical model and associated numerical simulations will be presented to rationalize the experimental observations.

12:05 PM

Adhesive Selection and Bonding Parameter Optimization for Hybrid Bonding in 3D Integration: *Kuan-Neng Chen*[1]; Chuan-An Cheng[1]; Wen-Chun Huang[1]; Cheng-Ta Ko[1]; [1]National Chiao Tung University

Bonding technology is one of the key technologies for 3D integration in future packaging and semiconductor applications. Bonding temperature optimization of several polymer materials for metal/adhesive hybrid bonding was investigated. In general, the strong bond quality can be achieved with the increase of bonding temperature. However, when the bonding temperature reaches to certain point, bond failure of adhesive wafers were observed. In this research, several analysis techniques were used to evaluate the evolution of adhesive properties during the bonding process. Mechanisms are proposed to explain the failure of each adhesive. This study further conducts the criteria of adhesive selection for hybrid bonding in different 3D-IC applications.

Physical and Mechanical Metallurgy of Shape Memory Alloys for Actuator Applications: Characterization of Shape Memory Alloys: Deformation Behavior

Sponsored by: The Minerals, Metals and Materials Society
Program Organizers: S. Raj, NASA Glenn Research Center; Raj Vaidyanathan, University of Central Florida; Ibrahim Karaman, Texas A&M University; Ronald Noebe, NASA Glenn Research Center; Frederick Calkins, The Boeing Company; Shuichi Miyazaki, Institute of Materials Science, University of Tsukuba

Monday AM
February 28, 2011

Room: 11B
Location: San Diego Conv. Ctr

Session Chairs: Ronald Noebe, NASA Glenn Research Center; Raj Vaidyanathan, University of Central Florida

8:30 AM Welcome and Introductory Remarks

8:35 AM Plenary
Recent Development of High Temperature Shape Memory Alloys for Actuator Applications: *Shuichi Miyazaki*[1]; Hee Young Kim[1]; [1]University of Tsukuba

Ti-Ni alloys have been extensively developed to expand their application fields by adding new characteristics, shapes and dimensions, e.g., small transformation temperature or stress hysteresis, high transformation temperatures, porous structures, tubes, fine wires and thin films. Among them, some high temperature shape memory alloys have been developed: e.g., Ti-Ni-X (X=Au, Pd, Pt, Hf, Zr). The transformation temperatures of these alloys reach up to 1273K. However, it is not easy to make fine wires or thin plates with these alloys because of their lack of ductility. Recently, deformable high temperature shape memory alloys such as Ti-Ni-Zr(Hf)-Nb, Ti-Ta-Al have been developed, and they can reveal SME at temperatures between 373K and 473K, where commercial Ti-Ni alloys can not reveal SME. They can be cold-rolled with reductions higher than 60% and 90%, respectively, without revealing final fracture. In this lecture, the development of the Ti-Ni-Zr(Hf)-Nb and Ti-Ta-Al alloys is to be presented.

9:05 AM Invited
Energetics of Plastic Deformation and Transformation in NiTi: *Huseyin Sehitoglu*[1]; Tawhid Ezaz[1]; [1]University of Illinois at Urbana-Champaign

The shape memory alloys are an important class of materials that undergo reversible transformations. We provide an overview of the plastic deformation and its consequences in NiTi class shape memory alloys. Initially, we are concerned with determining the energy barriers associated with twinning and slip in the B2 phase. The NiTi alloys are considerably more ductile compared to their B2 counterparts, which points to the occurrence of twinning in the B2 phase. We study the energetics of twinning via atomistic simulations and continuum theory. Then, we study twinning in the martensitic phase. The results should shed light into several issues. What are the approximate magnitudes of the energy barriers for different shears for twinning and slip? What new information can be obtained from simulations to design new shape memory alloys with higher levels of recoverable strains?

9:25 AM Invited
Deformation of the U-14at% Nb Shape Memory Alloy: Experiments and Modeling: *Robert Field*[1]; Carlos Tomé[1]; Rodney McCabe[1]; Amy Clarke[1]; Donald Brown[1]; Catherine Tupper[1]; [1]Los Alamos National Laboratory

U-14at%Nb is a shape memory effect (SME) alloy that undergoes deformation by the motion of complex twins and twin related lath boundaries up to the limit of SME deformation (~7%). In the SME regime a simple Bain strain model qualitatively predicts variant selection, texture development in polycrystalline samples, and stress-strain behavior as a function of parent phase orientation in single crystal micropillars. In the post-SME regime, unrecoverable deformation occurs by a combination of slip and twinning, with the first few percent of strain in tension apparently governed by a twin species specifically associated with the monoclinic distortion in the martensite phase. A review of the Bain strain model for SME deformation will be presented in conjunction with experimental data. In addition, results from modeling of post-SME behavior using the Visco-Plastic Self-Consistent (VPSC) model will be compared to experimental texture measurements.

9:45 AM Invited
Observations on the Deformation Characteristics of NiTi: *Santo Padula*[1]; [1]NASA Glenn Research Center

Transformation-induced plasticity (TRIP) and slip are among the more common explanations provided for the dimensional instability observed in shape memory actuators, but alternate mechanisms have been shown to be responsible for the evolution in macroscopic strain. Many conditions including altering the upper-cycle temperature (UCT), performing multiple thermal cycles under load or mechanically cycling the material have all been shown to create dimensional instability. Through in situ neutron diffraction experimentation, it has been shown that martensite texture evolution and retained martensite are the dominant mechanisms responsible for the observed evolutionary response. It has also been shown that the isothermal, cyclic deformation response of NiTi (in combined tension and compression) is different than that observed for traditional elastic-plastic materials. The aforementioned experimental observations will be discussed in relation to the limited role of plasticity in shape memory behavior.

10:05 AM Break

10:15 AM Invited
Modeling and Experimental Study of Simultaneous Creep, Plasticity and Transformation of High Temperature Shape Memory Alloys during Cyclic Actuation: Yves Chemisky[1]; Parikshith Kumar[2]; George Chatzigeorgiou[1]; Ibrahim Karaman[3]; *Dimitris C. Lagoudas*[1]; Glen Bigelow[4]; Ron Noebe[4]; [1]Texas A & M University, Aerospace Engineering; [2]Texas A & M University; [3]Texas A & M University, Mechanical Engineering; [4]NASA Glenn Research Center

High Temperature Shape Memory Alloys (HTSMAs) represent a class of Shape Memory Alloys (SMAs) with transformation temperatures greater than 100°C. As a consequence of their high transformation temperatures, the HTSMAs can be exposed to a temperature regime where creep behavior can occur simultaneously during the transformation. This effect has a critical impact on the actuation response, due to the generation of irrecoverable strains. The creep behavior of ternary NiTiPd and the interaction between transformation and creep occurring simultaneously has been recently studied and a thermodynamic model was developed to capture this interaction. However the experimental and modeling effort were limited to one actuation cycle. An extensive experimental effort on a NiTi30Pd was conducted in order to determine the cyclic actuation response of such HTSMAs. In addition to plastic strains during martensitic transformation, retained martensite due to thermomechanical cycling and irrecoverable strains due to primary and secondary creep

10:35 AM
Low Temperature Creep Behavior of Extruded Near-Stochiometric NiTi Alloy: *S. Raj*[1]; Ronald Noebe[1]; [1]NASA Glenn Research Center

It is expected that NiTi shape memory alloys (SMA) actuators would provide simple and light weight replacements for controlling chevron-shaped nozzles in aircraft engines to reduce jet engine noise. An understanding of the low temperature creep properties of NiTi is important in determining the design life of these SMA acutators. Long term constant load tensile creep tests lasting several months to about a year were conducted on hot-extruded NiTi specimens at room temperature, 373 and 473 K under initial applied stresses varying between 250 and 350 MPa. These temperatures correspond to the martensitic, two-phase and the austenitic phase fields, respectively. The range of stresses corresponded to the region of the stress-strain curves for the alloy, where detwinning of the martensitic structure occurs. The low temperature creep behavior of NiTi are discussed. The results of stress and temperature change experiments conducted between room temperature and 473 K are reported.

10:50 AM

In Situ Observations of Deformation and Fracture in Single Crystal NiTi: *Adam Creuziger*[1]; Laura Bartol[2]; Ken Gall[3]; Wendy Crone[2]; [1]National Institute of Standards and Technology; [2]University of Wisconsin-Madison; [3]Georgia Insititute of Technology

The pseudoelastic transformation and fracture behavior of two crystallographic orientations of a single crystal NiTi shape memory alloy (SMA) were investigated. Uniaxial tension and notched tension samples are considered with the tensile axes along the [100] and [111] directions. The phase transformation was observed using optical techniques in situ. For the uniaxial tension samples, martensite plates and Luders-band structures are observed. For the notched samples, transformation structures in the [100] sample appeared predominantly on the sides of the notch and crack tip with stable crack propagation. In the [111] notched samples transformation occurred directly ahead of the notch and unstable crack propagation is observed. An available work criterion is used to predict the location of the transformation, with good agreement to the experimental observations. The different fracture behavior of the two notched sample orientations is explained utilizing the available work calculations.

11:05 AM

NiTiHf High-Temperature Shape-Memory Alloys for near Term Applications: *Glen Bigelow*[1]; Sayed Saghaian[2]; Haluk Karaca[2]; Santo Padula[1]; Anita Garg[3]; Darrell Gaydosh[4]; Ronald Noebe[1]; Yuriy Chumlyakov[5]; [1]NASA Glenn Research Center; [2]University of Kentucky; [3]University of Toledo; [4]Ohio Aerospace Institute; [5]Siberian Physical Technical Institute

Although high-temperature shape-memory alloys (HTSMA) with useful transformation temperatures above 150ºC are highly desirable, there is a near-term need for alloys with transformation temperatures even slightly above what conventional binary NiTi alloys can provide. Such materials would find immediate on-wing and in-cabin applications in the aeronautics sector, and would be equally utilized in the automotive and other commercial sectors. Polycrystalline and single crystal ([001], [-340], and [678] orientations) Ni50.3Ti29.7Hf20 with an MF ~130ºC was investigated using isothermal compression tests in the martensite and austenite states and load-biased thermal cycling to determine its viability in the aforementioned applications. Unlike conventional SMA actuator materials, the NiTiHf alloy investigated here exhibited high work output, good dimensional stability, and varying amounts of superelasticity, without the need for complicated thermomechanical processing. Results of the polycrystalline and single crystal tests will be compared and reasons discussed for the inherent dimensional stability of the polycrystalline material.

11:20 AM

Thermo-Mechanical Behavior of Cu-Al-Ni SMA for High Temperature Actuators: *Jose San Juan*[1]; Iñaki Lopez-Ferreño[1]; Tomasz Breczewski[1]; Isabel Ruiz-Larrea[1]; Angel López-Echarri[1]; María Nó[1]; [1]Universidad del Pais Vasco

Among the different families of shape memory alloys, Cu-Al-Ni alloys are firm candidates to be used in the temperature range between 100ºC to 200ºC as SMA actuators. In the present work we have studied the thermo-mechanical properties of several alloys exhibiting the martensitic transformation in such temperature range 100ºC-200ºC. Both, superelastic and shape memory effects have been characterized, as well as the evolution of the transformation behavior with thermal treatments and transformation cycling. The thermal transformation behavior under stress has been also studied in order to obtain a precise determination of the Clausius-Clapeyron stress line, and the shape recovery under stress. Finally we discuss the micro-structural design of Cu-Al-Ni SMA and the different measured properties, in terms of their capability to be used in real high temperature actuators.

11:35 AM

Microstructure Analysis and Thermomechanical Behaviour during Superlastic Cycling in Cu-Al-Ni Single Crystals: *Maria Nó*[1]; A. Ibarra[1]; A. López-Echarri[1]; I. Ruiz-Larrea[1]; D. Caillard[2]; J. San Juan[2]; [1]Universidad del Pais Vasco; [2]CEMES-CNRS

The aim of this work is to establish the relationship between the macroscopic behaviour during tensile superelastic cycling in Cu-Al-Ni shape memory alloy single crystals and the evolution of the microstructure. The analysis has been carried out as a function of the temperature, the maximum reached deformation and the number of cycles. Tensile superelastic cycles were performed in two samples with different compositions, which themally transform to different kinds of martensite. A calorimetric study and a TEM (post mortem and in situ) analysis were carried out on these superelastic cycled samples in order to determine the evolution of thermal transformation. Finally the results obtained by tensile superelastic cycling are discussed and compared with the previously obtained results on the same single crystal alloys by compression superelastic tests.

11:50 AM

Superelastic Phenomena and Martensite Destabilization in Ni-Mn-Ga Alloys: *Volodymyr Chernenko*[1]; E. Villa[2]; S. Besseghini[3]; J.M. Barandiaran[4]; V. A. L'vov[5]; [1]Universidad del Pais Vasco UPV/EHU - and - Ikerbasque, Basque Foundation for Science; [2]CNR-IENI, C.Promessi Sposi; [3]CNR-IENI, C. Promessi Sposi; [4]Universidad del País Vasco; [5]Institute of Magnetism

Thermomechanical properties of two Ni-Mn-Ga single crystalline alloys with MT temperatures of 310 K (A1) and 620 K (A2) are studied. DMA Q800 analyzer made it possible to register, from the same sample, temperature dependencies of elastic modulus in the dynamic tensile mode, stress versus strain curves at constant temperatures, and strain versus temperature curves at zero stress in static tensile mode. A multistep superelasticity of about 10% and one-step superelasticity of about 7% have been found in alloys A1 and A2, respectively. The stress – temperature phase diagrams of MTs are constructed. The stress–strain curves for alloy A2 demonstrate the gradual elevation of plateau as a result of mechanical cycling through MT. Theoretical estimations showed that this effect may be due to a decrease of MT temperature caused by the mechanical cycling. In correspondence to the commonly used terminology this effect should be referred to as martensite destabilization.

Polycrystal Modelling with Experimental Integration: A Symposium Honoring Carlos Tome: Emerging Polycrystal Models with Experimental Integration I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, ASM-MSCTS: Texture and Anisotropy Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Ricardo Lebensohn, Los Alamos National Laboratory; Sean Agnew, University of Virginia; Mark Daymond, Queens's University

Monday AM — Room: 6C
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Ricardo Lebensohn, Los Alamos National Laboratory; Alain Molinari, Universite Paul Verlaine - Metz; Grethe Winther, RISOE National Laboratory

8:30 AM Introductory Comments

8:40 AM Keynote

Modeling of Polycrystal Plasticity: A Personal Overview of Past, Present and Future: *Carlos Tome*[1]; [1]Los Alamos National Laboratory

In this talk I review, from my personal experience, the advances made in the last 25 years in the areas of crystal and polycrystal plasticity. I will

emphasize how advances in experimental methods and in computational capabilities have motivated and accompanied the development of increasingly sophisticated models and increasingly physically-based constitutive descriptions. The temporal line of research spans from simple Taylor models of aggregates in the past, to sophisticated polycrystal models that account for microstructural evolution (dislocations, twins, grain size, grain boundaries) in the present, and ambitious, but at the moment incipient, scale-linking approaches in the future. The theoretical developments have been aided by phenomenal advances on material characterization techniques, such as automated EBSD, FIB sectioning, X-ray and neutron diffraction, in-situ TEM.

9:25 AM Invited
First Evaluation of ALAMEL - Predictions of Texture-Induced Plastic Anisotropy: *Paul Van Houtte*[1]; Jurij Sidor[2]; Xie Qingge[1]; Laurent Delannay[3]; Bert Van Bael[1]; Leo Kestens[2]; [1]Katholieke Universiteit Leuven; [2]Ghent University; [3]Université Catholique de Louvain

Texture-induced plastic anisotropy is usually assessed by means of mechanical testing, for example by measuring r-values. It can also be predicted by a multi-scale model for the plastic deformation of polycrystalline materials. Before using such virtual test data to identify an anisotropic constitutive law to be implemented in FE simulations of metal forming processes, it is recommended to make a serious assessment of the multi-scale model used. Best known model for this application is the Taylor model, experimental validation having been published. However, several other, more advanced models exist which could also be used. One of these is the ALAMEL model, known to be successful in the prediction of deformation textures. So far, no systematic experimental validations of plastic anisotropy modeling using ALAMEL have been published. In this paper, a first series of such results will be given, as well as comparisons with the results of some other models.

9:50 AM
Microstructural Influences on the Local and Global Deformation of TiAl Polycrystal Investigated by Experiments and Numerical Multi-Scale Approach Incorporating Crystal Plasticity FE Model: *Mohammad Rizviul Kabir*[1]; Liudmila Chernova[1]; Marion Bartsch[1]; [1]German Aerosapce Center (DLR)

The influences of the microstructrual heterogeneity on the local and global mechanical behavior of a TiAl polycrystal have been investigated with experiments and numerical modeling. Two types of microstructure, duplex and near-lamellar, which are classified according to the presence of globular grains (consists of \947-phases) and lamellar colonies (consists of $\945_2$+\947-phases), have been studied. The representative microstructures were calculated from SEM and TEM analysis and were incorporated in a two-scale FE model. The micromechanics of deformation of the phases are described in a crystal plasticity FE model. With this polycrystal modeling approach microstructure dependent alloy properties were reliably predicted, with focus on the influence of the microstructural heterogeneity on the elastic properties at grain scale, anisotropic dependency of the material yield and contribution of dislocation-slip of the phases to the plastic deformation at local and global scale.

10:10 AM Break

10:20 AM Invited
Elastic Viscoplastic Heterogeneous Materials: From the Inclusion Problem to Homogenization Schemes: *Alain Molinari*[1]; Sébastien Mercier[1]; [1]Université Paul Verlaine-Metz

Micro-macro transition schemes contain two important steps. First, the localization process must link the fields inside the inclusion to those applied at the remote boundary. Secondly, an averaging process is used to derive the macroscopic behaviour. Molinari et al. (1987) have developed the tangent model where the non-linear viscoplastic behavior of the material is approximated by a first order Taylor expansion at the overall strain rate. This model is dedicated to rigid viscoplastic material. To account for elasticity, a simple interaction law has been proposed based on the tangent approximation of the non-linear Eshelby problem. This interaction law has been validated using Finite Element calculations and is used to develop Mori-Tanaka and self consistent schemes, Mercier and Molinari, (2009). Applications are shown for cyclic loading of composite materials.

10:45 AM Invited
On New Intermediate Modeling for the Large Viscoplastic and Elastic-Viscoplastic Deformation Behavior of Polycrystals: The Intermediate Phi-Model: *Said Ahzi*[1]; Siham M'Guil[1]; [1]University of Strasbourg

In a first part of the presentation, we discuss the problem of homogenization of polycrystalline metals. Then, we present the new homogenization technique leading the formulation of the viscoplastic intermediate phi-model for crystal plasticity. With the phi-model, as in the self-consistent approach, a non-linear interactions law is derived but without the call for the Eshelby tensor. Thus, the phi-model takes into account the grain interactions with the homogeneous equivalent medium but without explicit shape effects. In a second part of the presentation, we present the extension of the phi-model to large elastic-viscoplastic deformation. Finally, we compare the results predicted from our new modeling, in terms of stress-strain response, textures, slip activity, …, to those of the viscoplastic self-consistent model as well as to the experimental results.

11:10 AM Invited
Insights into Microstructure Evolution Using Crystal Plasticity Modelling: *Matthew Barnett*[1]; [1]Deakin University

Crystal plasticity ignores certain mechanisms of microstructure evolution. However, it can shed important light on the operation of these very mechanisms. The present talk examines four attempts to use crystal plasticity models in this manner. Taylor type modelling is employed to examine the generation of high angle boundaries in shear. Self consistent type modelling is employed to reveal the abnormal formation of secondary twins, the possible role of grain boundary sliding and the formation of dynamically recrystallized grains. Although these uses of the models push the limits of what they were designed to achieve, important insight can be gained into how new structures are generated during plastic deformation.

11:35 AM Invited
Effects of Crystallographic Orientation vs. Grain Interaction on Slip Systems: *Grethe Winther*[1]; [1]Risø DTU

When studying the deformation behaviour of individual grains both the overall rotation of the crystallographic grain orientation and the subdivision of grains are of interest. Often grains of similar orientations are compared to study the effect of grain orientation vs. grain interaction. Such investigations reveal both similarities and differences. The present contribution gives an overview of a series of investigations, including transmission electron microscopy as well as three-dimensional x-ray diffraction on polycrystalline aluminium deformed to strains of 5-50%. The data are analysed focusing on the set of activated slip systems, more precisely whether the observed differences can be attributed to fluctuations in the relative activities of the same set of systems or whether activation of truly different systems is the origin of the variations between and within grains.

12:00 PM
VPSC Modelling in the Development of a Commercial Product: *Dincer Bozkaya*[1]; Peter Jepson[1]; [1]H.C. Starck Inc.

Texture Control is very important for planar sputtering targets, since the sputtering rate of a grain depends on its orientation. When tantalum is rolled to plate using conventional methods, the texture developed at mid-thickness is different from that developed near the surface because of differences in shear strain. At H.C. Starck, we used an FEM code and VPSC together to create models and thereby speed up the development of a product with no through-thickness texture gradient. The development of the new rolling process will be described, with emphasis on how VPSC was used and its usefulness.

12:20 PM

Modelling of Texture and Microstructure Evolution during Dynamic Recrystallization: *Denis Solas*[1]; Julien Thebault[2]; Julien de Jaeger[2]; Colette Rey[2]; Thierry Baudin[1]; [1]Univ Paris Sud; [2]Ecole Centrale Paris

The changes in texture and microstructure during thermomechanical process are related to crystal plasticity and annealing phenomena. Numerical studies has been carried out in which a crystalline plasticity modelling implemented in the finite element code Abaqus® coupled to a recrystallization Cellular automaton code. A sequential approach is used to simulate hot forging of a nickel based superalloy. Modelling is performed on aggregates built up from EBSD maps. Each grain is decomposed into smaller domains. In particular, the deformation model predicts intragranular deformation in terms of misorientations between elements and local variations in stored energy. A cellular Automaton algorithm is used for simulating nucleation and grain boundary migration. The parameters used for the sequential modelling were identified using an inverse method from experimental results.This numerical and experimental approach is used to understand the experimental observations and it improves our knowledge of the recrystallization process.

12:40 PM

A Physically-Based Fatigue Model for Prediction of Crack Initiation from Persistent Slip Bands in Polycrystals: *Huseyin Sehitoglu*[1]; Michael Sangid[1]; [1]University of Illinois

A model is constructed for prediction of fatigue crack initiation based on the material's microstructure. Our approach is to model the energy of a persistent slip band (PSB) structure and use its stability with respect to dislocation motion as our failure criterion for fatigue crack initiation. The components that contribute to the energy of the PSB are identified, namely, the stress field resulting from the applied external forces, dislocation pile-ups, and work-hardening of the material is calculated at the continuum scale. Further, energies for dislocations creating slip in the matrix/precipitates, interacting with the GBs, and nucleating/agglomerating within the PSB are computed via atomistics. The predicted fatigue life is driven by the microstructure such as grain orientations, widely distributed grain sizes, precipitates, PSB-GB interactions, as well as the effect of neighboring grains. Good agreement is shown between the model predictions and experimental data.

Recent Developments in the Processing, Characterization, Properties and Performance of Metal Matrix Composites: General and Nano-Composites

Sponsored by: The Minerals, Metals and Materials Society
Program Organizers: Martin Pech-Canul, Centro de Investigacion y de Estudios Avanzados del Instituto Politecnico Nacional; Zariff Chaudhury, Arkansas State University; Golam Newaz, Wayne State University

Monday AM — Room: 6A
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chair: Zariff Chaudhury, Arkansas State University

8:30 AM

Al/Al-Cu-Fe Metal Matrix Composites: *Guillaume Laplanche*[1]; Joël Bonneville[1]; Anne Joulain[1]; Véronique Brunet-Gauthier[1]; Sylvain Dubois[1]; [1]University of Poitiers

The mechanical properties of icosahedral quasicrystalline (i-QC) Al-Cu-Fe alloys make them very attractive as reinforcement particles for metallic matrix composites (MMC). Al-base composites reinforced by i-QC Al-Cu-Fe particles have been synthesised since nearly two decades, but their mechanical properties are still poorly known. In this work, Al/Al-Cu-Fe MMCs were produced at two temperatures, i.e. T=673K and T=873K, by hot isostatic pressing. Crystallographic and chemical composition investigations were performed on the as-synthesised MMCs for identifying the resulting phases. At T=673K, the reinforcement particles are still of the original i-QC phase, whereas at T=873K a phase transformation occurs for the reinforcement particles, which have the ω-Al-Cu-Fe tetragonal crystalline structure. The flow stress of both composites is strongly temperature dependent, suggesting that thermally activated processes control plastic flow. Transmission electron microscopy observations suggest that micro- and nano-particles play different roles in the strengthening of the two composites.

8:50 AM

Copper Matrix Composites Reinforced with a High Fraction of Alumina Particles: *Carmen Krueger*[1]; Andreas Mortensen[1]; [1]EPFL STI IMX LMM

The aim of this project is to explore the production of copper-based composites containing a high volume fraction of ceramic particles. Alumina is chosen as ceramic reinforcement phase due to its high Young's modulus, low density, chemical compatibilty with copper and high availability, in combination with matrices of aluminum bronze. Two processing routes are explored, namely (i) pressure infiltration, and (ii) in-situ reaction, the latter being based on the thermitic reduction of copper-oxides by aluminum. Success in the former method is shown to hinge on control of the structure and evolution of the metal/ceramic interface, while the latter is tributary to the rates of competing interdiffusion phenomena, as often found with in-situ composite production methods.

9:10 AM

Low Density Magnesium Matrix Syntactic Foams: *John DeFouw*[1]; Pradeep Rohatgi[1]; [1]University of Wisconsin Milwaukee

Low density magnesium matrix syntactic foams were synthesized through pressure infiltration of a packed bed of hollow carbon spheres with liquid magnesium of both pure and alloy (AZ91) compositions. Through varying the applied gas pressure for infiltration, the resulting composites varied in density as low pressures resulted in incomplete infiltration of regions between spheres and higher pressures resulted in infiltration into sphere interiors through cracks or small pores producing higher densities, ranging from 0.7 to 1.1 g cm^{-3} (40-60% relative density). The foams were studied under mechanical compression with modulus, strength, and energy absorption described in relation to foam density and heat treatment as well as microstructural observations of metal matrix fraction, hollow sphere porosity, incomplete infiltration porosity, and carbide formation.

9:30 AM

Metal Matrix Composites: History, Status, and Future: *Ajith Cyriac*[1]; Jay Hanan[1]; [1]Oklahoma State University

A study of the history, status, and opportunities of Metal Matrix Composites is presented by evaluating the progression of available literature. The trends that existed and issues that still prevail are discussed and a prediction of the future for MMCs is presented. In many developed countries and in several developing countries there exists continued interest in MMCs. Researchers tried numerous combinations of matrices and reinforcements since work on MMCs began in the 1950s. This led to developments for aerospace, but resultant commercial applications were limited. The introduction of ceramic whiskers as reinforcement and the development of in-situ eutectics in the 1960s aided high temperature applications in aircraft engines. In the late 1970s the automobile industries started to take MMCs seriously. In the last 20 years, MMCs evolved from laboratories to a class of materials with numerous applications and commercial markets. A summary histogram depicting the progression is presented.

9:50 AM

Processing, Characterization and Thermal Properties of Diamond-Containing Metal Matrix Composites: *Vikas Sinha*[1]; Sabyasachi Ganguli[1]; Robert Wheeler[1]; Jonathan Spowart[1]; [1]Air Force Research Laboratory

An ideal substrate/ packaging material for high power density electronic component should have a very high thermal conductivity and a coefficient of thermal expansion (CTE) comparable to the electronic component. Diamond-containing Ag-matrix and Cu-matrix composites potentially can be excellent solutions for thermal management. In the current research, diamond-containing Ag-matrix and Cu-matrix composites were fabricated

via spark-plasma-sintering or liquid-metal-infiltration techniques. Cu was pre-alloyed with Cr or B, whereas Ag was pre-alloyed with Si. The effects of different micro-alloying elements on thermal properties were compared. Thermal conductivity of composites was modeled to estimate thermal conductance for metal/diamond interfaces for different material and processing conditions. The CTE of composites were compared with model predictions. Prior studies indicate that the interface chemistry has a strong influence on thermal properties. The chemistry changes near the metal/diamond interfaces were examined via High-Resolution Transmission Electron Microscopy (HRTEM), using energy dispersive spectroscopy and energy-filtering techniques.

10:10 AM Break

10:30 AM

Use of Cenosphere for Making Functionally Graded Aluminum Cenosphere Syntactic Foam through Liquid Metallurgy Route: *Dehi Mondal*[1]; Satyabrata Das[1]; [1]Advanced Materials and Processes Research Institute

Attempts have been made to make functionally graded aluminum cenosphere syntactic foam wherein volume fraction of cenosphere varies along the thickness of the casting. Cenosphere has been used to make such microporous materials. The sample from different depths were examined for cenosphere volume fraction, density, hardness and compressive stress-strain curves and these properties are varying along the depth.The thickness of the layer corresponding to particular average cenosphere content and the average volume fraction of cenosphere in each layer was found to be strong function of processing parameters like melt temperature, cooling rate, number of heating and cooling cycles. As the frequency of heating and cooling cycles increases, the volume fracton of cenosphere at top most surface increases. Through this processe, thus a bulk dense material with functionally graded microporous syntactic foam on one side could be made where a strong bonding between the foam and bulk material will exist.

10:50 AM

Micro-Tomography Based Characterization and Geometrical Parameter Evaluation of Advanced Woven Ceramic-Matrix Composites: *Hrishikesh Bale*[1]; David Marshall[2]; Brian Cox[2]; Robert Ritchie[1]; [1]University of California Berkeley; [2]Teledyne Scientific LLC

Structural components operating under extreme environments (>1200°C) demand the use of a new class of advanced materials. Environments typically experienced in rocket engines, hypersonic flow paths and thermal protection systems are beyond the realm of current structural materials. However, woven ceramic-matrix composites (CMC's) incorporating textile manufacturing techniques utilizing 3-D architectures makes the development of such structures feasible. Lifetime prediction within CMC's is non-trivial though; microscopic flaws within fibres and matrix can be governing factors controlling the ultimate performance of these materials in service. Synchrotron micro-tomography was utilized to evaluate CMC's in 3-D with ~1-μm resolution. The use of micro-tomography in evaluating the otherwise difficult-to-measure structural parameters such as 3-D tow spacing, orientation, cross section and matrix porosity in SiC/SiC CMC's is demonstrated which facilitates the understanding of damage and failure. This would further lead in developing comprehensive and accurate constitutive laws for computational models.

11:10 AM

Synthesis and Characterization of Hot Extruded Al-15wt%Mg2Si In-Situ Composite: Ashkan Zolriasatein[1]; *Masoud Emamy*[2]; Amin Bahrami[3]; Hamid Reza Jafari Nodooshan[4]; Ali Shokuhfar[1]; [1]K. N. Toosi University of Technology; [2]Tehran University ; [3]Imam Khomeini International University; [4]Islamic Azad University

This study investigates the hot extrusion of Al-15wt%Mg2Si in-situ composite at various extrusion ratios (6:1, 12:1, and 18:1) to determine the relationship between the process parameters, microstructure and the properties of the extruded composite. Microstructure of the composite after this type of extrusion was studied by optical microscopy. Mechanical properties of the composite after various extrusions were tested by hardness and tensile tests. These results indicate that extrusion leads to reduction of porosity and particle fracture. With the increase of the extrusion ratio at a relatively low temperature, the microstructure of the extruded samples becomes finer and more uniform, and and higher mechanical properties can be brought for the as-extruded samples in comparison with as-cast samples.

11:30 AM

Thermal Conductivity and Interfacial Nanostructure in Diamond Based Composites: *F. Khalid*[1]; [1]GIK Institute of Engineering Sciences and Technology

Processing parameters is critical in achieving the interfacial nanostructure of Al/diamond and Cu/diamond composite materials. These composites are produced by sintering and pressurised liquid metal infiltration have the potential for thermal management Applications. Attempts have been made to develop new composites to improve thermal conductivity. In this work it is demonstrated that thermal conductivity of diamond based composites can be achieved up to 700 Wm/K. It is illustrated that the liquid metal infiltration is better production route for Al-diamond and Cu/diamond composites. The examination of the interfacial nanostructure of the composite samples by using advanced analytical techniques is carried out. Emphasis is also placed on the understanding of preferential interfacial bonding between matrix and diamond (100) and (111) surfaces. The formation of Al4C3 particles at the interface between Al and diamond particles was found that indicated a greater level of stability and bond strength between the diamond particles and matrix.

Size Effects in Mechanical Behavior: Size Dependent Mechanical Behavior of Nanotwinned and Nanocrystalline Metals

Sponsored by: The Minerals, Metals and Materials Society, Not Applicable, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Erica Lilleodden, GKSS Research Center; Amit Misra, Los Alamos National Laboratory; Thomas Buchheit, Sandia National Laboratories; Andrew Minor, UC Berkeley & LBL

Monday AM
February 28, 2011

Room: 2
Location: San Diego Conv. Ctr

Session Chairs: Erica Lilleodden, GKSS Research Center; Dominique Schryvers, University of Antwerp

8:30 AM

Study of Twins in Nanoscale Pd Films with High Strain Hardening Capacity: *Dominique Schryvers*[1]; Hosni Idrissi[1]; Binjie Wang[1]; Marie-Stéphane Colla[2]; Jean-Pierre Raskin[2]; Thomas Pardoen[2]; [1]University of Antwerp; [2]Université Catholique de Louvain

Nanocrystalline metallic films often suffer from a lack of ductility due to poor strain hardening capacity. Nanograin Pd thin films (80 - 310 nm film thickness) with very large strain hardening when deformed by on-chip nanomechanical tensile testing have been produced by low pressure chemical vapour deposition and photolithography. TEM and HRTEM reveal the presence of a few coherent growth twins in more than 20% of the grains, offering additional barriers to dislocation motion, as well as sources for dislocation storage and multiplication. The twin boundaries become incoherent after a few percent of plastic deformation. The related dislocation/twin boundary reactions explain part of the high strain hardening capacity, the remaining resulting from microstructure heterogeneities with softer and harder grains, leading to a long elastoplastic transition. These two aspects constitute the main ingredients of a multigrain homogenization model which provides a rational for the evolution of the strain hardening capacity.

8:50 AM
Effects of Differently Oriented Twin Boundaries on Mechanical Properties in Nanotwinned Ag Films: Daniel Bufford[1]; *Xinghang Zhang*[1]; Haiyan Wang[1]; [1]Texas A&M University

Epitaxial nanotwinned Ag (111) and Ag (110) films were deposited by magnetron sputtering. Although both films were deposited with similar deposition parameters, the twin boundary spacing is 9 nm in Ag (111) and 42 nm in Ag (110). Twin boundaries in the Ag (111) films are oriented normal to the growth direction and at an angle in the Ag (110) films. The fine twins greatly enhance the indentation hardness of both films, and there is a factor of two differences in hardness between the (111) and (110) Ag films. The mechanisms for twin formation and the influences of twin boundary orientation on mechanical deformation will be discussed.

9:10 AM Invited
Inverse Grain-Size Effect on Twinning in Nanocrystalline FCC Metals: *Yuntian Zhu*[1]; Xiaolei Wu[2]; [1]North Carolina State University; [2]Chinese Academy of Sciences

A long-standing controversy exists between molecular dynamics (MD) simulations and experiments on the twinning propensity of nanocrystalline (nc) face-centered-cubic metals. For example, three-dimensional MD simulations rarely observed twins in nc Ni, whereas experiments readily observed them. Here this discrepancy is resolved by experimental observation of an inverse grain-size effect on twinning. Specifically, decreasing the grain size first promotes twinning in nc Ni and then hinders twinning due to the inverse grain-size effect. Interestingly, no inverse grain-size effect exists on stacking fault formation. These observations are explained by generalized planar fault energies and grain-size effect on partial emissions.

9:40 AM
A Twist of the Eshelby Twist: Unraveling the Mystery of Twinning: *Ting Zhu*[1]; Ju Li[2]; Sankar Narayanan[1]; [1]Georgia Institute of Technology; [2]University of Pennsylvania

The principal difficulty in understanding the deformation mechanisms of small-volume crystals has been to explain how the plastic deformation carriers such as dislocations and twins are generated and multiplied. The sources of such carriers and associated triggering processes have not been well established. In 1953 Eshelby showed that the central region of a rod can act as a trap to stabilize the screw dislocation that, in turn, induces the torsional deformation of the rod. While studying this so-called Eshelby twist effect, we incidentally discovered a twinning mechanism in fcc Cu. We found that the axial screw can initiate the regenerative twinning through a 3D process of dislocation intersection followed by spiral gliding of a sweeping dislocation around the pole. Our work is the first atomistic realization of 3D twinning, and it opens up the possibilities of studying the experimentally relevant twinning mechanisms for a wide range of materials and applications.

10:00 AM Break

10:30 AM
Detwinning Mechanisms for Growth Twins in Epitaxial Nanotwinned Cu: *Nan Li*[1]; Jian Wang[1]; Xinghang Zhang[2]; Jianyu Huang[3]; Amit Misra[1]; [1]Los Alamos National Laboratory; [2]Texas A&M University; [3]Sandia National Laboratory

Using in situ nanoindentation in a transmission electron microscope (TEM), we have studied the migration mechanisms of S3 {112} incoherent twin boundary (ITB) in epitaxial nanotwinned Cu films. ITB's migrate via the collective glide of multiple twinning dislocations that form an ITB and the propagation steps or disconnections are with heights of three or multiples of three {111} interplanar distances. The migration may lead to de-twinning process of nanotwins in face-centered cubic metals. The transmission of a glide dislocation across an ITB is shown to form a sessile dislocation in the ITB and locally pin the boundary at the site of the slip transmission.

10:50 AM Invited
Deformation Effects in Cu with Highly Aligned Nanotwins: *Julia Weertman*[1]; Carla Shute[1]; [1]Northwestern University

Nanotwinned metals have been found, by the work of K. Lu, S. Suresh, and others, to be very strong, with stable microstructures and possessed of moderate ductility. The present research examines the effect of various forms of deformation on the internal structure, damage and crack nucleation in Cu with highly aligned columns of nanotwins. It is found that surface fatigue cracks form at the intersection of regions where there is incompatibility in the deformation process, e.g. between a nanotwinned and de-nanotwinned region. High densities of dislocations form in such places. The median spacing of nanotwin boundaries in the samples is unchanged by fatigue, except in the close vicinity of cracks, where there is a substantial increase. TB spacing under extreme compression decreases in the amount expected from the negative strain. HPT experiments indicate the limit of stability of the nanotwins under shear strain.

11:20 AM
Fatigue Properties of Nano Structured Copper: *Aparna Singh*[1]; Lei Lu[2]; Ming Dao[1]; [1]MIT; [2]Chinese Academy of Sciences

Nano twinned materials exhibit a combination of both high strength and ductility unlike nano grained materials. However, their properties under cyclic loading have not been explored as yet. In this presentation, we outline our results of fatigue studies done on nano twinned materials, and the observations are compared with those for nano grained materials in order to get conclusions on the role of micro structural size scale in influencing the damage tolerance characteristics of metals. General strategies for improving strength, ductility, damage resistance and tolerance will be presented.

11:40 AM
Tensile Plastic Deformation of Gradient Nano-Grained Copper: W. L. Li[1]; N. R. Tao[1]; *K. Lu*[1]; [1]Chinese Academy of Sciences

Nanocrystalline or nano-grained metals are strong, but brittle. Experimental identification of plastic deformation mechanisms of nano-grained structures is challenging, indeed, because tensile plastic strain cannot be sustained in nano-grained samples which has basically no work-hardening. In this work, we prepared a unique sample for experimental study on the plastic deformation of nano-grained metals: a gradient nano-micro-grained (GN) surface layer on a bulk Cu specimen. The GN layer is produced by means of surface mechanical grinding treatment (SMGT) at cryogenic temperature. The GN samples exhibit a considerable plastic strain above 30% in tension, indicating that the nano-grained structure can be plastically deformed with a substantial tensile strain. Systematic structure characterization revealed that plastic deformation of the nano-grained structure is localized and accompanied with stress-induced grain growth. Discussions will be made on accommodation capability of plastic deformation in the GN structure in comparison with that of the polycrystalline coarse-grained structure.

Surfaces and Heterostructures at Nano- or Micro-Scale and Their Characterization, Properties, and Applications: Growth, Characterization, and Devices I

Sponsored by: TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Nanomaterials Committee, TMS: Surface Engineering Committee

Program Organizers: Nitin Chopra, The University of Alabama; Ramana Reddy, The University of Alabama; Jiyoung Kim, Univ of Texas; Arvind Agarwal, Florida International Univ; Sandip Harimkar, Oklahoma State University

Monday AM
February 28, 2011

Room: 31B
Location: San Diego Conv. Ctr

Session Chairs: Nitin Chopra, The University of Alabama; Ritesh Agarwal, University of Pennsylvania

8:30 AM Introductory Comments

8:35 AM Invited

Transforming Semiconductor Nanowires into Heterostructures and Superlattices by Size-Dependent Cation Exchange Reactions: *Ritesh Agarwal*[1]; [1]University of Pennsylvania

The unique properties of nanostructured materials enable their transformation into complex, kinetically-controlled morphologies which cannot be obtained otherwise. Solution-phase cation-exchange reactions can transform sub-10 nm nanocrystals/nanorods into varying compositions and superlattices; however, due to their small size, cation-exchange reaction rates are extremely fast which limits control over the transformed products. Nanowires are routinely synthesized via gas-phase reactions with 5-200 nm diameters and to realize their full potential, it is desirable to develop techniques which can transform nanowires into tunable morphologies. We report transformation of single-crystalline cadmium-sulfide nanowires into composition-controlled ZnxCd(1-x)S nanowires, core-shell heterostructures, metal-semiconductor superlattices, ZnS nanotubes, and metallic Zn nanowires by utilizing size-dependent cation-exchange reactions. Simulations that account for elastic interactions and diffusional kinetics reveal the conditions for forming these structures and the peculiarities of 1D systems. This versatile ability to transform nanowires offers new opportunities to study size-dependent phenomena and tune their chemical/physical properties to design reconfigurable circuits.

9:05 AM Invited

Does Function Follow Form? Coiled Carbon Nanotube and Nanowire Structures- Thermodynamic Model, Experiment, and Applications: *Prabhakar Bandaru*[1]; Apparao Rao[2]; [1]University of California San Diego; [2]Clemson University

New paradigms in materials and devices lead to new technologies, which are necessary for continued scientific progress. We have determined, in foundational studies, that nonlinear nanostructures, such as carbon nanotube based helices and coils, may be synthesized through the use of specific catalysts in chemical vapor deposition and could open up new vistas in electrical, mechanical, and optical engineering, e.g. as electrical inductors, nanoscale mechanical springs, and metamaterials. First, a thermodynamic model based on exclusion volume principles common in chemical and biological systems that could potentially explain coiling in nanostructures, will be introduced. The crucial role of the interactions between specific catalyst particles, e.g. indium and tin, and the growing nanostructure for coil growth, which has been verified through experiment, will be elucidated. The talk will be concluded with a brief exposition on the unique applications of these structures, say in localized energy absorption and electromagnetic radiation coupling.

9:35 AM Invited

Atom-by-Atom Characterization of Low-Dimensional Materials: *Stephen Pennycook*[1]; Matthew Chisholm[1]; Gerd Duscher[2]; Timothy Pennycook[3]; James McBride[3]; Sandra Rosenthal[3]; [1]Oak Ridge National Laboratory; [2]University of Tennessee; [3]Vanderbilt University

The aberration-corrected scanning transmission electron microscope offers a new level of sensitivity for revealing the origin of nanomaterials functionality through its ability to image and identify individual substitutional dopants and adatoms. Examples will be shown of O and C dopants in monolayer BN and of defects and adatoms in monolayer graphene. In the case of semiconducting nanocrystals, their size, shape and crystal termination can be identified with no lower size limit. Such images reveal the mechanisms behind the growth and luminescent properties of core/shell quantum dots and ultrasmall white light-emitting nanocrystals.

10:05 AM Invited

Micro and Nanostructured Porous Heterostructures by Electrodeposition: *Martin Bakker*[1]; Nikolas Cordes[1]; Franchessa Sayler Maddox[1]; Jan-Henrik Smatt[2]; Mika Linden[2]; [1]The University of Alabama; [2]Ako Akademi University

Electrodeposition into insulating templates is an extremely powerful method of making metal/insulator heterostructures. With careful selection of the electrochemistry, electrodeposition can also be used to deposit the insulating templates. We will present recent work on 1) electrodeposition of mesoporous silica thin films, and electrodeposition of micrometer structured mesoporous silica on planar and three dimensional substrates, 2) electrodeposition of metal into electrodeposited mesoporous silica templates 3) the use of humidity to control the electrodeposition of metals into the mesoporous of hierarchically meso-microstructured porous silica monoliths.

10:35 AM Break

10:45 AM Invited

Synthesis and Characterization of Silicon Oxide Nanowires Using Nickel Nanoparticles: *Seonhee Jang*[1]; Youngil Lee[1]; Suhwan Cho[1]; Jungwook Seo[1]; Donghoon Kim[1]; [1]Samsung Electro-Mechanics

We attempt to produce silicon oxide nanowires (SiOxNWs) in a simple way without complicated deposition processes, gaseous Si containing precursors, or starting material of SiO2. Nickel (Ni) nanoparticles (NPs) were applied on Si wafer and thermally treated in a furnace. The temperature in the furnace was kept in the ranges between 900 and 1100 □ and a forming gas, i.e. mixture of nitrogen (N2) and hydrogen (H2) flowed through the furnace. The growth of SiOxNWs was obtained via carbothermal reduction of NiO. The SiOxNWs had widths ranging from 100 to 200 nm with length extending up to ~ 10 μm and their structure was amorphous. Ni NPs were acted as catalysts. Photoluminescence (PL) showed that blue emission spectrum was centered at the wavelength of 450 nm (2.76 eV). The details of growth mechanism of SiOxNWs and the effect of Ni NPs on the formation of SiOxNWs will be presented.

11:15 AM

Novel Architectures of Hierarchical Heterostructures: Non-Catalytic Growth of ZnO Nanowires and Their Multifunctional Heterostructures with Gold Nanoparticles: *Wenwu Shi*[1]; Austin Starnes[1]; Nitin Chopra[1]; [1]The University of Alabama

Multi-functional heterostructures comprised of semiconducting nanowires and metal nanoparticles are of significance for optoelectronics and energy applications. Herein, for the first time, we report a simple and non-catalytic CVD process that produced branched ZnO nanowires (BZN). As a next step, gold nanoparticles were nucleated on the surface of BZN in a surfactant-free approach resulting in novel nanowire-nanoparticle heterostructures. Fundamental understanding of the heterostructures and their crystal structures, phases, interfaces, chemical functionality, composition, and thermal stability was developed using SEM, HR-TEM, XRD, DTA-TGA, FT-IR. The kinetics of heterostructure formation and their unique optical properties were studied in real-time. The growth mechanisms of BZN and their unique branched configuration have also been discussed here.

11:30 AM
Induced Chemical Changes in Ni/NiO Core Shell-Carbon Nanotube Heterostructures in a High Temperature Post-Fabrication Treatment: *Hylton McWhinney*[1]; Wenwu Shi[2]; Nitin Chopra[2]; [1]Texas A & M University; [2]The University of Alabama

High temperature surface migration of Ni/NiO core-shell nanoparticles (~5-10 nm) on the surface of carbon nanotubes (CNC Heterostructures, Shi, Krews, and Chopra, Materials Technology, 2010) was thoroughly studied. Heat treatments were carried out in a nitrogen-enriched furnace and the CNC heterostructures were carefully dispersed on a substrate. The samples were treated between 125 – 750 oC for 1 h. It was observed in SEM, TEM, and EDS analysis that nanoparticles significantly migrate on the CNT surfaces and their morphology was changed. In addition, massive agglomeration of nanoparticles was observed at higher annealing temperatures. XPS analysis of the heterostructures, as a function of their heat treatments, suggested that nanoparticle precursor solution, CNT surface, and annealing temperature played an important role in the morphological evolution of nanoparticles on CNTs. Shifts in the XPS elemental peaks revealed new critical information about the bonding characteristics of such heterostructures.

11:45 AM
Nanoparticle Cluster Size Control using Chemically Modified Self-Assembly on Copolymer Surfaces: *Sarah Adams*[1]; Regina Ragan[1]; [1]University of California Irvine

Organized clustering of metallic nanoparticles was examined for the development of field-enhanced chemical and biological detection devices, with the capacity to achieve single-molecule level detection resulting from surface enhanced Raman scattering, associated with closely-spaced noble metal nanostructures. Using chemical self-assembly techniques, we attached monodisperse, colloidal gold nanoparticles on self-organized polymer templates, patterning arrays of nanoparticle clusters with sub-10 nanometer interparticle spacing, to engineer enhanced optical fields. Poly(methyl methacrylate) domains in phase-separated polystyrene-*b*-poly(methyl methacrylate) diblock copolymer thin films were chemically modified with surface amination for controlled arrangements. Chemically synthesized sub-20 nanometer diameter gold nanoparticles were attached to the amine-functionalized surfaces using EDC crosslinking chemistry with thioctic acid ligand-bound to the nanoparticle surface, preferentially immobilizing the Au nanoparticles on PMMA domains of the copolymer template. By controlling particle size relative to PMMA domain size, nanoparticle clustering was controlled.

12:00 PM
Thermodynamics and Processing of Nanomaterials: *Ramana Reddy*[1]; [1]The University of Alabama

This presentation includes nanoscale materials thermodynamics and processing. Thermodynamic properties of nano-particles are different as compared to that of bulk material due to the surface energy changes. Melting temperature of nano-particles varies with the size (radius) of particles. This variation in melting temperature was calculated for different nanoscale materials. In-situ processing of nanomaterials has immense potential over the conventional nanoscale materials due to several advantages such as inherent chemical stability, lack of impurities, and fine dispersion. The results of thermal plasma production of nanoscale metal, ceramic and composite powders and the various process parameters that influence the final product were discussed.

12:15 PM
Magnetron Sputtering of Thin Film MIM Capacitors with Al and Pt Electrodes: *Jack Murray*[1]; Wayne Huebner[1]; Matthew O'Keefe[1]; [1]Missouri University of Science and Technology

The deposition of thin film metal-insulator-metal (MIM) capacitors for high frequency applications by magnetron sputtering was investigated. Pt-Al2O3-Pt and Al-Al2O3-Al with symmetric electrode areas were the material combinations tested in this study. All devices were deposited through a shadow mask under the same sputtering conditions resulting in a metal electrode thickness of ~160 nm and dielectric thickness of ~450 nm. DC current-voltage measurements (E = 5MV/cm) coupled with impedance analysis (=15 MHz) was used to characterize the resulting devices. MIM structures with Pt electrodes exhibited a significantly lower resistivity and higher permittivity than devices with Al electrodes. These results indicate the formation of a space charge layer near the metal/insulator interface that depends on the electrode material.

The Second Symposium on the Recycling of Electronic Wastes: Technologies for the Recycling of Electronic Wastes

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Electronic Packaging and Interconnection Materials Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Lifeng Zhang, Missouri University of Science and Technology; Gregory Krumdick, Argonne National Laboratory; Jaan Kers, Tallinn University of Technology; Thomas P. Schuman, Missouri University of Science and Technology (Missouri S&T); Markus Reuter, Ausmelt Limited

Monday AM
February 28, 2011
Room: 15B
Location: San Diego Conv. Ctr

Session Chairs: Lifeng Zhang, Missouri S&T; Gregory Krumdick, Argonne National Lab.

8:30 AM Introductory Comments

8:35 AM
Mechanical Recycling of Electronic Wastes for Materials Recovery: Viktor Laurmaa[1]; *Jaan Kers*[1]; Kaspar Tall[1]; Valdek Mikli[1]; Dmitri Goljandin[1]; Kristiina Vilsaar[1]; Priidu Peetsalu[1]; Mart Saarna[1]; Riho Tarbe[1]; [1]Tallinn University of Technology

In this paper, the mechanical milling of the Printed Circuit Boards (PCB) was carried out. First goal of the work was to examine the management of the WEEE, in particular the re-use of PCB. Firstly, recycling methods of PCBs were summarized. During the study, mechanical separation methods (magnetic-, density- and air separaton), electrical, and chemical methods were examined. Secondly, the optimal particle size for air-classification was determined. Several tests were carried out to find the most effective separation method for separation of different material groups from PCB scrap. The new air classification stand was developed for testing the separation of lightweight particles such as tin foil stripes and plastics. The test results showed sufficiently good separation of heavier Al and Cu. For milled materials characterization, the SA, IA, laser diffraction analysis and SEM were used. The chemical composition of the PCB powders was studied by means of energy dispersive X-ray microanalysis (EDS).

9:05 AM
Processing of Discarded Liquid Crystal Display for Recovering Indium: *Gjergj Dodbiba*[1]; Kunihiko Takahashi[1]; Toyohisa Fujita[1]; N. Sato[2]; Seiji Matsuo[1]; Katsunori Okaya[1]; [1]The University of Tokyo; [2]Tohoku University

Different materials are used for manufacturing various parts of a cellular phone. Considering that a great number of mobile phones are being discarded every year, it is important to recover these materials and then recycle them in order to save resources. The primary objective of this work is, therefore, to find and suggest ways on how to recover various materials from discarded cellular phones for recycling. Much effort, for example, has been put into recovering and recycling tin oxide (ITO), a mixture of indium(III) oxide, (In2O3) and tin(IV) oxide from the discarded LCDs. Here we report a simple and cost-effective process, which includes a HCl treatment process for altering the structure of the indium(III) compound into a chloride-induced indium compound, in order to facilitate the vaporization of indium at a relatively low temperature. The chloride-induced indium compound is then vaporized, condensed and recovered.

9:35 AM
Green Pyrolysis of Used Printed Wiring Board Powders: *Lucas Damoah*[1]; Xiangjun Zuo[1]; Lifeng Zhang[1]; Thomas Schuman[1]; [1]Missouri University of Science and Technology

Rapid technological innovation has propelled the use of electronic equipment leading to the generation of more and more waste electrical and electronic equipments (WEEE). Printed wire board (PWB) is a component made of one or more layers of insulating material with electrical conductors. Current technology for recycling PWBs uses pyrometallurgical or hydrometallurgical methods, which generate atmospheric pollution releasing dioxins and furans or high effluent volumes. To investigate an environmentally friendly process to recycle PWBs, 50g each of PWB samples with and without 10g of CaCO3 additives were pyrolyzed in a tube furnace. Liquid, gas and solid products were realized with a PWB conversion fraction of 52.65 %. Analysis of the exhaust gases from the experiments using GCMS and MS showed that without CaCO3 additives poisonous gases such as C6H6 and HBr are produced which are adequately controlled when CaCO3 is added. The exhaust gas was treated using concentrated NaOH solution.

10:05 AM
Leaching of Lead from Solder Material Used in Electrical and Electronic Equipment: *Manis Kumar Jha*[1]; Pankaj Choubey[1]; Archana Kumari[1]; Rakesh Kumar[1]; Vinay Kumar[1]; Jae-chun Lee[2]; [1]National Metallurgical Laboratory (CSIR); [2]Korea Institute of Geosciences & Mineral Resources

Present work is a part of developing novel recycling technique for waste printed circuit boards (PCBs), i.e. the liberation of metals from PCBs by organic swelling followed by the treatment of resin to remove/ recover hazardous soldering materials. In order to recover the hazardous metallic constituent lead from the liberated resin, initially the leaching studies were made using fresh solder containing 63.9% Pb and remaining tin. Experimental results obtained in different conditions viz. time, temperature and acidity showed ~97.20% of lead dissolution with 6M HNO3 at solid to liquid (S:L) ratio 1:10 (g/mL) and temperature 90°C in 75 minutes. The result of the studies validated with crushed PCBs shows that almost total lead and tin was leached out with 6M HNO3 and 6M HCl respectively at S:L ratio 1:10 (g/mL) and temperature 90°C within 50 minutes. The results will be useful for the treatment and safe disposal of PCBs resin.

10:35 AM Break

10:45 AM
Copper Recovery from Printed Circuit Board of E-Waste: *Toyohisa Fujita*[1]; Hiroyuki Ono[1]; Gjergj Dodbiba[1]; Seiji Matsuo[1]; Katsunori Okaya[1]; [1]The University of Tokyo

Here, we discuss the copper recovery from circuit boards. In an attempt to raise the copper grade of printed circuit boards (PCB), carbonization treatment was investigated. The crushed PCB without surface-mounted parts was carbonized under nitrogen atmosphere. After screening, the char was classified by size into oversized pieces, undersized pieces and powder. The copper foil and glass fiber pieces were liberated and collected in undersized fraction. The copper foil was liberated easily from glass fiber by a "stamping" treatment. Liberation rate of copper foil was high at high carbonized temperature. The experimental results indicated that 90% of oversized char was liberated and the copper foil recovery from PCB carbonized at 1073 K was 110 kg/t. The copper recovery from char powder by flotation was also investigated. Of the copper grade 15% and of the copper recovery 80% were obtained.

11:15 AM
Recovery of Silver from Spent Plasma TV Monitors: *Katsutoshi Inoue*[1]; Biplob Biswas[1]; Hidetaka Kawakita[1]; Keisuke Ohto[1]; Atsushi Hoshino[2]; [1]Saga University; [2]Nishinihon Kaden Recycle Corporation

The method of extraction of silver from scrap of plasma TV monitors using noncyanide reagents – sodium thiosulfate and acidothiourea – was investigated, which was followed by silver recovery from acidothiourea leach solutions. It was found that acidothiourea solution gave almost complete extraction of silver while thiosulfate provided only 40% extraction. The rate of silver extraction using acidothiourea solution was found to be extremely fast. An increased dissolution rate was also found at elevated temperature. The recovery of silver through coagulation-precipitation from acidothiourea leach solutions was carried out with a natural biodegradable material, persimmon tannin extract. It was proved to be a promising material for complete silver recovery. Besides, precipitative recovery of silver by means of cementation with zinc powder was conducted. It was found that simple cementation with zinc powder exhibits a very good precipitative property for which it could be used in hydrometallurgical processing.

11:45 AM
A Process for Efficient Material Recovery from Scrap Electronics: *Jeffrey S. Spangenberger*[1]; Joseph Pomykala[1]; John Hryn[1]; Bassam Jody[1]; Edward Daniels[1]; [1]Argonne National Laboratory

End-of-life electronic devices continue to grow rapidly. With only about 20% of these devices entering the existing recycling infrastructure valuable resources are ending up in our landfills limiting the supply of raw material feed for the electronics industry. We evaluated the recovery rate of various materials in electronic scrap using our versatile material recovery pilot plant. The process uses a 2-stage approach; the first stage is a bulk separation which classifies materials into specific types while the second stage refines these materials further. The scrap processed for this study included electronics of various types including computers, copiers, and musical keyboards but not video monitors. The study focused on the segregation of circuit board material from commingled shred. A novel method was evaluated to concentrate this material so that platinum group metals could be recovered. Ferrous and non-ferrous metals, along with polymers were separated for recovery and subsequent re-use as well.

Thermally Activated Processes in Plastic Deformation: Nucleation and Diffusive Processes

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizer: Christopher Woodward, Air Force Research Laboratory

Monday AM
February 28, 2011

Room: 1B
Location: San Diego Conv. Ctr

Session Chairs: Jeffery Rickman, Lehigh University; Carelyn Campbell, NIST

8:30 AM Invited
Challenges in the Modeling of Nucleation and Growth Processes: *Jeffrey Rickman*[1]; [1]Lehigh University

In this talk I will review several methodologies for modeling nucleation and growth processes in different contexts. In particular, I will first outline the calculation of nucleation rates using both atomic-level and coarse-grained descriptions of transforming systems and highlight the computational challenges that must be overcome to perform these calculations. In addition, I will explore the use of stochastic geometry to characterize the kinetics of a first-order phase transition and suggest several microstructural descriptors that can be employed to distinguish among different nucleation scenarios. Finally, as an application of the methods described here, I will examine the problem of deducing nucleation conditions from a coalesced microstructure and the tailoring of microstructures in transforming, misfitting thin films.

9:00 AM Invited
Analysis and Modeling of Nucleation Controlled Reactions: *John Perepezko*[1]; Seth Imhoff[1]; [1]University of Wisconsin Madison

Nucleation reactions control the initial stage of microstructure development. The product phase number density, size distribution and morphology are nucleation reaction signatures that are directly related to the nucleation

pathway. An effective experimental study of nucleation reactions requires the documentation of the nucleation reaction signatures and the associated stochastic behavior. Kinetic models based upon the classical nucleation theory can provide valuable mechanistic insight on the rate controlling steps. An inherent characteristic of nucleation reactions is the initial metastable state of the parent phase that is often treated as a uniformly undercooled or supersaturated condition, but recent developments in local atomic structure analysis indicate that nanoscale heterogeneities can be important at high driving free energies. Several aspects of the experimental modeling and analysis approaches are illustrated from recent progress in the examination of nanoscale microstructures and nucleation statistics measurements.

9:30 AM Invited

Diffusive Molecular Dynamics: *Ju Li*[1]; Sanket Sarkar[2]; William Cox[2]; Thomas Lenosky[2]; Erik Bitzek[1]; Yunzhi Wang[2]; [1]University of Pennsylvania; [2]The Ohio State University

The interplay between diffusional and displacive atomic movements is a key to understanding deformation mechanisms and microstructure evolution in solids. The ability to handle the diffusional time scale and the structural complexity in these problems poses a general challenge to atomistic modeling. We present here an approximate approach, called Diffusive Molecular Dynamics (DMD), which can capture diffusional time scale while maintaining atomic resolution, by coarse-graining over atomic vibrations. DMD combines long-range elastic and short-range atomic coordinate interactions simultaneously with gradient chemical thermodynamics. The model is applied to nanoindentation, void growth under tension and sintering. Intimate coupling between diffusional mechanisms such as diffusional creep and displacive mechanisms such as dislocation nucleation, are observed in these processes.

10:00 AM Invited

Predicting Diffusion Coefficients of Multi-Component Solids from First Principles: *Anton Van der Ven*[1]; [1]University of Michigan

Diffusion in technologically important materials often involves non-dilute atomic transport that depends on the nature of intrinsic defects, the energetically most favorable hop mechanisms and the degree of short- and long-range order among the constituents of the solid. In this talk, I will describe how these factors can be rigorously accounted for in a first-principles prediction of diffusion coefficients in non-dilute alloys. The approach relies on the evaluation of Kubo-Green expressions, which provide the link between macroscopic diffusion coefficients and atomic trajectories sampled in kinetic Monte Carlo simulations. A first-principles description of the thermodynamics of short- and long-range order in multi-component solids is achieved with the cluster expansion formalism. I will describe recent work on the prediction of diffusion coefficients in intermetallic compounds and Li ion battery electrode materials.

10:30 AM Break

10:45 AM Invited

Predicting Volume-Based Diffusion in Multicomponent Multiphase Alloys: *Carelyn Campbell*[1]; [1]National Institute of Standards and Technology

Modeling of many thermally activated processes requires accurate prediction of the volume-based (bulk) diffusion in a multicomponent multiphase alloy. CALPHAD-based multicomponent diffusion mobility databases, in combination with multicomponent thermodynamic databases, provide a powerful tool for predicting multicomponent diffusion behavior. The development of these multicomponent diffusion databases requires assessment of diffusion mobilities based on the optimization of individual diffusion coefficient measurements, measured multicomponent diffusion couple profiles, and available first principle data. These assessment methods are applied to both disordered and ordered phases in a variety of alloy systems, including Ni-based superalloys, steels, and Cu-In-Se based photovoltaic absorber materials. The quality of the assessment is determined by comparing the assessment predictions to available experimental data not included in the assessment process and various published diffusion correlations. The inclusion of additional diffusion mechanisms, such as grain boundary and stress-induced mechanisms, to the bulk diffusion will be discussed.

11:15 AM Invited

Advanced Methods for Modeling Thermally Activated Processes: *Arthur Voter*[1]; [1]Los Alamos National Laboratory

A significant issue in accurately modeling diffusion in materials is that the dominant mechanisms are not always the simple unit processes we might think they are. Thus, even if barriers and prefactors are calculated accurately using a high-quality potential or electronic structure method, the predicted evolution may be wrong because key reaction paths are missing from the kinetic model. I will present an introduction to methods such as adaptive kinetic Monte Carlo and accelerated molecular dynamics, developed beginning in the late 1990's, for simulating the long-time dynamics of diffusive systems with no prior assumptions about the reaction mechanisms. I will discuss the strengths and weaknesses of the different methods and give some examples of recent results. I will also discuss prospects for the future and the main issues standing in the way of making these methods into general tools that can reach relevant time scales for real engineering materials.

11:45 AM Invited

Equilibrium and Time-Dependent Solute Segregation at Grain Boundaries: Systematic Monte Carlo Studies: Irina Belova[1]; *Graeme Murch*[1]; Thomas Fiedler[1]; [1]The University of Newcastle

Solute segregation to grain boundaries has long been quantified by segregation factors. Segregation factors are generally obtained in a grain boundary diffusion experiment in the Harrison Type-B and C kinetics regimes. The location of the limits and even the existence of the Harrison kinetics regimes are poorly known in the presence of segregation and this has important consequences on the experimental values of segregation factors. Another important aspect is that the segregation is assumed to be at equilibrium at all times. This requirement may not always be fulfilled in a transient diffusion experiment. In this talk, we provide an overview of solute segregation, the Harrison kinetics regimes and the recent progress that has been made using the Lattice Monte Carlo method for delineating these regimes in the presence of segregation for various grain boundary diffusion models. The time dependence of the segregation factor and associated effects will also be discussed.

12:15 PM

Atomistic Modeling of Interactions of Dislocation Pile-Up with Grain Boundaries: *Jian Wang*[1]; Steven Valone[1]; Richard Hoagland[1]; Timothy Germann[1]; [1]Los Alamos National Laboratory

Interfaces are a common planar defect in materials and can act as Sources, Sinks and Barriers for point defects and line defects. Interfaces possess many metastable states for a given set of macroscopic degrees-of-freedom. These various states may differ in energy, but by relatively small amounts, and may be separated by small energy barriers. Consequently, they may easily change state and configuration in response to changes in stress, temperature, and composition. These easy configurational changes enable such multi-state interfaces to actively participate in and influence a broad array of reactions and processes. Using atomic scale models (topological model and atomistic simulation), interface structures and properties and the interaction of interface with dislocations are studied. The results show that interfaces play a crucial role in determining material strength due to the dislocations-interfaces interactions and in nucleating lattice dislocations in associated with the reconstruction of interface structures.

Ultrasonic Welding for Lightweight Components: Session I

Sponsored by: The Minerals, Metals and Materials Society, American Welding Society, TMS Light Metals Division, TMS Structural Materials Division, TMS: Young Leaders Committee, ASM-MSCTS: Materials and Processing Committee, METSOC-CIM: Metal Processing and Fabrication Committee
Program Organizer: Frank Balle, University of Kaiserslautern

Monday AM
February 28, 2011
Room: 33A
Location: San Diego Conv. Ctr

Session Chair: Frank Balle, Institute of Materials Science and Engineering, University of Kaiserslautern (Germany)

8:30 AM Introductory Comments, Frank Balle, Organizer

8:35 AM
Mechanisms of Joint Formation in Ultrasonic Spot Welding Aluminium Automotive Sheet: *Phil Prangnell*[1]; Dimitrios Bakavos[1]; Yingchun Chen[1]; [1]The University of Manchester

High power ultrasonic spot welding (HP-USW) is an extremely efficient new method for welding Al alloys using ~ 2% of the energy of RSW. The overall objective of this work was to improve our current poor understanding of the mechanisms of weld formation when HP-USW is applied to aluminium automotive sheet. To this end, we report on the results of a detailed investigation, where we have studied the material flow, combined with x-ray tomography, FEG-SEM and EBSD, to characterise the weld defects, stages of weld formation, and microstructure evolution, as a function of welding energy, for a standard automotive material AA6111-T4 in 0.92 mm thick sheet. Results include a discussion of the origin of the complex flow features, noted by other researchers, which were found to occur on three length scales, the observation of a HAZ, and the grain structure developed in a typical weld.

8:55 AM
Ultrasonic Welding of Cables and Wires: *Stefan Heinz*[1]; Dietmar Eifler[1]; Guntram Wagner[1]; [1]University of Kaiserslautern

In the automotive industry the ultrasonic metal welding technology is an established method to join aluminium or copper wires. At the WKK systematic investigations with ultrasonic welded Al-wires and flat flexible cables (FFCs) made of copper were carried out. In the case of Al-wires, joints with a cross sectional area of up to 120 mm^2 and a maximum tensile shear load of about 3500 N were realized. In addition, methods to reduce the unintentional adherence between the sonotrode coupling face and the Al-wires were developed. For FFCs, ultrasonic spot welding systems as well as ultrasonic torsion welding systems were used to weld wire to wire and wire to connector joints. With these joints maximum tensile shear loads comparable to the base material were achieved. Furthermore, a system was developed, which enables the welding of FFCs through the insulation of the cables.

9:15 AM Invited
Ultrasonic Metal Welding of Hybrid Joints: *Guntram Wagner*[1]; Frank Balle[1]; Dietmar Eifler[1]; [1]University of Kaiserslautern

A central research field of the WKK is the realization of innovative hybrid joints by ultrasonic metal welding. The presentation gives an overview about suitable ultrasonic welding systems and essential machine and material parameters which influence the quality of the welds. Besides the ultrasonic welding of dissimilar metals such as Al to Cu or Al to steels, also welds between nonstandard materials like aluminum foam sandwiches or Al-wires can be realized. Moreover, the joining of glass and ceramic with metals is a point of interest at the WKK. By using the ultrasonic metal welding process, it is possible to realize glass/metal welds with tensile shear strengths of 50 MPa. Even, for ceramic/metal-joints actually shear strengths values up to 120 MPa were measured. Finally, selected results about the effects of thermally induced residual stresses on the hybrid joints and questions of the bonding mechanisms will be discussed.

9:45 AM
Optimization of Aluminium to Magnesium Ultrasonic Spot Welding: *Lexi Panteli*[1]; Yingchun Chen[1]; David Strong[1]; Xiaoyun Zhang[2]; Phil Prangnell[1]; [1]The University of Manchester; [2]Beijing Institute of Aeronautical Materials

Dissimilar joining of aluminium and magnesium alloys is problematic due to the formation of brittle intermetallic phases at the interface. Successful joining will afford significant weight savings in the automotive industry as dissimilar materials combinations could be used to create lighter, more fuel efficient multi-material structures. Ultrasonic welding may offer a joining solution, but the process of joining aluminium to magnesium is not well understood. Here, we have investigated the effect of energy input and welding time on joint formation between aluminium 6111 and magnesium AZ31, and report on the optimum welding conditions, interfacial heat generation and the formation of a significant intermetallic layer. Further, the effect of pre-coating the magnesium with aluminium is discussed and the resulting reduction in intermetallic formation is shown.

10:05 AM Break

10:15 AM
Fatigue Failure Behavior and Life Estimation of Ultrasonic Spot Welds in Lap-Shear Specimens of Magnesium and Steel Sheets: *Teresa Franklin*[1]; Jwo Pan[1]; Michael Santella[2]; Tsung-Yu Pan[2]; [1]University of Michigan; [2]Oak Ridge National Laboratory

Fatigue behavior of ultrasonic spot welds in lap-shear specimens of magnesium AZ31B-H24 and hot-dipped-galvanized mild steel sheets is investigated based on experimental observations and a fatigue life estimation method. Optical micrographs of the welds before and after fatigue tests are examined to understand the failure mechanism of the welds. The optical micrographs indicate that the magnitude of the load range has significant influence on the location of kinked fatigue crack initiation. The location of the kinked fatigue crack initiation affects the failure mode of the welds. The failure mode changes from a partial nugget pullout mode through the magnesium sheet at high load ranges to an eyebrow crack growth mode through the magnesium sheet at lower load ranges. A structural stress model based on the closed-form structural solution is adopted to predict the fatigue lives of spot welds. The predicted fatigue lives are compared with the experimental results.

10:35 AM
Effect of Zinc Coatings on Joint Properties and Interface Reactions in Aluminum to Steel Ultrasonic Spot Welding: *Farid Haddadi*[1]; Phil Prangnell[1]; [1]The University of Manchester

Dissimilar joining of aluminium to steel sheet is an important potential application of USW for multi-material automotive structures. Here, the weldability of un-coated and zinc coated steel with aluminium is discussed, using a 2.5 kW USW welder. Results show that zinc steel results in a higher weld performance than uncoated steel. The effect of parameters such as energy and clamping force have been investigated. For Al6111-T4 to DC04 joints it has been found that the tensile strength reaches a maximum after ~1.5 second welding time before decreasing, which is about 90% of the strength of an Al–Al joint. In comparison welds between Al6111-T4 and zinc coated steel took longer to achieve the same weld energy and maximum strength, because of melting of the Zinc coat, but matched the Al-Al joint properties. The reasons for these different behaviours are discussed in terms of the interfacial reactions, that take place.

10:55 AM
Ultrasonic Spot Welding of AZ31B to Galvanized Mild Steel: *Michael Santella*[1]; Teresa Franklin[2]; Tsung-Yu Pan[1]; Elliot Brown[3]; Jwo Pan[2]; [1]Oak Ridge National Laboratory; [2]University of Michigan; [3]EB Scientific Enterprises

Ultrasonic spot welds were made between sheets of 0.8-mm-thick hot-dip-galvanized mild steel and 1.6-mm-thick AZ31B-H24. Lap-shear strengths of 3.0-4.2 kN were achieved with weld times of 0.3-1.2 s. Failure to achieve

strong bonding of joints where the Zn coating was removed from the steel surface indicate that Zn is essential to the bonding mechanism. Microstructure characterization and microchemical analysis indicated temperatures at the AZ31-steel interfaces reached at least 344°C in less than 0.3 s. The elevated temperature conditions promoted annealing of the AZ31-H24 metal and chemical reactions between it and the Zn coating.

11:15 AM

Ultrasonic Welding of Hybrid Aluminum/CFRP-Joints: Microstructure, Monotonic Properties and Fatigue Behavior: Stefan Huxhold[1]; *Frank Balle*[1]; Guntram Wagner[1]; Dietmar Eifler[1]; [1]University of Kaiserslautern

Ultrasonic metal welding is well suited to realize aluminum alloy/carbon fiber reinforced polymer (CFRP) – joints. Important advantages of the ultrasonic welding process are: welding times less than three seconds and welding temperatures below 450°C. The joints are realized by softening and displacing of the polymer out of the welding zone as a result of the ultrasonic shear oscillation. In contrast to conventional joining processes this is the pre-condition that a direct contact between the aluminum and the carbon fibers takes place. In investigations with rolled blank sheets shear strengths of about 30 MPa were realized for AA5754/CF-PA66 – joints. By special surface pre-treatments of the metal, like shot peening or etching, the joint strength was increased up to 60% and the aging behavior of the joints was enhanced. Furthermore, bonding mechanisms and the fatigue behavior of the ultrasonic welded hybrid joints were examined in detail.

11:35 AM Concluding Comments

2011 Functional and Structural Nanomaterials: Fabrication, Properties, Applications and Implications: Nanomaterials: Energy

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

Program Organizers: Jiyoung Kim, Univ of Texas; David Stollberg, Georgia Tech Research Institute; Seong Jin Koh, University of Texas at Arlington; Nitin Chopra, The University of Alabama; Suveen Mathaudhu, U.S. Army Research Office

Monday PM — Room: 8
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Seong Jin Koh, University of Texas at Arlington; Seung Kang, Qualcomm

2:00 PM Introductory Comments

2:05 PM Invited

Titanium Oxides Thin Film Anodes for All-Solid-State Lithium Ion Batteries: Ming-Che Yang[1]; Danijel Gostovic[2]; *Shirley Meng*[2]; [1]University of Florida; [2]U.C. San Diego

The requirements of low power and small size energy source in CMOSRAM and Micromechanics (MEMS) have brought up the use of all-solid state thin film batteries. Metallic lithium is not a suitable anode material for all-solid-state thin film batteries because of the large irreversible capacity and high reactivity with air. Spinel lithium titanate Li4Ti5O12 and anatase TiO2 have attracted great attention as potential anode materials because of the following attributes: stable in the air atmosphere, good Li-storage capacity, safety against overcharging and stable voltage plateau. Pulsed laser deposition was used for the electrode deposition. Film depositions were carried out at different temperature, different oxygen partial pressure and different substrate. In order to understand the relationship between crystal structure and electrochemical properties of thin films, the thin films were studied by X-ray diffraction, scanning electron microscopy (SEM), and TEM. Electrochemical performances were investigated by galvanostatic techniques in lithium cells.

2:35 PM

Synthesis of Titania Nanotubes for Lithium Ion Batteries: *Hyukjae Lee*[1]; Sang-Jun Park[1]; [1]Andong National University

Titania nanotubes are synthesized via hydrothermal and anodization techniques for an alternative anode material of lithium ion battery and their properties are measured via X-ray diffraction, electron microscopy, potentiostat, etc. Different synthetic conditions and post synthesis treatments cause various crystallographic phases and structural morphologies in the synthesized titania nanotubes. The lithium electrochemical behavior of the synthesized titania nanotubes is studied by the use of coin cells, and the results show that final crystallographic phase and morphology are the most important factors for lithium intercalation behavior in both synthetic methods. However, there are distinct differences in lithium intercalation behavior between titania nanotubes synthesized from hydrothermal and anodization methods as well. The lithium intercalation performance of titania nanotubes are further enhanced by the introduction of the carbon sources during synthesis which, in turn, results in carbon coating or doping at titania nanotubes.

2:50 PM

Nanogenerators from Piezoelectric-Coated Carbon Nanotubes: *David Stollberg*[1]; [1]Georgia Tech Research Institute

This research creates an energy harvesting device that harnesses ambient and extraneous motion of a vehicle or wind, etc. and converts it to electrical energy: a "nanogenerator" using carbon nanotubes (CNTs) and piezoelectrics. The CNTs serve as an efficient, lightweight, robust electrical conduit and structural support for nanothickness piezoelectrics, zinc oxide (ZnO). Arrays of ZnO-coated CNTs are built on rigid silicon substrates or flexible carbon fiber substrates. Disturbances, vibrations and other inertial motions cause the ZnO-coated CNTs to bend and stress, producing a current. A simple ZnO-coated CNT structure can generate an electric current density of ~0.3 μA/cm2 to 4 mA/cm2 with a power density of 0.1pW/cm2 to 0.01μW/cm2 under a small applied load. With optimization of the pattern and dimensions, there is promise for significantly increasing the current density to over 40 mA/cm2 and the power density to over 10μW/cm2, enough to power small sensors and/or MEMS/NEMS devices.

3:05 PM

Structural and Electrochemical Characterization of Individual Nanowires for Li-ion Batteries: *John Sullivan*[1]; Arunkumar Subramanian[1]; Jianyu Huang[1]; Michael Shaw[1]; Nicholas Hudak[1]; Yongjie Zhan[2]; Jun Lou[2]; [1]Sandia National Labs; [2]Rice University

Nanowire (NW) anode and cathode materials offer the possibility of increased capacity and/or rate performance in Li-ion batteries through better accommodation of the large stresses incurred during lithation/delithiation and through reduction in the path length for Li-ion or electron transport. One challenge with NW materials development is their electrochemical characterization, particularly diagnostics at the individual NW level. To meet this need, we have developed chip-based platforms that allow the isolation and electrochemical, structural, and electrical characterization of individual NWs. We have demonstrated this approach by isolating single MnO_2 NW cathodes and measuring their structural changes by transmission electron microscopy following increasing levels of electrochemical lithiation. We observe a rapid disordering of the MnO_2 lattice and a monotonic decrease in electrical conductance with increasing Li content. Sandia is operated by Sandia Corp., a wholly owned subsidiary of Lockheed Martin Co., for the U.S. DOE's NNSA under contract DE-AC04-94AL85000.

3:20 PM

Phase Field Simulations of Morphological Evolution during Lithium Intercalation/Extraction in Li-ion Batteries: *Saswata Bhattacharya*[1]; Linyun Liang[1]; Long-Qing Chen[1]; [1]Pennsylvania State University

We present a three-dimensional phase field model to study the morphological evolution during the intercalation/extraction of Li-ions into a host electrode in Li-ion batteries. The effects of anisotropic diffusional mobility of Li-ions in the electrode host lattice, flux of Li-ions across the electrode/electrolyte interface, and coherency strains arising from the lattice parameter mismatch

between the lithiated and unlithiated phases are studied using $LiFePO_4$ as an example. We use spectral smoothed boundary method to solve the governing equations. Our simulations show the formation of plate-like crystallites of the lithiated phase when anisotropic bulk transport of Li-ions is assumed to be the rate limiting mechanism. We compare the microstructural features obtained from our simulations with available experimental observations.

3:35 PM Break

3:50 PM Invited
Dye Sensitized Solar Cells with 3-Dimensional Anodes, and Prospects for Incorporating Unusual Photophysical Processes: *Michael Tauber*[1]; [1]University of California, San Diego

Dye sensitized solar cells (DSSCs) are an emerging class of photovoltaics that are potentially much less expensive than traditional silicon solar cells. Presently the record-holding 11% efficient DSSC is only half as efficient as the best commercial silicon solar cells. Furthermore, the challenge of large-area scaling of DSSCs has slowed their commercialization and utilization. We have designed and constructed new DSSCs that incorporate titanium dioxide nanotubes anodically grown in a 3-D array from titanium metal substrate. The cells have >5% efficiency, and are free of transparent conductive oxide. The highly conductive all-metal substrates will make cells of this kind easy to scale to large areas, and at low cost. The 3-D cells are an optimal platform for testing new dyes and dye mixtures in DSSCs. Prospects for exploiting unusual photophysical processes in DSSCs, including triplet excited states and singlet fission, will be described.

4:20 PM
Formation of Silver Nanocube Array via Silica-Polymer Nanocomposites: Chi-Kai Chiu[1]; Yong-Jae Choi[1]; *Tzy-Jiun Luo*[1]; [1]North Carolina State University

Silver nanocubes are spontaneously formed at room temperature on the surface of sol-gel silica nanocomposite film that consists of polyethyleneglycol (PEG) and silver ions. The formation of silver nanocubes can be achieved without the presence of poly(vinylpyrrolidone), which was found to enhance the sharpness of nanocube edges. The geometry (e.g., spherical or cubic shape) nanocubes are critically dependent on the thickness of nanocomposite film while the dimension of nanocubes are the function of film thickness. The spontaneous reduction of silver occurs inside the nanoporous structures of silica where PEG serves as both mobile phase and reducing agent. This phenomenon is driven by the shrinkage of the sol-gel silica layer during aging process and is controllable through surface functional groups. The ability to create silver nanocubes on a solid substrate at room temperature suggests that it is possible to control both position and size of silver nanocubes using imprint technique.

4:35 PM
Self-Assembled Epitaxial Quantum Dot Multilayers: A Stochastic Continuum Modeling Approach: *Lawrence Friedman*[1]; [1]National Institute of Standards and Technology

Some strained semiconductor heterostructures such as Ge_xSi_{1-x}/Si and $In_xGa_{1-x}As$/GaAs spontaneously form into zero-dimensional nanoelectronic structures known as self-assembled quantum dots (SAQDs). These structures are simple in composition, yet they exhibit rich behavior such as strain-induced zero- and finite-temperature phase transitions, nanoscale stochastic behavior and the ability to form multilayer superlattices that are ordered by strain-coupling. Thus, modeling their formation addresses many interesting aspects of nanoscale science and nanomechanics, while at the same time aiding in the design and manufacture of functional materials. To investigate the ordering (quality improving) and disordering (quality degrading) influences, a previously developed continuum stochastic model is refined and adapted to SAQD multilayers. The continuum stochastic approach enables large volume and long-time scale modeling that is necessary to collect statistics that characterize the order and quality of self-assembled quantum dots.

4:50 PM
Three Dimensional Carbon Nanotube Photovoltaics: *Jack Flicker*[1]; Jud Ready[2]; [1]Georgia Insitute of Technology; [2]Georgia Tech Research Institute

The use of a nanostructured, three dimensional morphology in photovoltaics devices has occurred only relatively recently. We introduce a three dimensional photovoltaic device with carbon nanotube pillars coated with photoactive materials to create a solar cell. The extra dimensionality of this cell added by the nanotubes has been theorized to increase the relative energy generated over planar cells by up to four times. The energy increase is due to an increase in the interactions between photons and the photoactive material as the sun is at an off normal angle to the cell substrate. Prototypes of these cells have been made and, although suffering from a low overall efficiency, do show an increased energy production in the same manner that theory predicts when the light source is at an off normal angle. Increases in device efficiency by evolution of processes and structures will also be discussed.

5:05 PM
New Silicon Alloy Nano-Particulate Materials for Lithium Ion Battery Anodes: *Emma White*[1]; Lisa Rueschhoff[1]; Iver Anderson[2]; Steve Martin[3]; [1]Iowa State University & Ames Laboratory; [2]Ames Laboratory & Iowa State University; [3]Iowa State University

Lithium ion batteries currently offer the highest energy density, ~400 mAh/g, combined with high voltage, but the theoretical energy density of metallic lithium, ~4,000 mAh/g, has been difficult to achieve so far in a secondary cell. Poor cycle life has limited the use of higher charge capacity materials, such as silicon. A novel composite material made of alloyed silicon nano-particles has been produced which may circumvent the previous issues in using pure silicon as an anode material. The spark discharge method of particle production, the characterization of the resulting particles, and their initial performance in a lithium battery cell will be described. Funding provided by Ames Lab Seed Fund through Ames lab Contract DE-AC02-07CH11358.

5:20 PM Concluding Comments

2nd International Symposium on High-Temperature Metallurgical Processing: Microwave Heating and Iron and Steel Production

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee, TMS: Energy Committee
Program Organizers: Jiann-Yang Hwang, Michigan Technological University; Jerome Downey, Montana Tech; Jaroslaw Drelich, Michigan Technological University; Tao Jiang, Central South University; Mark Cooksey, CSIRO

Monday PM — Room: 18
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Chenguang Bai, Chongqing University; Jerome Downey, Montana Tech of the Univ of Montana

2:00 PM
A Study of Coal-Based Direct Reduction of Composite Binder Magnetite Preheated Pellets: *Deqing Zhu*[1]; Tiejun Chun[1]; Vinicius Mendes[1]; Jian Pan[1]; Jian Li[2]; [1]Central South University; [2]Research Institute of Baosteel

A study of the coal-based direct reduction behaviors of composite binder magnetite pellets was carried out in a simulating coal-based grate-rotary kiln process. It is shown that preheated pellets possess much better reducibility than fired oxide pellets: 40 min are required for preheated pellets to reach over 90% metallization degree compared to 100 min for fired oxide pellets. The compressive strength of preheated pellets decreases dramatically at the earlier stage of reduction, climbs quickly after reducing for 30min and achieves a high value at the end of reduction. However, the compressive strength of metallized pellets from reducing of fired pellets is much lower,

more cracks and fractures being formed. In the preheated pellets, crystallite of metal iron is coarse. In the oxide pellet, a large number of interconnected crystals of metal iron are formed inside pellets, and pellet structure is very compact.

2:20 PM

A Model of Decarburization of Iron/Carbon Droplets: *Mark Schwarz*[1]; [1]CSIRO

A computer model which follows decarburization of Fe/C/S droplets and predicts the onset of "carbon boil" has been developed based on the mechanism of El-Kaddah and Robertson. According to this mechanism, boil occurs when the supersaturation pressure of CO within the droplet reaches a critical value. Predicted times to boil have been compared with measurements made at CSIRO under various operating conditions. The model results generally agree well with measured times to boil, thereby validating the boil mechanism. The critical supersaturation pressure for boil determined by comparing predicted and measured boil times is similar to that determined by El Kaddah and Robertson (48-240 atm) and depends on the kinetics of the surface decarburization reaction. The computer model presicts that the time to boil decreases as the gas-droplet velocity increases, as the drop temperatures decreases and as the droplet diameter decreases.

2:40 PM

Non-Isothermal Kinetics of Reduction Reaction of Oxidized Pellet under Microwave Irradiation: Wu Kai[1]; *Huang Zhu-Cheng*[1]; Peng Hu[2]; [1]CSU; [2]Syno-Therm Co. Ltd

The microwave heating characteristics of the mixture of acid pellet and coal was researched, and the non-isothermal reductive dynamics was discussed in the paper. The results show that, the slow-heating stage of the temperature rising process can be segmented into two heating temperature curve that have good linear relationship. It can be seen temperature programming. In the first stage, i.e., between 827K and 1073K, the reaction mechanism obeys three-dimensional diffusion model. In the second stage, i.e., between 1093K and 1323K, the reaction mechanism obeys chemical reaction model. The apparent activation energies are found respectively to be 63.96kJ/mol for the first stage and 61.27kJ/mol for the second stage. That is lower than the apparent activation energy under conventional heating. The research of the microstructure shows microwave can improve the kinetics of the reduction. Microwave has anxo-action to the reaction obviously.

3:00 PM

Microwave Dielectric Properties of Pyrolyzed Carbon: Zhiwei Peng[1]; *Jiann-Yang Hwang*[1]; Wayne Bell[1]; Matthew Andriese[1]; Shuqian Xie[2]; [1]Michigan Technological University; [2]Northeastern University

Pyrolyzed carbons are generally known as good microwave absorbers and their dielectric properties still remain to be fully explored. In the present study the dielectric properties and dielectric polarization-relaxation phenomenon of a typical activated carbon was investigated. The experimental results indicate the complex permittivity is highly dependent on temperature and frequency. The decrease of permittivity with increasing temperature from room temperature to ~100 ^{o}C is probably ascribed to the release of the water vapor adsorbed on the surface of activated carbon, and the variations of permittivity with temperatures between 100 ^{o}C and 450 ^{o}C in the frequency range of 300 MHz to 3000 MHz are mainly attributed to the decreased relaxation time of dielectric polarization.

3:20 PM

Fugitive Emissions Related to Oxidation of Liquid Silicon During Ladle Refining: *Mari Næss*[1]; Gabriella Tranell[1]; Nils Eivind Kamfjord[1]; [1]Norwegian University of Science and Technology

In oxidative ladle refining (OLR) of silicon, the metal surface is oxidized resulting in the formation of a condensed silica fume (SiO2). In the current work, industrial measurement campaigns were performed aiming to measure the fume generation during OLR. A thorough discussion of the possible mechanisms has been included in order to get a better understanding of the system. The measurement campaigns were done at the Elkem Salten plant in Norway. In addition to fume generation from OLR, the metal temperatures and ladle purge gas amount were recorded. Other mechanisms of SiO2 formation were considered, however found insignificant. The results of this work suggest that the dominant source of SiO2 fume generation during OLR is the oxidation of the metal surface, with oxygen transport to the metal surface being the limiting factor.

3:40 PM

Reduction Kinetics of Iron Oxide in CaO-SiO2-Al2O3-FexO-C Mixtures: *Yuanyuan Zhang*[1]; Patrick Masset[1]; [1]TU Bergakademie Freiberg

In iron and steelmaking technologies, the determination of the interfacial reaction kinetics between CO/CO2 based mixtures and iron ores represents a challenging and valuable input for the modelling of such high temperature conversion processes. In this work, experiments on the reduction of iron oxides were carried out using high pressure thermogravimetric analysis (HP-TGA) under CO/CO2 mixtures at temperatures and pressures up to 1000°C and 5 bars, respectively. Through analyzing the TGA curves recorded at different heating rates, the reduction rate of the iron oxides by CO/CO2 mixtures and the activation energies of the reduction reactions have been determined. For well defined transformation rate, the iron oxide particles were observed by SEM/EDX analysis to understand the reaction mechanism occurring at the oxide/gas interface. In addition, Mössbauer spectroscopy was used to estimate Fe3+/Fe2+ ratio.

4:00 PM Break

4:10 PM

Optimization of the Process Variables for Making Direct Reduced Iron by Microwave Heating using Response Surface Methodology: *Linqing Dai*[1]; Jinhui Peng[1]; Hongbo Zhu[1]; [1]Kunming University of Science and Technology

To optimize the process variables for making direct reduced iron (DRI) by microwave heating, the reduction temperature, reduction time and the ratio of coal to material were studied with the central composite design (CCD) and their interactions on the Metallization rate were also investigated. The predictive polynomial quadratic equations model was analyzed by ANOVA. Optimal conditions of making DRI can be concluded as follows: 28 min at 1139^{o}, the ratio of coal to material is 20.95%. Under these conditions, the metallization rate is up to 97.06%.

4:30 PM

Study on Nucleation and Growth Mechanism of Iron Crystal Grain in Coal-Based Shaft Furnace Direct Reduction Iron Pellets by Microwave Heating: *Zhucheng Huang*[1]; Zhenyuan Liao[1]; Bing Hu[1]; Lingyun Yi[1]; Yuanbo Zhang[1]; [1]Central South University

Nucleation and growth mechanism of iron crystal grain in coal-based shaft furnace direct reduction process by microwave heating were investigated by optical microscope, scanning electron microscope and EDX. The results indicate that microwave has selectivity on various minerals which absorb microwaves. The high temperature in certain area of pellets and thermal stress on the interface of minerals are formed, results in the wustite grain smashed along the joints, and iron crystal grains turned out a spherical shape obviously. The iron crystal nucleus is formed firstly at the edge of wustite, on the interface between grains and in the holes in pellets, and grows gradually while reduction carries on from surface layer to inner core. Microwave heating promotes the rate of transportation and accumulation of the iron crystal grain, increases growth rate of iron crystal grain and contributes to formation of dense iron-jointed crystal texture eventually.

4:50 PM

In-Situ Mass, Temperature, and Resistance Measurements during Microwave Metallization of Iron Ore and Zink Dust for On-Line Optimization: *Wayne Bell*[1]; [1]Michigan Technological University

Microwave reduction furnaces have a great potential to eliminate traditional electric arc and blast furnaces that are inefficient and environmentally unfriendly. A rotary microwave/electric arc reduction furnace was developed at Michigan Technological University to be marketed commercially in the United States with the hope of rapidly becoming the first green alternative to existing reduction-metallization technologies.Materials are fed onto a 6'

refractory disc in a waveguide attached to 3 100kW microwave applicators. Upon formation of a conductive metallic network from microwaving, an electric arc is used to melt the network into a nugget for extraction. To effectively and continuously run a microwave/EAF furnace, several working parameters must be controlled by the user before operation: Microwave and EAF power, processing speed, mixing ratios of input materials. Using a simple household microwave, several materials properties (resistance, mass, and temperature) can be easily measured and correlated to expose the unknown on-line working parameters.

5:10 PM

Research on the Reduction Mechanism of Vanadium Oxides in Lumpy Zone in Blast Furnace: *YongHong Wang*[1]; Bing Xie[1]; QingYun Huang[1]; JiaRong Yang[1]; [1]ChongQing university

Vanadium-bearing titanomagnetite is used as one of raw materials in blast furnace in Panzhihua Iron and Steel Group Corp. Thermodynamic calculation on reduction of vanadium oxides from ore in lumpy zone of blast furnace showed that with the occurrence of iron the pressure of PCO2/PCO of the vanadium oxides reduction could evidently increased because vanadium could unlimitedly dissolve in liquid iron. Also, the process of iron and vanadium reduction from magnetite ore is simulated in reduction furnace. Chemical analysis method is used to analysis the composition of reduction samples. It is concluded that the reduced degree of iron oxide can be higher than 80% after three hours. And when the reduced degree of iron oxide is between 85% and 90%, vanadium oxides begin to be reduced and the content could reach 0.083% when the reduced degree is 97.4%.

5:30 PM

Investigation on a Microwave High-Temperature Air Heat Exchanger: Jianhua Liu[1]; Yingwei Li[1]; Lijun Liu[1]; Jinhui Peng[1]; Libo Zhang[1]; *Shenghui Guo*[1]; Huilong Luo[1]; Hongpo Wang[1]; Guo Chen[1]; [1]Kunming University of Science and Technology

As essential equipment in the metallurgical industry, current air heat exchanger can not meet the requirements of high temperature heat transfer. In present paper, an energy efficient air heat exchanger, based on accumulation of the heat generated by microwave absorbing materials is presented according to heat transfer theory and principle of the microwave field. A tubular shape structure of heat exchanger was designed and built up through temperature-rising curve, heat transfer performance of different materials heat exchanger. And it was indicated from experiments that tubular shape heat exchanger fabricated by using material B could be used for high temperature heat transfer system, and equilibrium temperature could reach up to 457—485°C.

Advances in Science-Based Processing of Superalloys for Cost and Sustainment: Application of Modeling and Simulation to Component Design and Life Prediction

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: High Temperature Alloys Committee, TMS: Advanced Characterization, Testing, and Simulation Committee

Program Organizers: Donna Ballard, US Air Force; David Furrer, Pratt & Whitney; Paul Jablonski, US Department of Energy; Christopher Woodward, Air Force Research Laboratory; Jeff Simmons, AFRL; Mark Blodgett, Wright-Patterson AFB

Monday PM
February 28, 2011

Room: 33B
Location: San Diego Conv. Ctr

Session Chairs: David Furrer, Pratt & Whitney; Jeff Simmons, AFRL

2:00 PM Invited

Extreme Values in Materials Microstructure: *Anthony Rollett*[1]; Seth Wilson[1]; Jeff Simmons[2]; Katayun Barmak[1]; Michael Groeber[2]; David Rowenhorst[3]; [1]Carnegie Mellon University; [2]Air Force Research Lab.; [3]Naval Research Lab.

Materials scientists and engineers commonly report a single value for a property or microstructural feature. Some properties, however, probe the weakest link in a system. Thus there are references to the use of extreme value statistics to link particle sizes to fatigue life, for example, going back to the 1940s. Grain size is an example where the community has been content to argue over the applicability of, say, the log-normal distribution versus other candidate distributions. Yet, the impact of grain size may be through the presence of large grains in the system, rather than the average. Thus determining the extreme values for grain size is also important for understanding microstructure-property relationships. Several experimental data sets and some derived from computer simulation of microstructure evolution are analyzed to characterize the upper tails of the grain size distribution.

2:30 PM Invited

Uncertainty in Process-Structure-Property Relations: Robust Materials Design: *David McDowell*[1]; [1]Georgia Institute of Technology

Designing materials for targeted performance requirements demands a combined strategy of bottom-up and top-down modeling, simulation, experiments, and systems engineering. In materials design applications, uncertainty can be both stochastic and epistemic. Stochastic (i.e., aleatory) uncertainty stems from stochastic variability and inherent randomness of material processing and morphology, as manifested in heterogeneous, randomly distributed microstructure attributes and defects. Epistemic (i.e., model) uncertainty stems from limits to the knowledge captured in models (model idealization and approximation of reality) and databases. This manifests itself in the limited fidelity and accuracy of predictions, inevitable lack of information, and modeling errors due to interpolations, approximations, convergence, assumptions, and other factors related to methods of obtaining approximate solutions. Characterizing and managing both types of uncertainty are essential in pursuing materials design applications. Components of a practical approach for robust design of materials to achieve specified functionality are presented, addressing uncertainty.

3:00 PM Invited

Rolls-Royce Perspective on Advances in Science-Based Processing of Superalloy Materials and Components: *Mary Lee Gambone*[1]; [1]Rolls-Royce

Recent advances in superalloy material and component capabilities have been attributed to the refinement of processing methods and controls. These refinements have been supported and driven by further understanding of the metallurgical phenomena that subsequently result in enhanced mechanical performance. Physics-based understanding of microstructure evolution and deformation processes has lead to unique combinations of processes and processing control methods. Efforts to develop and apply fundamental understanding of physical and mechanical metallurgy of superalloy materials is not new, however recent discoveries in mechanical behavior and material characterization methods have provided renewed emphasis and insight into the importance of processing. This talk will provide an industrial perspective on how recent advances in focused technical areas are leading to further superalloy capabilities through advances in science-based processing.

3:30 PM Break

3:45 PM Invited

Modeling and Simulation in Fossil Energy Systems – Current Prospectus: *Jeffrey Hawk*[1]; Liang Jiang[2]; [1]U.S. Department of Energy; [2]GE Global Research

Within the realm of energy research, there are many opportunities for modeling and simulation, from large scale power plant systems to the interaction between atoms in an alloy. Specifically in the materials science focus area at NETL, materials modeling and simulation has been used to expedite the development of new materials as well as improve upon, and/or optimize, existing alloys for new fossil energy applications. At GE modeling and simulation research is directed at accelerating the introduction of a component into an existing energy platform through Materials Design Acceleration (MDA). Superalloys remain near the forefront of these activities due to their widespread use in current generations of gas turbines and their potential incorporation into A-USC power plants. Modeling activities at NETL and GE specific to superalloy development and product incorporation will be discussed with emphasis on the computational materials modeling approaches in each instance.

4:15 PM

Modeling Deformation Mechanisms and Grain Structure Evolution during Forging of Powder-Metallurgy Nickel-Base Turbine Disk Alloy: *Wen Tu*[1]; Tresa Pollock[2]; [1]University of Michigan; [2]University of California - Santa Barbara

The properties of superalloy disk materials are sensitive to the microstructure at the end of a series of thermomechanical processing. In this study, high temperature compression testing combined with high resolution Electron Backscatter Diffraction (EBSD) analysis has been used to analyze microstructural-scale straining processes that occur during high temperature forging of a nickel-base superalloy, René 88DT. Orientation imaging has been employed to study grain-level straining and strain storage. Using experimental evidence, constitutive models of deformation mechanisms and microstructure evolution during high temperature compression are proposed. The models predict: 1.) boundaries of superplastic deformation as a function of temperature, strain rate, and grain size, 2.) rate of superplasticity-enhanced grain growth as a function of strain, strain rate, and temperature and 3.) the saturation recrystallized grain size as a function of strain rate and temperature. The sum of the models predicts grain structure evolution during high temperature forging of Ni-base superalloys.

4:35 PM

The Progression of Oxidation Damage for an Advanced Powder Metallurgy Disk Superalloy: *Chantal Sudbrack*[1]; James Smialek[1]; Tim Gabb[1]; David Hull[1]; Timothy Gorman[2]; Doug Wei[3]; Jeff Marshman[3]; [1]NASA Glenn Research Center; [2]University of Dayton; [3]Carl Zeiss SMT Inc.

With the drive towards higher operating temperatures in newly developed gas turbine engines, oxidation resistance and protection of advanced nickel-based turbine disk components are becoming increasingly important. Oxidizing environments are known to impair disk fatigue life at temperatures above 500°C, yet development of protective disk coatings is still in its infancy. Microstructural response of an ME3 disk superalloy is evaluated for moderate (704°C) and aggressive (760-816°C) isothermal exposures up to 2,020 hours. Slow growing Cr-rich scales form superficially and deviate from classic parabolic growth, possibly due to Cr_2O_3 grain boundary (GB) diffusion or shifts in Cr_2O_3-TiO_2-$CrTaO_4$-Al_2O_3 complex scale phase make-up. Cross section analysis reveals sub-surface damage (significant for aggressive exposures) that consists of Al_2O_3 "fingers", interfacial voids, a recrystallized precipitate-free layer and GB carbide dissolution. The effects of a Nichrome coating on this response are also assessed. Detailed spatial and chemical quantification of these microstructures are presented.

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Diffusion Simulation and Oxidation Life Prediction of Turbine Metallic Coatings: *Fan Zhang*[1]; Shuanglin Chen[1]; Weisheng Cao[1]; Y. Austin Chang[2]; Kang Lee[3]; Donna Ballard[4]; Jeff Simmons[4]; [1]CompuTherm, LLC; [2]University of Wisconsin-Madison; [3]Rolls-Royce Corporation; [4]Air Force Research Laboratory

Oxidation protection coatings have become an integral part of advanced gas turbine engine hot section components. These coatings rely on the formation of a slow-growing and adherent Al2O3 scale. Due to the scale formation/spallation at the coating/gas interface and Al diffusion into the superalloy at the coating/substrate interface during service, aluminum is depleted from coating materials. The oxidation life of a coating ends when aluminum is depleted below the critical concentration. In this study, we have developed a modeling tool which integrates thermodynamic calculation, 1-D diffusion model, and an empirical oxidation model (COSP) to understand the oxidation life of coatings. With this modeling tool, the aluminum depletion due to diffusion and Al2O3 scale formation/spallation can be simulated. The oxidation life of a coating, therefore, can be predicted as a function of coating and alloy type and service condition, which is critical for intelligent coating selection, component design, and component maintenance.

5:15 PM

Nitridation of HAYNES® NS-163® Alloy: Thermodynamics and Kinetics: *Michael Fahrmann*[1]; Krishna Srivastava[1]; [1]Haynes International Inc.

Nitride dispersion-strengthened HAYNES® NS-163® alloy is a new unique cobalt-base sheet alloy that offers significantly enhanced creep strength over HAYNES® 617, 230®, and even 188 alloys at temperatures exceeding 1650°F (900°C). This new alloy approaches the performance of oxide dispersion-strengthened alloys while being fabricable and less costly to process since it employs conventional mill technologies. Nitride dispersion strengthening is accomplished via a post-fabrication heat treatment. A thorough understanding and control of the nitridation process will be key to the alloy's commercial viability. In this context, microstructures of partially and fully nitrided sheet samples have been examined. The nitride reaction front advanced following a parabolic growth law. The nature and sequence of formation of the various nitride compounds formed is rationalized by comparing their respective Gibbs free energies of formation. Dispersoid nucleation densities and morphologies are discussed in terms of the locally varying speed of the reaction front.

Alumina and Bauxite: Bauxite Resources and Utilisation

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee
Program Organizers: James Metson, University of Auckland; Carlos Suarez, Hatch Associates Consultants Inc

Monday PM
February 28, 2011
Room: 17A
Location: San Diego Conv. Ctr

Session Chair: To Be Announced

2:00 PM Introductory Comments

2:05 PM
New Development Model for Bauxite Deposits: *Peter-Hans ter Weer*[1]; [1]TWS Services and Advice

Developing a greenfield bauxite deposit generally includes constructing an alumina refinery. Economics have resulted in ever-increasing production capacities for recently-built and future planned greenfield refineries. Rationale: economy of scale. As a result the complexity of a greenfield project has significantly increased and its capital cost has grown to several billion USD. Important consequences: 1. Project owners aim at risk reduction through project financing and formation of joint ventures, further complicating project implementation; 2. Globally only a limited number of (large) companies have the resources to develop greenfield bauxite & alumina projects; 3. Only a limited number of engineering firms have the required skills and experience to successfully implement these mega projects; 4. Only large bauxite deposits get developed. This paper proposes an alternative development model for bauxite deposits resulting in a more efficient use of resources and a lower threshold to develop bauxite & alumina projects.

2:30 PM
Study on the Characterization of Marginal Bauxite from Pará/Brazil: *Fernanda Silva*[1]; João Sampaio[2]; Marta Medeiros[1]; Francisco Garrido[1]; [1]IQ/UFRJ - CETEM; [2]Centro de Tecnologia Mineral

Bauxite from Pará is divided into five different layers. However, only one is processed. The crystallized-amorphous (BCBA) phase is considered a marginal bauxite because it presents a high quantity of SiO2reactive and its use depends on special technologies. BCBA was characterized and results were compared to the bauxite used, nowadays, in the alumina plant. Characterization was performed by XRD, XRF, chemical analysis, TGA and SEM. XRD determined the mineral content: such bauxite is gibbsitic and has been associated with kaolinite and hematite. XRF determined the sample's chemical composition. The chemical content of Al2O3available and SiO2reactiva was determined by potentiometric titration and FAAS. The results found for the Bayer process sample were 41.7% and 7.1%, respectively. TGA observed the bauxite decomposition and SEM supplied chemical and thermal analysis. Thus, based on stoichiometric relations of the bauxite components decomposition, it was possible to confirm the following phases: gibbsite and kaolinite.

2:55 PM
Resource Utilization of High-Sulfur Bauxite of Low-Median Grade in Chongqing China: *Jianguo Yin*[1]; Wentang Xia[1]; Mingrong Han[1]; [1]Chongqing University of Science and Technology

Resource utilization of high-sulfur bauxite is one of technical problems for alumina refineries in China. There are a lot high-sulfur bauxite of low-median grade in Chongqing China, which are not been utilized until now. Sulfur in bauxite will cause many negative effects on the alumina production. There exist some disadvantages for current desulfurization technologies. It is difficult to attain breakthrough progress from the view of desulfurization technologies. So it is necessary to have fundamental theoretical studies on the occurrence of sulfur in high-sulfur bauxite, occurrence, reaction behavior and changing rule for sulfur in the production process and its effect. Combined theoretical research with experiments, it is possible to develop a feasible desulfurization process. It will be technical support and theory guidance for utilization of high-sulfur bauxite in Chongqing, and also advantageous for prolonging bauxite resource guarantee for Chinese alumina industry.

3:20 PM Break

3:30 PM
Development of Bauxite and Alumina Resources in the Kingdom of Saudi Arabia: *AbdulGhafoor Al-Dubaisi*[1]; [1]Saudi Arabian Mining Co. (Maaden)

Driven by desire to diversify its economy in an oil rich country and by the need to create jobs for the increasing number of Saudi youth, the Kingdom of Saudi Arabia established the Saudi Arabian Mining Company (MA'ADEN) to develop its mineral resources. Local bauxite will be developed into an integrated mine-to-metal aluminum industry. Numerous challenges have to be overcome. Major Infrastructure has to be built; construction and operating costs have to be contained to ensure the economic viability of the project. The lack of expertise in the kingdom to run such an operation created the need to involve an international joint venture partner. The human development is no less challenging and early plans have to be in place to recruit and train a large number of Saudis to be the core of the operating organization.

3:55 PM
Digestion Studies on Central Indian Bauxite: *Puliyur Krishnaswamy Narasimha Raghavan*[1]; Nand Kumar Kshatriya[1]; [1]Bharat Aluminium Co. Ltd., (A Unit of Vedanta Resources Plc.,), BALCO Nagar, Korba

Indian bauxite deposits are grouped into five namely, Eastern Ghats, Central Indian, West Coast,Gujrat and Jammu & Kashmir. Each of the Bauxite has its own typical digestion and settling conditions due to variation in minerology. Bauxite digestion depends much on the temperature-pressure conditions, the recycled liquor concentration bauxite mineralogy and charge quantity. The productivity of the entire Bayer process depends to a large extent on the digestion process. In an attempt to optimize the digestion conditions for the central Indian Bauxites, in order to achieve low caustic soda loss coupled with minimum bauxite consumption a series of experiments were conducted to find out the best parameter for conducting digestion at laboratory scale.The effect of deslication and digestion temperature, bauxite charge, lime quantity and digestion liquor MR is discussed in this paper. Attempt shall be made to compare the conditions with Bauxite from other locations.

4:20 PM
Effects of Roasting Pretreatment in Intense Magnetic Field on Digestion Performance of Diasporic Bauxite: *Zhang Ting-an*[1]; Dou Zhihe[1]; Lv Guozhi[1]; Liu Yan[1]; Du Juan[1]; Wang Xiaoxiao[1]; Li Yan[1]; [1]Northeastern University

This paper investigated the changes of phase and apparent morphology under the combined effects of intense magnetic field and temperature field and the effect law of different roasting conditions on the digestion performance of roasted diaspore. The results indicated that the roasting pretreatment under high magnetic field can change the microstructure and improve the digestion properties of bauxite. The reasonable roasting conditions of intense magnetic field follow as that the roasting temperature is 550°, roasting time is 60min and the magnetic field intensity is 6T. The digesting rate of alumina of roasted ore is 84.17% and the digesting liquid ratio is 1.39 while the digesting temperature is 190° with the digestion time of 60mins. The digesting rate of alumina of roasted ore increases higher 52.85% than this of the raw ore. The digesting temperature of roasted ore decreases lower 30° than this of the raw ore.

Aluminum Alloys: Fabrication, Characterization and Applications: Numerical Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
Program Organizers: Subodh Das, Phinix LLC; Zhengdong Long, Kaiser Aluminum; Tongguang Zhai, University of Kentucky

Monday PM
February 28, 2011

Room: 14A
Location: San Diego Conv. Ctr

Session Chair: John Chinella, U.S. Army Research Laboratory

2:00 PM

Modeling Performance of Protection Materials Aluminum 7020-T651 and Steel: *John Chinella*[1]; [1]U.S. Army Research Laboratory

This presentation compares protection and characteristics of aluminum 7020-T651 and a modern rolled homogeneous armor steel (RHA) and compares performance to historical protection levels of RHA. Comparisons are shown with RHA plots of protection level mean response, confidence intervals, and cumulative probability distributions over the range of performance. The protection levels and the failure modes of 7020-T651 aluminum suggest that weight savings will be achieved through: (1) excellent protection, multiple hit or projectile damage tolerance, and minimal need for spall liners; and (2) equal structural stiffness at half the weight of steel. Results of ballistic test characterization in this study, literature of Al-Zn-Mg alloys, Thermo-Calc phase diagrams, and JMatPro temperature step calculations reveals alloy 7020 has unique metallurgical characteristics and capabilities to meet improvements in structural, monolithic or advanced armor designs, and the needs of processing, fabricability or field repair, durability, weight and cost efficiency, and extreme climate.

2:20 PM

The Influence of Temperature on the Tensile Anisotropy of a Forged 7XXX Aluminum Alloy: *Agouti Siham*[1]; Bozzolo Nathalie[1]; Bouchard Pierre-Olivier[1]; Le Brun Pierre[2]; Piellard Mickael[3]; [1]Centre of materials forming; [2]Voreppe Research Center; [3]Aubert et Duval Issoire

Aluminum ingots are usually submitted to hot forging processes to increase their ductility before the final stamping process. The subject of this work is to investigate the effect of temperature on the mechanical anisotropy of a forged 7XXX Aluminium alloy. At the as forged state, this material contains iron-intermetallics observed as large heaps of particles. At room temperature, the longitudinal direction is more ductile than the forging direction as the iron-intermetallics are oriented along the longitudinal direction. On the contrary, tensile tests at high temperatures (300-430°C) showed that the forging direction is more ductile than the longitudinal direction. Microstructure investigations along the strain gradient produced on tensile test specimens allowed to understand deformation and damage mechanisms occurring at high temperature and the resulting mechanical anisotropy.

2:40 PM

Modeling the Strain Path Change Effect in 5754-O Aluminum Alloy Sheet: *Lin Hu*[1]; Anthony Rollett[1]; Mark Iadicola[2]; Tim Foecke[2]; Steve Banovic[2]; [1]Carnegie Mellon University; [2]National Institute of Standards and Technology

Strain-path change tests were performed on 5754-O aluminum alloy sheet sample by using equibiaxial stretch followed by uniaxial tension. A self-consistent viscoplastic polycrystal model was used to simulate the material response under non-linear strain paths and fit the experimental data. Recovery that occurred during the first and second loading steps was also assessed. Anisotropic strain hardening behaviors were incorporated in the model, which accounted for the transient behaviors upon the strain path change.

3:00 PM

Micromechanical Simulations for Fatigue Damage Incubation at Debonded Particles Using Cohesive Zone Model: Tong Li[1]; *Yibin Xue*[1]; [1]Utah State University

Fatigue damage initiates at debonded micrometer-sized intermetallic particles for some high strength Al alloys in the high cycle fatigue regime. Cohesive zone model is implemented to investigate the interfacial debonding and propagation under cyclic loads. Representative Volume Elements (RVE) are set to position the intermetallic particle at different spatial locations regarding the boundary of the RVEs to account for the random distribution of the particle in the alloys. Maximum plastic shear strain range (MPSS) and Fatemi-Socie (FS) parameter for multiaxial fatigue are introduced as nonlocal damage parameters to evaluate the severity of the damage level of RVEs and loading sets. This set of the simulations will be compared with simulation results on fatigue damage incubated at the fractured particles. This effort completes the fatigue damage incubation evaluation for wrought Al alloys.

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Comprehensive Thermo-Mechanical Validation of Extrusion Simulation Cycle for Al 1100 Using HyperXtrude: AbdulAfoo Parkar[1]; Clemence Bouvard[1]; Stephen Horstemeyer[1]; *Esteban Marin*[1]; Paul Wang[1]; Mark Horstemeyer[1]; [1]Center for Advanced Vehicular Systems, Mississippi State University

A laboratory-scale extrusion capability facilitates parametric study of metal extrusion processes. Commercial simulation code, HyperXtrude, was used to simulate laboratory experiments performed using a flat-die. In the experiments, ram velocity and billet temperature were the process parameters that were controlled. Simulation model was validated by comparing the results with experimentally obtained load and temperature histories on tooling. The results of simulation and experiments were found to be in good agreement. 'Optimized' values for friction and convection film-coefficient were used to reach the best fit. The breakthrough load is predicted with good accuracy; however, the accuracy in load correlation is not sustained throughout the process duration as the predicted load trend deviates from experimental data towards the end of the ram stroke. The aim of this work is to benchmark a commercial extrusion simulation code for a range of profiles.

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Deformation and Rupture Modeling of an Aluminum Metal Matrix Composite: *James DeMarco*[1]; Justin Karl[1]; Ali Gordon[1]; [1]UCF MMAE Dept.

While aluminum metal-matrix composites (MMCs) have come into broader usefor high-tech applications where their combined light weights and large elastic moduli are sufficiently advantageous, widespread application is limited by high processing costs. In the case of hot-rolled aluminum MMC sheets, material lost to edge cracking is a major contributor to decreased production yield and high cost. Determining the optimal thermo-mechanical processing parameters which minimize losses due to edge cracking can be achieved through numerical modeling of the temperature-dependent mechanical response of the material. An elastic-viscoplastic constitutive model incorporating strain rate and temperature dependence is presented for A359-SiC_p-30%. Perzyna viscoplasticity and Cockroft-Latham ductility are assumed. Modeling parameters for the as-cast material are optimized to fit tensile and torsion data obtained at a variety of temperature and strain rate combinations.

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Mechanical Properties and Casting Characteristics of the Secondary Aluminum Alloy AlSi9Cu3(Fe) (A226): *Philip Pucher*[1]; Holm Böttcher[2]; Helmut Kaufmann[3]; Helmut Antrekowitsch[1]; Peter J. Uggowitzer[4]; [1]University of Leoben; [2]AMAG Casting GmbH; [3]Austria Metall AG (AMAG); [4]ETH Zurich

Due to its excellent castability, good mechanical properties and cost-effectiveness the secondary alloy AlSi9Cu3 (A226) finds wide application for

the production of permanent mould castings and high pressure die castings (HPDC). Variations of the chemical composition within the tolerance limits cause pronounced changes in the mechanical properties and castability. For example the yield strength of permanent mold cast samples ranges from 100 MPa to 200 MPa, while the elongation varies between 0.3 % and 4 %. The present study was performed to investigate the mechanical properties and casting characteristics depending on the combined effect of the main alloying elements in the whole composition range. The results – based on 48 industry-oriented permanent mold casting experiments – are discussed on a systematic basis of thermodynamic calculations and metallographic investigations. These results were implemented in the software tool AMAG Top Cast® Alloy Designer (TCAD) for the prediction of mechanical properties and casting characteristics.

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Comparison of Different FEM Codes Approach for Extrusion Process Analysis: *Lorenzo Donati*[1]; Luca Tomesani[1]; Nooman Ben Khalifa[2]; A. Erman Tekkaya[2]; [1]University of Bologna; [2]Technische Universität Dortmund

The simulation of the extrusion process by means of FE codes has been applied in a great number of papers available in literature but its application in everyday production was limited due to several factors like computational times, user's skills as well as prediction accuracy. Indeed, the inner complexity of the process, characterized by extremely high deformations, strain rates and heat exchange phenomena, has lead only in the last few year commercial FE codes to gain sufficient accurate solving capabilities. In the paper, five FEM codes based on different approaches were applied in the simulation of the same experiment: the results were compared in term of set-up times, computational time as well as process prediction accuracy. Process load, profile speeds, die and profile temperatures were accurately monitored during the experiment in order to realize an effective comparison of the different FEM codes approaches.

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Numerical Prediction of Grain Shape Evolution during Extrusion of AA6082 Alloy: *Antonio Segatori*[1]; Lorenzo Donati[1]; Luca Tomesani[1]; [1]University of Bologna

Extruded profiles applications require a strict control of the mechanical proprieties of the extrudates, in particular when undergoing severe loading conditions like in the transportation sector. Profile mechanical properties directly depend from its microstructure and texture, which are the result of multiple mechanisms based on precipitation mechanism or on grain shape evolution (grain refinement, recrystallizations, recovery and grain growth). In this direction the opportunity to predict the final profile microstructure under specific process parameters directly in the die design stage is of great relevance.The study involved experimental activity on grain size measurements during interrupted direct extrusion of an AA6082 round profile. The grain size measurements were coupled with the results of the simulation in order to regress analytical models based on effective strain, strain rate and temperature. Finally, the developed model was implemented in the numerical code through user subroutine so as to be used as microstructure prediction tool.

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Analysis of Charge Weld Evolution for a Multi-Hole Extrusion Die: *Antonio Segatori*[1]; Lorenzo Donati[1]; Barbara Reggiani[1]; Luca Tomesani[1]; [1]University of Bologna

Extrusion process presents two types of welding mechanism inside the die: seam and charge weld. The first type is unavoidable in the production of hollow profiles while the second is generated during billet to billet transition. Differently from seam weld, charge weld represent an unacceptable defect that require profile discard. Nevertheless charge weld extent and location on extrudates is not evident, thus requiring scrap definition by means of time-consuming trials or to repetitive tests on each batch. In the paper an industrial four holes porthole die for hollow profile production was investigated during billet to billet transition. Extrusion of four billet was performed under strict parameter control. The profiles were microscopically analyzed in order to determine weld evolution: in particular start and end points of weld for each profile were determined. Finally, weld evolution was studied through HyperXtrude code so as to evaluate weld prediction capabilities and accuracy.

Aluminum Reduction Technology: Environment-Emissions/ Anode Effect I

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

Program Organizers: Mohd Mahmood, Aluminium Bahrain; Abdulla Ahmed, Aluminium Bahrain (Alba); Charles Mark Read, Bechtel Corporation; Stephen Lindsay, Alcoa, Inc.

Monday PM
February 28, 2011

Room: 17B
Location: San Diego Conv. Ctr

Session Chair: Robert Baxter, Bechtel Corp

2:00 PM

HF Measurements Inside an Aluminium Electrolysis Cell: *Karen Osen*[1]; Thor Aarhaug[1]; Asbjørn Solheim[1]; Egil Skybakmoen[1]; Camilla Sommerseth[2]; [1]SINTEF Materials and Chemistry; [2]Norwegian Institute of Science and Technology, NTNU

HF emissions to the working atmosphere may still be a problem for the aluminium industry. The objective in the present work was to study how the HF evolution is distributed between feeder holes, other openings in the crust, gases diffusing through the crust, fumes from the secondary alumina residing on top of the crust etc. A movable "gas sniffer" connected to a Tunable Diode Laser was used to measure the HF concentrations at the above mentioned locations. The stationary HF level in an open flaming feeder hole was approximately 9000 ppm, when measured a few cm above the bath surface. In comparison, when the probe was positioned 5-10 cm above a crust area with good integrity, the HF concentration was in the range 5-10 ppm. The results support the notion that most of the HF evolves at the open flaming feeder holes and tap hole.

2:20 PM

LasIRTM -R – The New Generation RoHS-Compliant Gas Analyzers Based on Tunable Diode Lasers: *Jean-Pierre Gagne*[1]; John Pisano[2]; Alak Chanda[3]; Gervase Mackay[3]; Keith Mackay[3]; Pierre Bouchard[1]; [1]STAS; [2]University of California at Riverside; [3]Unisearch Associates Inc.

The laser-based optical gas sensor using Tunable Diode Laser Absorption Spectroscopy (TDLAS) is rapidly gaining favor wherever high sensitivity, real time measurement and freedom from interferences are required, specifically for the measurement of HF in primary aluminium smelters. It eliminates the problems associated with extractive gas sampling techniques. A first generation of equipment designed by Unisearch Associates and based on the TDLAS appeared on the market in the mid 90s. It has since been improved significantly by employing fast scan measurement techniques. Now, the system is more robust, easier to operate and calibrate, and it can be simply audited. The utilization of fast scan measurement techniques not only provides enhanced stability and sensitivity but expands the dynamic range of the measurements. This new generation instrument is also very inexpensive compared to other TDLAS instruments. This paper describes the evolution of the new generation gas sensor.

2:40 PM

Use of Spent Potlining (SPL) in Ferro Silicomanganese Smelting: *Paulo von Krüger*[1]; [1]Universidade Federal de Ouro Preto

In this work an evaluation on the possibilities of the employ of Spent Potlining (SPL), as a component of the burden of a Submerged Arc Furnace, producing Ferro Silicomanganese Alloy, is investigated. On this subject, a characterization of the SPL's most probable components as well as their interaction with the existing species in the ferroalloy furnace were carried out. Additionally relevant features of the ferroalloy smelting were identified and characterized. Those figures were introduced in a thermochemical

program where the SAF operational conditions were simulated in order to check the technical feasibility of that use.The simulation results showed that, on the technical point of view, the SPL carbonaceous fraction is a suitable component of the SAF burden, producing Ferro Silicomanganese alloys. Although some of the figures generated by simulation were confirmed in a couple of industrial exploratory tests, a more detailed test program is advisable.

3:00 PM

Reduction of PFC Emissions at Pot Line 70 kA of Companhia Brasileira De Alumínio: *Henrique Santos*[1]; Danilo Melo[1]; Jocimar Calixto[1]; Jefferson Santos[1]; João Miranda[1]; [1]Companhia Brasileira de Aluminio

Following world tendency Primary Aluminum Industry has been committed in the reduction of PFC's emissions in order to reduce the greenhouse effect. Anode Effect is the source for such emission. The CBA Pot Room 70kA VSS Montecatini technology from the end of 60's with side feed pots, has been making efforts and developing actions to improve process with the aim of reducing both the duration and frequency of anode effect. Projects were developed and implemented as follow: Liquids control, Resistance control, Feed control, Anode/Cathode control and mainly the team qualify through an operating training. In this paper, are shown achievements for projects cited, that caused significant impacts in reducing the duration and frequency of anode effect at the level of world reference. A reduction of 66 % of PFC's emissions was achieved from 1,2 to 0,4 t CO2 eq./t Al.

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Towards Redefining the Alumina Specifications Sheet – The Case of HF Emissions: *Linus Perander*[1]; Marco Stam[2]; Margaret Hyland[1]; James Metson[1]; [1]Light Metals Research Centre; [2]Aluminium Delfzijl B.V.

For smelting applications, alumina quality is typically defined in terms of chemical and physical properties, with emphasis on impurity elements, surface area, moisture content, particle size distribution and attrition index. However, these properties fail in prediction of the true HF generation potential, as well as the real capacity for HF removal in the dry scrubbers. Using plant measurements and additional laboratory characterization of a number of alumina samples a broadening of how alumina quality is specified is argued for. Measurements of the residual gibbsite/boehmite content and the pore size distribution, coupled with characterization of the alumina microstructure, can be used to predict and understand the generation of HF during feeding and dissolution as well as the ability to capture HF in the dry scrubbers.

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Design of Experiment to Minimize Fluoride and Particulate Emissions at Alumar: *Eliezer Batista*[1]; Paulo Miotto[1]; Edson Montoro[1]; Luciano Souza[1]; [1]Alcoa

Most of Aluminum plants have been struggling to minimize the fluoride and particulate emissions to reduce the environmental impacts. Nowadays, this challenge has been more difficult to be reached because of amperage increase, alumina quality deterioration and pot room expansions. Alumar, one of Alcoa's units, following a corporate vision, is continuously searching for alternatives to the environmental impact caused by its operations.This full factorial experiment 2k identified the main factors and their impacts on fluoride and particulate emissions. The statistical model indicates that the fluoride emission has been affected mainly by Pot Draft, Pot Dressing, and the Usage of Compressed air for Housekeeping with R2 at 82%, and for particulate at 58%. Based on the models, certain actions were recommended to minimize both of these emissions. In addition, this paper describes, step by step, how this kind of experiment can be applied to the Aluminum industry.

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Innovative Distributed Multi-Pollutant Pot Gas Treatment System: *Geir Wedde*[1]; Odd Bjarno[1]; Anders Sorhuus[1]; [1]Alstom

Gas Treatment Centers (GTC) are traditionally arranged in courtyard between pot rooms and handle vast quantities of pot gas (3- 4mill. m3/h) in large number of filter compartments (15-30) with demanding space requirement and challenging control of operation. Arranging the gas treatment in decentralized installations as Distributed Decentralised Scrubbers (DDS) will obviously save on duct work, alumina handling, storage and civil work. In addition Alstom's DS integrates silos, heat exchangers, scrubbers, fans and stack into an extremely compact and efficient multi-pollutant control and recovery technology with incomparable footprint. The close integration with pots improves pot gas collection and simplifies the alumina distribution to pots. Module based design allows for short delivery time and early start-up. This paper discusses and reviews the new technologies, benefits and mitigation of the technical challenges of the DS solution.

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Fluoride Emissions Management Guide (FEMG) for Aluminium Smelters: *Nursiani Tjahyono*[1]; Yashuang Gao[1]; David Wong[1]; Wei Zhang[1]; Mark Taylor[1]; [1]Light Metals Research Centre

All smelters worldwide operate under strict fluoride emission limits and the reduction of fluoride emissions is further driven by health considerations. A Fluoride Emissions Management Guide (FEMG) has been written by the Light Metals Research Centre, on invitation of the Australian Aluminum Council, under the Asia-Pacific Partnership on Clean Development and Climate. The FEMG aims to provide better understanding of factors affecting fluoride evolution and emission in smelters, and further, to provide smelters with an operational guide for reducing and managing fluoride emissions. The guide uses practical examples with pictures of improvements that are applicable to operational, control and maintenance practices in potrooms, GTCs and other smelter systems. It is customizable to meet the need of any specific smelter. Along with incorporated audit guidelines and training packages, implementation of this guide can, in a short time, lead to significant reduction in fluoride emissions and improvement of operational standards in smelters.

4:50 PM

Considerations Regarding High Draft Ventilation as an Air Emission Reduction Tool: *Stephan Broek*[1]; Neal Dando[2]; Stephen Lindsay[2]; Alain Moras[2]; [1]Hatch Ltd; [2]Alcoa

High draft ventilation is an effective technique for reducing emissions from electrolysis cells while panel covers are removed to perform maintenance. In recent years, many new smelters have implemented high draft ventilation as one of the tools to further reduce air emissions from potrooms. In this paper the principles of high draft ventilation are discussed followed by a presentation on its impact on smelter performance. Comments are provided that concern implementation of high draft ventilation in greenfield and brownfield smelters.

Approaches for Investigating Phase Transformations at the Atomic Scale: Transformations in Fe, Ni and Al based Systems I

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
Program Organizers: Neal Evans, Oak Ridge National Laboratory; Francisca Caballero, Spanish National Research Center for Metallurgy (CENIM-CSIC); Chris Wolverton, Northwestern University; David Seidman, Northwestern University; Rajarshi Banerjee, University of North Texas

Monday PM
February 28, 2011

Room: 32B
Location: San Diego Conv. Ctr

Session Chairs: Francisca Caballero, Spanish National Research Center for Metallurgy (CENIM-CSIC); Michael Miller, Oak Ridge National Laboratory

2:00 PM Invited
Nano-scale Analysis of Precipitation in Nitrided Steels: *Tadashi Furuhara*[1]; Goro Miyamoto[1]; [1]Institute for Materials Research, Tohoku University

Microstructure formed during nitriding of steels is a quite complex phenomena because precipitation of fine alloy nitrides occurs in a chemically inhomogeneous matrix under continous supply of nitrogen from the surface. In this presentation, the precipitation behaviors of nano-sized nitrides in various ferritic alloys are discussed based on the results of advanced analysis in atomic scale utilizing HREM and 3DAP.In the alloys containing strong nitride forming elements, precipitation of metastable nitrides occurs. Particularly, phase separation in ferrite is enhanced by strong interaction between the element such as Ti or V and nitrogen, resulting in uniform and fine dispersion of G.P zone like metastable clusters. Transition of those metastable phases to stable nitrides takes place gradually during further nitriding. Presence of excess nitrogen in ferrite is also recognized which might contribute to hardening by nitriding partly. A numerical approach to simulate the precipitation of alloy nitrides will be also presented.

2:25 PM Invited
Atomic Structure of NbN GP Like Zones in a Model Fe-Nb-(C)-N Alloy: *Frederic Danoix*[1]; Thierry Epicier[2]; David Tingaud[3]; Philippe Maugis[4]; [1]CNRS - Université de Rouen; [2]INSA de Lyon; [3]Université Paris 13; [4]Université Paul Cezanne - Marseille

The precipitation of GP like zones in a model Fe-Nb-(C)-N alloy is evidenced on the basis of atomic scale investigations using field ion microscopy (FIM), atom probe tomography (APT) and high resolution electron microscopy (HREM). These techniques support the existence of Nb and N rich monolayers, fully coherent, and lying in the {001} planes of the bcc ferrite (α-Fe). HREM image simulations strongly suggest that Nb substitutes Fe in these monolayers. However, the position of N atoms, and therefore the elementary unit cell of these platelets, is still not fully understood. First-principle calculations, using the VASP software, were used to investigate the stability of possible unit cells, on the basis of the various NbX (X=C, N) bulk phases under strains similar to the coherency strains. The role of structural vacancies in the stabilisation of the NbN platelets is also investigated.

2:50 PM Invited
Nanometric Scale Investigation of Phase Transformations in Advanced Steels for Automotive Applications: *Josée Drillet*[1]; Thierry Iung[1]; Nathalie Valle[1]; [1]ArcelorMittal

The current trend towards vehicle lightening in the automotive industry is driven by the need to conform to the new exhaust emission control regulations. This objective presents a challenge to steel manufacturers. The difficulty lies in designing new alloys with the optimum strength/formability/cost balance for the various components. Here, the key to success lies in controlling the steel microstructure and especially phase transformations at the finest possible scale. Among the different alloying elements, light elements as carbon and boron are of prime importance due to their major effects on the kinetics for phase transformations. Characterization tools combining high spatial and analytical resolution as NanoSIMS and FEG -TEM, have been used. In the paper, examples are presented concerning: • Local carbon distribution in advanced high strength steels, with a specific emphasis on martensite tempering, austenite stabilisation or low density Fe-Mn-Al-C steels.• Boron segregation and precipitation effects to control hardenability.

3:15 PM
Precipitation Strengthening of a Nano-Cluster-Strengthened Ferritic Steel: *Z. W. Zhang*[1]; Ai Serizawa[2]; C. T. Liu[1]; Xun-Li Wang[2]; M. K. Miller[2]; Bryan Chin[1]; [1]Auburn University; [2]Oak Ridge National Laboratory

The strength of Cu-rich nanocluster strengthened ferritic steels is derived mainly from nanocluster precipitation. This gives the ability to control the alloy's strength and ductility through the control of the precipitation process of nanocluster. The studies on the effects of composition and processing routes on the nature and development of these nanoclusters will enhance our understanding of the underlying mechanisms and to enhance the mechanical properties further. In this study, atom probe tomography (APT), small angle neutron scattering (SANS) have been used to study the nanocluster precipitation in FeCuNiAlMn and FeCuNiAlMnMoVTi steels thermally aged up to 500 h at 500□. The microhardness results show a typical dependence on Cu-rich precipitate aging time. APT results show no precipitates in as-quenched specimens while Cu-rich clusters appeared and grew during aging for both steels. The results on cluster composition, number density, size distribution obtained by using APT and SANS were compared.

3:30 PM Break

3:45 PM Invited
Atom Probe Tomography as a Tool to Advance Steels Design and Performance: *Elena Pereloma*[1]; Ilana Timokhina[2]; [1]University of Wollongong; [2]Deakin University

Development of modern steels consisting of complex or nano-scale microstructures with advanced properties requires in-depth understanding of the mechanisms responsible for their microstructure/property relationships. The evolution of microstructure during processing is often associated with various changes taking place at atomic level. These include solute distribution between phases as a result of phase transformations, formation of atmospheres at dislocations, clustering and precipitation phenomena due to various thermo-mechanical processing schedules and/or heat treatments. Atom probe tomography (APT) is invaluable tool for gaining insight into events at atomic scale determining the steel properties. This technique also contributes to the fundamental understanding of phase transformations, which is essential for nano-scale engineering of modern steels and optimization of their performance. In this work application of APT to study solute segregation, clustering and precipitation in TRIP steels and nano-structured bainitic steels after isothermal heat-treatment and after thermo-mechanical processing will be discussed.

4:10 PM Invited
Transformation of Martensite to Austenite During Aging in Steel Studied by Atom Probe Tomography and Simulation: *Dirk Ponge*[1]; Olga Dmitrieva[1]; Gerhard Inden[1]; Julio Millán[1]; Pyuck-Pa Choi[1]; Jilt Sietsma[2]; Dierk Raabe[1]; [1]Max-Planck-Institut für Eisenforschung GmbH; [2]Delft University of Technology

We present atom probe tomography results on phase boundaries between martensite and retained austenite in steel after aging at 450°C. Due to partitioning of Mn and very different diffusibilities of Mn in martensite and austenite a strong accumulation of Mn at the interface is developed. The Mn profile can be explained in terms of the successive motion of the phase boundary into the martensite during aging owing to the formation of new austenite driven by diffusion of Mn into the austenite. This interpretation

is supported by dynamic diffusion calculations where we use the modeling method DICTRA to solve this coupled initial and boundary value problem, as well as the mixed-mode approach involving also the interface mobility. Good agreement between experiment and simulation is obtained by taking into account an increased mobility of Mn in martensite which is attributed to the high defect density of martensite.

4:35 PM

Complementary Use of Transmission Electron Microscopy and Atom Probe Tomography for the Examination of Plastic Accommodation in Nanocrystalline Bainitic Steels: *Francisca Caballero*[1]; Hung-Wei Yen[2]; Michael Miller[3]; Jer-Ren Yang[2]; Juan Cornide[1]; Carlos Garcia-Mateo[1]; [1]CENIM-CSIC; [2]National Taiwan University; [3]Oak Ridge National Laboratory

A displacive transformation involves the motion of a glissile interface. As in work–hardening, its motion can be halted by defects, such as dislocations, stacking faults or twins in the austenite. The defects are created when the shape deformation accompanying bainite growth is accommodated by plastic relaxation of the surrounding austenite. The growing plate stops before it collides with the austenite grain boundary. Because transformation from strong austenite leads to fine plates, alloys can be designed such that the bainite transformation is suppressed to low temperatures (200-350ºC) leading to a nano-scale bainitic microstructure. Complementary high resolution transmission electron microscopy and atom tomography has provided new experimental evidence on the accommodation of transformation strain, subjects critically relevant to understand the atomic mechanism controlling bainitic ferrite growth. Research at the SHaRE User Facility was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

4:50 PM Invited

Effect of Solute Atoms Distribution on the Phase Transformation in Steel: *Genichi Shigesato*[1]; Shunsuke Taniguchi[1]; Taishi Fujishiro[1]; Masaaki Sugiyama[1]; [1]Nippon Steel Corporation

The phase transformation in steel is strongly affected by the distribution of solute elements. Boron is one of the most effective elements to hinder the diffusional transformation from austenite to ferrite. It has been considered that boron atoms segregate on grain boundaries in austenite phase and lower the grain boundary energy resulting in delaying the ferrite transformation. Manganese also impedes the phase transformation. The segregation of Mn atoms on the grain boundaries of austenite disturbs the nucleation of ferrite while the depletion of Mn around Ti oxide inclusions dispersed in austenite phase promotes the nucleation of ferrite transformation. The behavior of distribution of B and Mn atoms in the vicinity of the grain boundaries and the oxide-matrix interface examined with conventional and aberration corrected STEM will be presented and the influence on the phase transformation will be discussed.

5:15 PM

Influence of Forging Conditions on Mechanical Properties of Ti and V-Bearing High Strength Forging Steels and Associated Precipitation Microstructure Characterized by TEM and 3D-Atom-Probe: *Naoyuki Sano*[1]; Tatsuya Hasegawa[2]; [1]Sumitomo Metal Industries, Ltd.; [2]Sumitomo Metals (Kokura), Ltd.

Non-heat treated V-bearing high strength low alloy (HSLA) forging steels have been widely used for machine structural use. To improve mechanical properties further, other alloying elements are added in association with vanadium. It has been found that the addition of 0.15wt.%Ti to a V-bearing HSLA steel with the chemical composition of Fe-0.28C-0.55Si-0.75Mn-0.17V leads to lower the Charpy impact toughness and to increase tensile strength, however this effect is quite sensitive to processing conditions, i.e., hot forging temperatures and subsequent cooling rates. Precipitation microstructure evolved during forging process pronouncedly varied depending on the forging temperature range. At high finishing temperature, aligned fine coherent precipitates of (V, Ti)C carbides originated from interphase interface precipitation developed. However, lower finishing temperature induced coarsened and randomly-distributed precipitate particles, which resulted in the increased impact energy and lower tensile strength.

Biological Materials Science: Bio-Inspiration and Bio-Inspired Materials II: Soft Biomaterials

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Biomaterials Committee
Program Organizers: Jamie Kruzic, Oregon State University; Nima Rahbar, University of Massachusetts, Dartmouth; Po-Yu Chen, University of California, San Diego; Candan Tamerler, University of Washington

Monday PM
February 28, 2011

Room: 15A
Location: San Diego Conv. Ctr

Session Chairs: Paul Calvert, University of Massachusetts; Nima Rahbar, University of Massachusetts Dartmouth

2:00 PM Invited

Gelation of Mucin Glycoproteins: *Rama Bansil*[1]; [1]Boston University

In this talk, I will describe the unusual properties of mucin, a high molecular weight glycoprotein found in mucus, which iprevents the stomach from being digested by the acidic gastric juices. Light scattering and rheological techniques show that purified pig gastric mucin solutions form a gel under acidic pH. Atomic Force Microscopy (AFM) of mucin solutions and gels as a function of pH provide direct visual evidence of aggregation. A model of gelation based on the interplay of hydrophobic and electrostatic interactions will be discussed. Molecular Dynamics simulation studies of folding and aggregation of mucin domains provide further support for this model. As an application I will describe how the motility of H. Pylori, the bacterium that causes ulcers, is influenced by the pH –dependent gelation of mucin, and conversely how the bacterium affects the gelation of mucin.

2:30 PM

Synthesis of Vitrified Collagen Gels with Optimized Transparency and Mechanical Strength for Repair of Ocular Injuries: Jennifer Breidenich[1]; Daniel Mulreany[2]; Freddy Espinoza[2]; Yo-Rhin Rhim[1]; Xiomara Calderon-Colon[1]; Qiongyu Guo[2]; R.L. McCally[1]; *Morgan Trexler*[1]; Oliver Schein[3]; Jennifer Elisseeff[2]; [1]Johns Hopkins University Applied Physics Laboratory; [2]Johns Hopkins University; [3]Wilmer Eye Institute

The frequency of ocular injuries on the battlefield has been steadily increasing during recent conflicts. Combat-related eye injuries are difficult to treat and solutions requiring donor tissue are not ideal. Vitrified collagen gels have been developed for repair of ocular injuries, but increased transparency and mechanical strength are desirable for improved vision and ease of handling, respectively. Synthesis of collagen vitrigels was optimized to yield the best combination of high transparency and high mechanical strength. Vitrification parameters that were investigated included temperature, humidity, and vitrification time. Transparency as well as tensile and tear strengths were characterized for each combination of synthesis conditions to evaluate the effects of the vitrification parameters on material properties. Changes in denaturing temperature and collagen fiber organization were also evaluated to correlate properties with structure. Preliminary results of ocular injury repair studies utilizing vitrigels with optimized properties will also be reported.

2:50 PM Invited

Progress Towards Cartilage Engineering Using Peptide Hydrogels: *Joel Schneider*[1]; [1]National Cancer Institute

Hydrogel materials are finding use for the encapsulation and delivery of mammalian cells for tissue regenerative therapy and cytomedical applications. We have designed a class of small peptides that undergo a triggered intramolecular folding event to form an amphiphilic beta-hairpin conformation that is prone to self-assembly. Self-assembly leads to the formation of a physically crosslinked network of beta-sheet rich fibrils that

constitute a mechanically rigid hydrogel. When peptide folding and self-assembly is triggered in the presence of cells, they are directly encapsulated in a uniform and cytocompatible manner. Peptide hydrogels, with or without cells, are shear thinning but have the capacity to quickly re-heal after the cessation of shear. This attribute allows hydrogel loaded with cells to be easily delivered from a syringe/catheter. Primary chondrocyte encapsulation and delivery is demonstrated and phenotype response studied, highlighting our efforts towards cartilage engineering.

3:20 PM
Molecular Biomimetics Enables Biological Materials Science and Engineering: *Mehmet Sarikaya*[1]; [1]University of Washington

Proteins enable biology to be viable through molecular interactions. Using biology as a guide, we biocombinatorially select, bioinformatically enhance and genetically tailor solid binding peptides and utilize them as molecular building blocks in carrying out molecular and nanomaterials science and engineering through biology. In this emerging field of molecular biomimetics, genetically engineered peptides for inorganic materials (GEPI) are used as bionanosynthesizers in biomaterialization, heterofunctional linkers to create thermodynamically stable interfaces between dissimilar materials, and as molecular assemblers for the targeted and directed assembly of nanomaterials towards addressable ordered architectures with genetically designed functions. Here, we will give an update of the utility of various kinds of GEPIs in nanoparticle formation for hybrid probe design and for bionanosensors; biomineral formation for tissue regeneration, and graphite-binding peptides for nano-electronics to demonstrate the power of the approach enabling nanotechnology and nanomedicine through materials science. Funded by NSF-MRSEC and NSF-BioMat programs.

3:40 PM Break

3:50 PM Invited
Hydrogels Formed by Inkjet Printing through Ionic Self-Assembly for Tissue Engineering and Drug Delivery: Skander Limem[1]; Donald McCallum[2]; David Kaplan[3]; Marc in het Panhuis[2]; Gordon Wallace[2]; *Paul Calvert*[1]; [1]University of Massachusetts; [2]University of Wollongong; [3]Tufts University

Self-assembled ionic hydrogels are familiar when formed by the layer-by-layer method, which adds a monolayer of each polymer on every dipping cycle. We have been using inkjet printing to form similar gels at a rate 100x faster by printing anionic (polystyrenesulfonate) and cationic (polydimethyldiallylamine hydrochloride) polymers sequentially from a two-color printer. Gels of this type are not well understood because they have not previously been formed in bulk. We have characterized the swelling and dye-release properties and find that these change depending on the relative proportions of the two polymers and the extent of annealing of the structure. Similar gels can be formed from carboxylate polymers such as alginate and hyaluronic acid with chitosan and from polyglutamic acid with polylysine. Growth of cells on and in these gels is also being studied as a model for intercellular matrix.

4:20 PM
Fabrication of 3D Hydrogel Matrices Containing Yeast and Human Cells: *Paul Calvert*[1]; Swati Mishra[1]; Dapeng Li[1]; [1]University of Massachusetts Dartmouth

Acrylate hydrogels containing yeast and mammalian cells are formed by a blue-light activated polymerization that does not damage the cells. The viability and metabolic rate of the cells entrapped in the gel are determined and related to the thickness of the gel layers. Using a monomer paste thickened with nanoparticulate silica, 3D porous structures can be built This is viewed as a route to tissue mimics and bioreactors but methods must be developed to control cell multiplication in order to avoid disruption of the gel layers.

4:40 PM
Synthesis of Cellulose Hydrogels with High Strength and Transparency for Use as an Ocular Bandage: *Morgan Trexler*[1]; Jenna Graham[1]; Jeffrey Maranchi[1]; Jennifer Breidenich[1]; Russell McCally[1]; Marcia Patchan[1]; Freddy Espinoza[2]; Jeremy Chae[2]; Iossif Strehin[2]; Oliver Schein[3]; Jennifer Elisseeff[2]; [1]Johns Hopkins University Applied Physics Laboratory; [2]Johns Hopkins University; [3]Wilmer Eye Institute

Cellulose is a biologically-derived material with excellent wound healing properties. The high strength of cellulose fibers and the ability to synthesize gels with high optical transparency makes these materials amenable for ocular bandage applications, which are becoming increasingly important in combat. Hydrogels were synthesized from cellulose derived from plant and bacterial sources. Material properties including mechanical strength, optical transparency, oxygen permeability, water content, and contact angle were evaluated, as these parameters are all critical for ocular applications, and compared with the natural cornea. Based on these materials characterization results and biocompatibility tests, cellulose types were down-selected for use in a temporary ocular bandage. The resulting cellulose gels were then functionalized with amine groups to achieve compatibility with a previously developed ocular adhesive. Results from preliminary in vivo tests will also be presented.

5:00 PM
Fabrication of a Cellulosic Nanocomposite Scaffold with Improved Supermolecular Structure as a Potential Cardiovascular Tissue-Engineered Graft: *Parisa Pooyan*[1]; Hamid Garmestani[1]; Rina Tannenbaum[1]; [1]Georgia Institute of Technology

Cellulose nanowhiskers (CNWs) with its renewable and environmentally benign nature, and its abundance and excellent biocompatibility could potentially open a new avenue in cardiovascular tissue engineering for small caliber grafts. Inspired by this bioapplication, we have designed a fully bio-based nanocomposite of aligned CNWs embedded in a matrix of cellulose acetate possessing a controlled biodegradability, 3D porosity, and non-acidic byproducts as opposed to degradable PLA/PGA. To ensure uniform distribution, CNW were delicately extracted from a multi-stage process and dissolved in a solvent of choice prior to mixing with the matrix to inhibit whiskers flocculation. Comparable to Carbon Nanotubes and Kevlar, CNWs imparts significant strength and directional rigidity to the composite even at 0.2 wt% yet intensifies to about 40% within a controlled magnetic field of 0.3T. We believe our novel aligned nanocomposite could have ground-breaking features withstanding the physiological pressure and mimicking the topographical texture of the native extracellular graft.

5:20 PM
Lessons from Nature: Biomimicry of Leaf Surfaces: *John Nychka*[1]; [1]University of Alberta

The biological environment is vast yet the common denominator for life is water. While water is a requisite for most life on Earth too much water can be deleterious; for example plant leaf surfaces must remain dry to maintain gas exchange and prevent rotting. Various plant surfaces showing different wetting behavior will be described in this presentation; analysis of the structure of the epicuticular wax morphology on leaf surfaces will be discussed in the context of its effect on wetting behavior. Biomimicry of certain leaf surfaces using engineering materials will be described along with potential biomaterials applications of such technology.

Bulk Metallic Glasses VIII: Alloy Development and Application II

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

Program Organizers: Gongyao Wang, University of Tennessee; Peter Liaw, Univ of Tennessee; Hahn Choo, Univ of Tennessee; Yanfei Gao, Univ of Tennessee

Monday PM
February 28, 2011

Room: 6D
Location: San Diego Conv. Ctr

Session Chairs: J. Eckert, IFW Dresden; Jinn Chu, National Taiwan University of Science and Technology

2:00 PM Invited

Ti-Based Bulk Metallic Glasses and Composites for Structural and Functional Applications: *J. Eckert*[1]; [1]IFW Dresden

Ti-based bulk glassy alloys and composites have potential for use in various fields due to the low density of the main constituent element Ti and high corrosion resistance. Their ultimate strength exceeds the values achievable for conventional microcrystalline alloys significantly. However, the strong monolithic amorphous/nanostructured alloys usually lack ductility, and an additional toughening phase is often needed to improve plasticity and to promote local shear events to be distributed more homogeneously. Recent results obtained for the structural and compositional analysis of the microstructures of Ti-based bulk metallic glasses and composites will be presented. The deformation behaviour and possible phase transitions during deformation and upon heating to elevated temperatures will be related to the structure of the glass in order to derive guidelines for the design of macroscopically ductile high-strength materials. The role of mechanically induced phase transitions and twinning of crystals for improving the ductility will be critically discussed.

2:20 PM

Evaluation of Physical and Mechanical Properties of Metallic Glasses in Micro/nano Scales: Jinn Chu[1]; *Yen-Chen Chen*[1]; Jason Jang[2]; Tsong-Ru Tsai[3]; Hidemi Kato[4]; [1]National Taiwan University of Science and Technology; [2]National Central University; [3]National Taiwan Ocean University; [4]Tohoku University

Recently, the process development of metallic glasses and the improvement of their mechanical properties have attracted considerable attention from the view point of potential applications. However, for industrial applications, the important issues that need to be overcome are their size limitation and room-temperature ductility. From this point of view, metallic glasses (MGs) exhibit viscous flow at high temperatures in the supercooled liquid region; thus they can be used to produce micro-devices via micro-/nano-imprinting. In this study, we demonstrate that various micro-/nano-patterns of Pd-based BMG can be imprinted from a mold in air. In addition, the physical and mechanical properties of the BMGs are evaluated. The obtained results suggest that the BMG is a suitable material for micro-/nano- imprinting, and is also a good mold material for imprinting.

2:30 PM Invited

Alloying Effects on Glass-Forming Ability and Soft Magnetic Properties of Fe-Based Bulk Metallic Glasses with a High Fe Concentration: H. X. Li[1]; J. E. Gao[1]; Z. B. Jiao[1]; *Z. P. Lu*[1]; [1]University of Science and Technology Beijing

Compared to the commercial silicon steels, Fe-based amorphous and nanocrystalline alloys which usually exhibit lower coercive force and core loss are attracting more and more attention as the transformer cores for high energy efficiency. However, the low glass-forming ability makes them available only as ribbons, powders or wires, as demonstrated by commercial products of FINEMET and NANOPERM alloys. In this talk, effects of metallic elements on the ferromagnetic system Fe-(P,C,Si,B) will be summarized and a group of newly developed soft magnetic Fe-C-Si-B-P-TM (TM-transition metals) bulk amorphous alloys with a unique combination of super-high saturation magnetization (>1.5T), high glass-forming ability (=1mm) and high compressive fracture strength (= 3GPa) will be reported. Alloying effects on glass-forming ability and soft magnetic properties will also be discussed in detail.

2:50 PM Invited

Superplastic Deformation at Nanoscale in Metallic Glass: *Scott Mao*[1]; J. Luo[1]; Jianyu Huang[2]; [1]University of Pittsburgh; [2]Sandia National Lab

Metallic glasses are regarded as potential engineering materials for their high strength, hardness and large elastic strain. However, due to their high plastic instability, metallic glasses fail catastrophically once the shear band propagates throughout the specimen body, leading to near zero global plastic strain at room temperature. However, if the "brittle" metallic glass has extremely small dimensions at tens of nanometres, the shear bands will no long operate, the flow and fracture mechanisms may change. Here we show that Al90Fe5Ce5 metallic glass with nanoscale size can undergo superplastic deformation with tensile elongation of ~200%. Finally necking occurred without shear bands in the nanoscale sample with the area reduction nearly 100%. Remarkably, even an atomic chain can be formed in the necking area approaching fracture, which is never found in metallic glass systems. Our discovery is fundamentally important for understanding the physics nature of metallic glasses in small scale.

3:10 PM

Atomic Mobility Inside Shear Bands and the Impact on Tracer Diffusion, Nucleation and Growth: *Gerhard Wilde*[1]; Joachim Bokeloh[1]; Gerrit Reglitz[1]; Harald Rösner[1]; Sergiy Divinski[1]; [1]University of Muenster

Although the occurrence of shear bands during plastic deformation of metallic glasses is well known, their actual physical properties remain fairly unknown. Here, experimental data on the rate of atomic diffusion within shear bands has been obtained using the radiotracer method on post-deformed specimens. The experimental results indicate unambiguously that the diffusivity is largely enhanced as compared to volume diffusion in metallic glasses. Moreover, nanocrystal formation has been observed in metallic glasses during different deformation processes. This finding has generated great interest as well as controversial discussions concerning the underlying mechanism. Here, different deformation methods with largely different strain and pressure levels have been applied on metallic glasses to investigate the deformation-induced nanocrystallization reaction with the strain as a metric for the transformation. The experimental results are discussed with respect to the underlying mechanism, utilizing also the first experimentally determined values of the diffusivity within a shear band.

3:20 PM Break

3:30 PM Invited

Bending-Ductility Improvements of Bulk Metallic Glass by Surface Coatings: *Jinn Chu*[1]; Bo-Shian Houng[1]; Jason S. C. Jang[2]; Yin-Yu Chang[3]; Peter K. Liaw[4]; Yoshihiko Yokoyama[5]; Akihisa Inoue[5]; [1]National Taiwan University of Science and Technology; [2]National Central University; [3]Mingdao University; [4]The University of Tennessee; [5]Tohoku University

Room-temperature mechanical properties of bulk metallic glasses (BMGs) are characterized by excellent strength and elasticity with a lack of plasticity. Many attempts have been made to improve the ductility of BMGs, including surface modifications by shot peening. Our present study reports the use of Zr-based metallic-glass thin films as a promising coating to enhance the BMG bending ductility. The Zr-based BMG substrate is sputtered and coated with a 200 nm-thick Zr-based film and thin Ti adhesive layer. The coated sample has a maximum four-point bending loading of ~ 7 KN, a rise of ~ 75% from the uncoated one, with the 10-fold increased toughness to ~ 1,432 J and surface elongation reaching ~ 10%. Fractographic studies reveal that shear bands become highly populated for the coated BMG, suggesting that the ductility is closely related to the multiplication of the shear bands in the BMG substrate.

3:50 PM
Characterization and Mechanical Response of Amorphous $Fe_{45}Ni_{45}Mo_7B_3$ Honeycombs: *Balaji Jayakumar*[1]; Jay Hanan[1]; [1]Oklahoma State University

The high yield strength and elastic modulus of metallic glasses implies that they could perform an important role in structural applications. In order to produce materials with a high strength-to-weight ratio and excellent energy absorption capabilities, it is advantageous to form amorphous alloys as cellular solids. Using the elastic property of slip cast amorphous $Fe_{45}Ni_{45}Mo_7B_3$ ribbons, a metallic glass honeycomb was manufactured with a unique manufacturing approach. First prototypes were manufactured with a porosity of 96%, cell wall thickness of 0.028 mm and a cell size of 3 mm. We report ongoing studies of the mechanical properties of $Fe_{45}Ni_{45}Mo_7B_3$ honeycombs. Experimentally measured mechanical properties are comparable to that of existing analytical models. Also, suggesting that potentially a 3X improvement in the strength properties in the out-of-plane direction are achievable when compared to that of crystalline Aluminum and Nomex honeycombs.

4:00 PM Invited
Air-Oxidation of a $[(Co_{50}Cr_{15}C_{15}Mo_{14}B_6)_{97.5}Er_{2.5}]_{93}Fe_7$ Bulk Metallic Glass at 600 - 725 °C: *Wu Kai*[1]; P.C. Lin[1]; Y.H. Wu[1]; W.S. Chen[1]; Z.Z. Liang[1]; H.L. Jia[2]; P.K. Liaw[2]; [1]Institute of Materials Engineering, National Taiwan Ocean University; [2]Department of Materials Science and Engineering, The University of Tennessee

The oxidation behavior of a $[(Co_{50}Cr_{15}C_{15}Mo_{14}B_6)_{97.5}Er_{2.5}]_{93}Fe_7$ bulk metallic glass (Co7-BMG) was studied over the temperature range of 600 – 725 °C in dry air. Nearly no oxidation was observed after oxidation at 625 °C for 36 h, indicative of an excellent oxidation resistance of the amorphous alloy. In addition, the oxidation kinetics of the Co7-BMG followed a two- to three-stage parabolic-rate law at T > 650 °C, having its steady-state parabolic-rate constants (kp values) increased with temperature. The scales formed on the Co7-BMG were very thin, consisting of an intermixed layer-structure mostly of $CoMoO_4$, minor amounts of Cr_2O_3, $CoCr_2O_4$, and uncorroded Co_3B at T > 650 °C. The formation of Co_3B implied that a phase transformation of the amorphous substrate was occurred during oxidation at the temperature of interest.

4:20 PM
Deformation Behaviour of Metallic Glasses: *B Vishwanadh*[1]; R Tewari[1]; S Sharma[1]; R Kishore[1]; G K Dey[1]; [1]Bhabha Atomic Research Centre

The present paper reports on the influence of deformation on the structure and on mechanical properties of rapidly solidified ribbon (RSR) and bulk metallic glass (BMG) of the $Zr_{52}Ti_6Al_{10}Cu_{18}Ni_{14}$(at%) alloy. The structure of as-solidified as well as deformed glasses has been probed by X-ray diffraction, transmission electron microscopy, positron annihilation, differential scanning calorimetry and nano indentation. Through these studies it has been established that the as-solidified structure is different at the different regions of BMG. For example, the peripheral region had lower medium range order (MRO) and higher free volume in comparison to the central region leading to higher hardness at periphery than in the central region. Through nano-indentaion experiments, it has been demonstrated that subsequent to deformation BMG undergoes work softening whereas RSR undergoes work hardening. These behaviors have been explained in the present paper on the basis of change in free volume and MRO.

4:30 PM Invited
Deformation Behavior and Deformation-Induced Nanocrystallization of Bulk Metallic Glasses: *Tao Zhang*[1]; [1]Beihang University

For extensive applications of bulk metallic glasses (BMGs) with high strength as structural materials, development of BMGs with good plasticity and understanding the mechanism of their plastic deformation are of great importance, which have been widely studied in the last decade. We will review our recent work on the syntheses and deformation behavior of Cu-, Ti- and Zr-based BMGs with good plasticity. Especially, the Zr-based BMG exhibited remarkable work hardening besides plastic strain of ~20% in compression. For understanding the underlying mechanism of the deformation behavior of the present BMGs, the structural changes induced by deformation as well as thermal stability of the BMGs were studied. It was found that deformation-induced nanocrystallites in glassy matrix as well as their crystalline defects could be responsible for the improved deformation behavior.

4:50 PM
Dependence of Fracture Toughness on the Configurational State of a Metallic Glass: *Glenn Garrett*[1]; Jin-Yoo Suh[1]; Michael Floyd[2]; Angeliki Kapoglou[3]; Marios Demetriou[1]; William Johnson[1]; [1]CalTech; [2]UC Berkeley; [3]European Space Agency

The fracture toughness of a metallic glass is thought to be determined by the ability of the glass to undergo extensive plastic shearing by hopping across a potential energy landscape while simultaneously resisting crack formation. Here, the fracture toughness of Zr35Ti30Cu8.25Be26.75 relaxed at different configurational states in the potential energy landscape was measured using single-edge-notch bending specimens. Key potential energy landscape variables such as the elastic constants and stored enthalpy were measured at each configurational state, and correlated to fracture toughness. The fracture toughness is found to correlate more strongly with the shear elastic constant, thereby revealing that toughness is limited predominantly by the resistance of the glass to undergo plastic shearing.

5:00 PM Invited
Dynamic Mechanical Behavior of Bulk Metallic Glass and Its Composite: Morgana Trexler[1]; *Naresh Thadhani*[2]; [1]The Johns Hopkins University Applied Physics Laboratory; [2]Georgia Institute of Technology

The mechanical behavior of bulk metallic glass and its composite with tungsten particles is investigated as a function of dynamic uniaxial-stress and uniaxial-strain conditions. Anvil-on-rod impact tests instrumented with high-speed digital photography and velocity interferometry, were performed to determine the localized deformation and failure response, and elastic-plastic wave propagation characteristics. Imaging of transient deformation states and sample back surface velocity profiles obtained from the experiments were correlated with predictions from numerical simulations to validate the applicability of the Stassi-Drukker-Prager constitutive model used for describing the behavior of the BMG and the composite. Uniaxial-strain shock loading experiments were also performed to obtain evidence of shock-induced polymorphism which was observed to result in increase in strength and failure stresses. The state of the understanding of high-strain-rate deformation and shock compression response of bulk metallic glasses and their composites will be discussed in this presentation. [Research funding provided by U.S. Army Research Office].

5:20 PM
Dynamic Compressive Behavior of Fe Based Amorphous Metal Honeycomb Cellular Structures: *Advait Bhat*[1]; Jay Hanan[1]; Ganapathi Mahadevan[1]; [1]Oklahoma State University

Cellular solids like honeycombs have potential applications as energy absorbers. The typical applications are in dynamic stress environments. Quasistatic compression behaviors of honeycombs have been widely explored, however, their dynamic compression properties remain largely unknown. Also, no prior testing has been carried out on amorphous metal based honeycombs. The dynamic compressive behavior of a Fe45Ni45Mo7B3 amorphous metal based honeycomb was investigated and mechanical properties were compared to those at quasistatic strain rates. The results indicate significant strain rate sensitivity. The observations can be compared to those seen in amorphous metal foams tested under similar conditions. The energy absorption capacity of this honeycomb is found to exceed that of aluminum counterparts of similar relative density.

5:30 PM Invited
Atomic-Scale Structural Heterogeneity, Percolation and Unified Yielding Behavior in Metallic Glasses: *Yong Yang*[1]; Jianchao Ye[1]; C.T. Liu[1]; Jian Lu[1]; [1]the Hong Kong Polytechnic University

Recent studies indicate that metallic glasses (MGs) can be viewed as atomic-scale composites containing soft free-volume zones and hard atomic clusters. In this study, the dynamic microcompression method developed recently was

utilized to characterize the atomic-scale structural heterogeneity of MGs and further to understand their yielding mechanisms. Based on the experimental findings, a percolation model was constructed to quantify the observed deformation-controlled yielding phenomenon in MGs. The experimental and theoretical results clearly show that the apparent universal yielding strain of ~2%, as measured from a wide range of MG alloys, originates from a percolation process that involves the destabilization of the sharing zones among solute-centered atomic clusters. The deformation mechanism proposed here is basically consistent with the spirit of the potential energy landscape (PEL) concept and provides an insight into the structure-property relationship in MGs, which has been remaining as a longstanding unresolved scientific problem for many years.

5:50 PM

The Oxidation Behavior of an FeCo-Based Bulk Metallic Glass at 600 - 700°C: *Wu Kai*[1]; Pin-Chen Lin[1]; Yan-Hao Wu[1]; Z.Z. Liang[1]; P.K. Liaw[2]; [1]Taiwan Ocean University; [2]University of Tennessee

The oxidation behavior of an $[(Fe_{50}Co_{50})_{75}B_{20}Si_5]_{96}Nb_4$ bulk metallic glass (FeCo5-BMG) was studied over the temperature range of 600 – 700 °C in dry air. The results showed that nearly no oxidation was observed for the FeCo5-BMG after oxidized at 600 °C for 36 h, indicative of a better oxidation resistance. In addition, the oxidation kinetics of this glassy alloy followed a two- to three-stage parabolic-rate law at higher temperatures, having its parabolic-rate constants (k_p values) increased with temperature. The scales formed on the FeCo5-BMG were very thin, consisting of an intermixed layer-structure of Fe_3O_4, Co_3O_4, B_2O_3, and uncorroded Fe_3B. The formation of Fe_3B further indicated that a phase transformation of the amorphous substrate was taken place during oxidation at the temperature of interest.

Cast Shop for Aluminum Production: Casthouse Productivity and Safety

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

Program Organizers: Geoffrey Brooks, Swinburne University of Technology; John Grandfield, Grandfield Technology Pty Ltd

Monday PM
February 28, 2011

Room: 16A
Location: San Diego Conv. Ctr

Session Chairs: Leonard Aubery, SELEE Corporation; David De Young, Alcoa

2:00 PM Introductory Comments

2:10 PM

New Casthouse Smelter Layout for the Production of Small Non-Alloyed Ingots: Three Furnaces/Two Lines: Jacques Berlioux[1]; Arnaud Bourgier[1]; Jean-Louis Baudrenghien[1]; Christian Jonville[1]; *Nicolas Tardy*[1]; [1]Rio Tinto Alcan

This paper deals with a layout for a smelter casthouse producing small non-alloyed ingots.The layout of aluminium casthouses has important consequences with respect to safety, investment and operating cost. Traditional solution for casthouse producing non-alloyed ingots is two furnaces per one ingot line. In order to improve the economics and logistics the casthouse a detailed study was done, which required: detailed performance analysis, in particular relating to furnace/ingot line connections, impact on maintenance, operations, etc, hazard study, modeling of metal flow from tapping in the potline through to the end-product, including breakdowns and downgraded operation, thermal and flow modeling in the launder, in steady state and temporary operation and detailed determination of the launder. It was demonstrated that a layout based on such a configuration is superior in terms of full economic cost. This design is now part of our casthouse basic engineering package.

2:35 PM

Use of Process Simulation to Design a Billet Casthouse: *Gwenola Jaouen*[1]; [1]Rio Tinto - Aluval

Designing a casthouse for billet is a complex activity. The billet casthouse must be properly sized to optimize the metal flow, and continuously feed the homogenization shop, while bearing in mind that oversizing adds no value and is costly. To solve this problem, it is necessary to accurately analyze the operations of the casthouse in real time and this requires a discrete simulation model. A model has been developed with a library that includes all the necessary equipment: conveyors, continuous and batch furnaces, finishing stations. The model was used to design an expended casthouse which requests the management of complex product mixes and which challenges the robustness and flexibility of the installation. By combining this model with the metal flow sizing model, it was possible to validate the performance of the design and its impact on casthouse operation. This resulted in a shop designed for customer needs, at optimal cost.

3:00 PM

Optimizing Scrap Reuse as a Key Element in Efficient Aluminium Cast Houses: *Tom Schmidt*[1]; Jan Migchielsen[1]; [1]Otto Junker

In the last decades, the market share of secondary aluminum is slowly replacing primary aluminium. However, as the demand for aluminum is still considerable larger than the collection of used aluminium, primary aluminium will also be needed for the coming decades. This paper will address the current position of this development and the possibility to further increase the part of used aluminium scrap as the source for production of new aluminium to come to a durable production. The second part of the paper will concern the melting facilities for recycling used scrap.

3:25 PM

Implementation of an Effective Energy Management Program Supported by a Case Study: *Roger Courchee*[1]; [1]K B Alloys, LLC

Effective energy management programs require commitment and leadership from Upper Management supported by an experienced Energy Manager. The vehicle for change is the Energy Management Working Party. The Working Party will be led by a Senior Manager and include other line managers under the technical guidance the Energy Manager. The first step is to use historical data to calculate an energy management performance indicator called Specific Energy Consumption [SEC] and then continue the calculations using real time data. The Working Party will initiate programs to achieve a continuous improvement in the SEC and in conjunction with Upper Management agree long term targets. Techniques will include evaluating base load data (energy consumed when not in production), projects targeted at energy inefficient equipment and a review of the various manufacturing processes to develop low energy solutions. This presentation is supported by a case study of the KB Alloys facility at Wenatchee WA.

3:50 PM Break

4:00 PM

Molten Metal Safety Approach through a Network: *Christian Pluchon*[1]; Bruno Hannart[1]; Laurent Jouet-Pastré[1]; [1]Alcan Engineered Product

Molten Metal explosion or splash is a major risk encountered in the aluminium casthouses. Alcan EP has had to face an accident in 2006, in one explosion in its casthouse. Unfortunately an employee had 2nd degree burns on the face. After expertise of the accident and implementation of corrective action in the plant, it appeared that we had a potential of improvement in management of Molten Metal risk by sharing experience coming from the merged companies,Alcan, Pechiney and Algroup.The EP management decided to create a network constituted of experienced managers of most of the casthouses.It was key to look deeply in the details and to think to the practical aspects of every decision.A novel approach of the management of Molten Metal risk was born in the company. The system was very powerful to align the whole organization on the objective to minimize risks.

4:25 PM

Improved Monolithic Materials for Lining Aluminum Holding and Melting Furnaces: *Andy Wynn*[1]; John Coppack[1]; Tom Steele[1]; Ken Moody[1]; [1]Thermal Ceramics

To remain competitive, aluminum producers continue to increase productivity through their Melt-Hold furnaces. Increasing heat input to the furnace using more powerful burners is common practice. But faster melting leads to increased metal losses from surface oxidation and to segregation from large heat gradients. These effects are countered by increased use of fluxes and increased stirring. Given the increasingly challenging environment within which the refractory lining has to work, traditional lining solutions can no longer be relied upon to provide the service lives that were previously achieved. Therefore, a new generation of furnace lining materials is required to cope with today's aluminum furnace. This work reports on a new monolithic material with improved performance, compared to existing materials, suitable for use in the critical metal contact areas of aluminum furnaces. Improved corundum growth resistance, salt resistance and thermal shock resistance are demonstrated in the laboratory using industry standard test methods.

4:50 PM

Cost of Poor Quality in Aluminium Cast House Processes: *Puliyur Krishnaswamy Narasimha Raghavan*[1]; [1]Bharat Aluminium Co. Ltd., (A Unit of Vedanta Resources Plc.,), BALCO Nagar, Korba

Over 30 years aluminium casthouse technology has been driven by a number of factors such as, Competition from alternative materials, Lightweighting, Market requirements for enhanced properties affecting gas levels, impurities, inclusions, physical and chemical properties, ease of downstream processing, reduced cost and improved delivery, Reduction in conversion cost by various means including capacity increase, maximising asset utilization, minimizing scrap, reducing melt loss, labour, reduction in inspection with improved processes control activity, energy costs and need for improved safety performance. This paper will explore how these, and related considerations have provided the stimulus for improved casthouse technology by reducing Cost Of Poor Quality (COPQ). An attempt has been made to monitoring the COPQ and there by brining in improvement in the quality of the Aluminium Cast House products with a competitive reduction in Cost of production COP).

Characterization of Minerals, Metals and Materials: Mineral Processing and Analysis

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee
Program Organizer: Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF

Monday PM
February 28, 2011

Room: 14B
Location: San Diego Conv. Ctr

Session Chairs: Donato Firrao, Politecnico di Torino; Joner Alves, University of Sao Paulo

2:00 PM

A Study on a Greensand Slate from Abaeté as a Source of Potassium: Thermal Treatment on Different Temperatures and Extraction on Acidic Media: *Adriana da Silva*[1]; Francisco Garrido[1]; Marta Medeiros[1]; João Sampaio[2]; [1]UFRJ; [2]CETEM

Brazil is a great agricultural potential and its crop success is related to the soil's chemical and physical characteristics. The production of potassium fertilizers is a major concern because in 2009 Brazil produced only 10% of its needs and spent almost US$ 2 billion on the purchase ofthis fertilizer. In this context, a potassium rock has been studied in order to evaluate its use as an alternative source of potassium to the Brazilian agriculture. This rock has been thermal treated with CaO (from 10 to 30%) at different temperatures (from 800 to 1200ºC) and its structural modifications have been supervised using x-ray diffraction and infrared spectroscopy. At the temperature of 1200ºC and 10% of CaO, it was possible to observe the formation of new phases, such as spinel, that could improve the potassium availability that was quantified on acidic extractions using citric acid.

2:15 PM

Application of High-Power Nanosecond Pulses to Flotation Separation of Sulfide Minerals: *Igor Bunin*[1]; Valentine Chanturiya[1]; Alexey Kovalev[1]; Irina Khabarova[1]; Elizaveta Koporulina[1]; [1]Research Institute of Comprehensive Exploitation of Mineral Resources RAS

The paper presents new theoretical and experimental results about mechanisms of disintegration of mineral complexes and structural transformations of the sulfide surface under high-power nanosecond electromagnetic pulses (HPEMP). The heated gas outflow from nanosecond breakdown channels of sulfide minerals under HPEMP is considered. It is shown that the gas outflow from channels can be an additional destructive factor in the processes of the electric pulse discharge disintegration of mineral complexes. It is shown that the effect of HPEMP changes chemical surface composition and, respectively, technological properties of pyrrhotite and pentlandite. Morphology and elementary composition of new micro- and nanoformations on mineral surface of pentlandite and pyrrhotite have been investigated using up-to-date methods of SEM/EDX and Scanning Probe Microscopy. Preliminary electropulse effect on mineral products before flotation allows producing optimal conditions for flotation separation of pentlandite and pyrrhotite owing to forming the new nanophases and defects on the surface of sulfides.

2:30 PM

Sulfur Deposition Impact on Surface Morphology of Fly Ash-Based Adsorbent for Mercury Removal: Yaohua Chen[1]; Wei Gao[1]; Qingcai Liu[1]; Cunfang Lu[1]; Jian Yang[1]; [1]University of Chongqing

Mercury is a toxic air pollution, and coal-fired utility plants are the largest anthropogenic emission source in China. Due to rapidly decreasing the cost and increasing the efficiency, it is becoming necessary to develop alternative technologies/materials for mercury removal. The adsorbents made from fly ash of coal-fired, and became spherical after sintering at temperature 500□. Finally, it has been treated by thermal precipitation sulfur, to remove mercury in fuel gas. High temperature deposition furnace was applied to increase sulfur content in fly ash based adsorbents produced in lab. The experiments can increase its sulfur content of adsorbent, and found that sulfur attached to the surface, mesopore structure, microporous structure of fly ash-based adsorbents. So these treatments can increase their surface area and enhanced their adsorption capacity, while the sulfur was filled in interspace as small molecule S2 and S5. And the specific surface area increased by sulfur-loading and increasing temperature.

2:45 PM

Sulfur Contents on the Distribution Characteristics of Mercury in Coals: *Wei Gao*[1]; Qingcai Liu[1]; Cunfang Lu[1]; Jian Yang[1]; Huimin Zheng[1]; Juan Wen[1]; Chunling Yao[1]; [1]university of Chongqing

The coal samples were collected from coal fields in 12 provinces. There were studied on concentration, distribution, mode of occurrences and impact factors of mercury in Chinese coals. Sulfur and mercury in bituminous coals have been examined using X-ray fluorescence spectrometry, X-ray diffraction, and mercury spectrometer. Mercury levels in coal are related to the geographic location of the coals and types of the coals. A synthesis of result indicated that most likely occurrence modes of mercury in high-sulfur and high-mercury coals are in solid solution in pyrite. Coal samples showed that most values of mercury concentration in coal varied from 100 to 1000 ng/g with an average of approximately 500ng/g, and mathematical model about correlation between mercury and sulfur was gained by mathematical statistics. This is apparent that mercury and sulfur vary concomitantly from coal data,the high correlation between them at confidence of 95%(a=0.05).

3:00 PM

Characterization of Burrows from Mining District of Pachuca - Real Del Monte, in Hidalgo State and Viability Study to Use These Residues as Alternate Industrial Material: Juan Hernández[1]; *Eleazar Salinas*[1]; Francisco Patiño[1]; Isauro Rivera[1]; Martín Reyes[1]; Miguel Pérez[1]; Eduardo Cerecedo[1]; [1]Universidad Autónoma del Estado de Hidalgo

A complete characterization of burrows from mining district of Pachuca - Real del Monte was done. These kind of residues had not been seriously caharacterized, and the results found lead to an average chemical composition of 79.43 % wt SiO2, 7.032 % wt Al2O3, 2.69 % wt Fe2O3, 0.70 % wt Mn, 3.98 % wt K2O, 3.34 % wt CaO, 2.50 % wt Na2O, 1.01 % wt MgO, 0.26 % wt TiO2, 0.04 % wt Zn, 0.026 % wt Pb, 56 g/t Ag and 0.6 g/t Au; showing as principal mineralogical phases: Quartz, Berlinite, Orthoclase, Feldspar, Anorthoclase and Calcite. By the other hand, joint with the big volume of these deposits and its chemical and mineralogical characterists, this material can be used to the fabrication of ceramics, mixing it with agglutinants to produce, refractory bricks, in the industry of cement and to recover metallic values such as gold and silver.

3:15 PM

Structural Characterization of Apatite Type Rare Earth Silicates: *Lii-Cherng Leu*[1]; Sherin Thomas[2]; Mailadil Sebastian[2]; Rick Ubic[1]; Scott Misture[3]; [1]Boise State University; [2]Nationals Institute for Interdisciplinary Science and Technology; [3]Alfred University

Apatite-type rare-earth silicates have received much attention recently owing to their wide range of applications in microelectronics, solid-oxide fuel cells, solid-state lasers and phosphors. The crystal structure of Rare-earth apatite type Sr2RE8(SiO4)6O2 (RE=La, Pr, Tb, Tm and Y) ceramics which were synthesized in a phase-pure form are characterized crystallographically via both electron and X-ray diffraction. The results show evidence of hexagonal crystal structure with space group P63/m (No.176) for Sr2RE8(SiO4)6O2 (RE=La, Pr, Tb and Y). The Rietveld structure refinement on X-ray diffraction data indicated a decrease in metaprism twist angle for Sr2Tm8(SiO4)6O2 and suggested that the hexagonal metric of apatite might not be sustained and the symmetry is reduced to monoclinic space group P21/m (No.11) in order to compensate shorter bond length of the Tm ions.

3:30 PM Break

3:45 PM Keynote

Materials Characterization is the Key to Effective and Efficient Processing: Case Studies in Extractive Cu Leaching and Mineral Processing Post-Evaluation of Drywall: *Ann Hagni*[1]; [1]Geoscience Consultant

Obtaining efficient and effective processing is a main component to profitability in materials processing and production environments. Materials characterization,in turn,is the key to understanding pre-processing and post-processed materials,which enables improvements in processing and increasing profits.Two case studies will be examined showing how the same materials characterization skills and abilities can be applied to very different real world scenarios in resolving processing dilemmas.Case 1 is an extractive copper leaching project at Johnson Camp Mine in Cochise County, Arizona,in which economic copper-bearing phases and deleterious clay phases are identified,quantified,and phase relationships imaged. Case 2 is a characterization study of one of the largest building construction materials problems ever to occur in the US of problematic drywall installed primarily during 2006 and 2007. Examining American-made versus Chinese-made drywall reveals clear distinctions between the two as post-mortem(after installation) evaluations.

4:25 PM

The Influence of Ca on $(Sr_{2-x}Ca_x)(MgTe)O_6$ Double Perovskites: *Steven Letourneau*[1]; Rick Ubic[1]; Sherin Thomas[2]; G. Subodh[3]; M. Sebastian[2]; [1]Boise State University; [2]National Institute for Interdisciplinary Science and Technology; [3]Physikalishches Institut

Complex perovskite oxides $(A_{2-x}A'_x)(B_{2-y}B'_y)O_6$ are ubiquitous in electronic applications, and a complete understanding of such structures and the mechanisms which affect them is essential in predicting and controlling properties. Oxygen octahedral tilting and chemical ordering, in particular, are known to lower symmetry and influence dielectric properties. By investigating the appearance of superlattice reflections in electron and X-ray diffractograms, a summary of the octahedral tilting (tilt system) of the structure can be identified, cation ordering inferred, and space group symmetry established. In the case of the $(Sr_{2-x}Ca_x)(MgTe)O_6$ system, a phase transformation occurs from a tetragonal $I4/m$ symmetry at $x = 0$ (Sr_2MgTeO_6) to monoclinic $P21/n$ symmetry for $x = 0.5$; however, B-site cation ordering causes ½{odd,odd,odd} type reflections in all compositions, complicating crystallographic characterization. The temperature coefficient of resonant frequency is negative throughout the series; permittivities were in the range 13.2-14.3, and quality factors varied from 27,500 to 81,000 (5-6GHz).

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Technological Characterization of Phonolite Rock to Be Applied as Source of Nutrients to the Brazilian Agriculture: *Aline Maria Teixeira*[1]; João Sampaio[2]; Francisco Garrido[3]; Marta Medeiros[3]; [1]IQ/UFRJ - CETEM; [2]CETEM-MCT; [3]IQ-UFRJ

The rocks for crops is a natural practice for soils fertilization and consists on adding rocks to the soil while the water by chemical weathering decomposes the rock and release the nutrients gradually to the soil solution. This practice can be considered an alternative to the low production of potassium fertilizers in Brazil. In this context, the phonolite rock will be characterized in order to evaluate its application as an alternative source of nutrients to the Brazilian agriculture. The phonolite that will be studied is an igneous, volcanic rock as alkaline character that chemically is composed by a high grade of potassium due to the presence of feldspar minerals. In order to quantify the minerals present in the sample the characterization will be carried out using several techniques such as X-ray diffraction, X-ray fluorescence and scanning electron microscopy.

4:55 PM

Study on Thermal Stabilities of 2- Substituted Benzimidazoles Copper and Zinc Complex: Yan Shi[1]; Dan Li[1]; Qingqiao Liu[2]; *Zhongliang Xiao*[1]; [1]Changsha University of Science and Technology; [2]Hunan Hualing Xiantan Steel Co. Ltd.

2-substituted benzimidazole-zinc and 2-substituted benzimidazole-copper complex were main components of organic solderability preservativ(OSP) in PCB producing process and affect the thermal stability of OSP film.So these two complexes were prepared by 2-substituted-benzimidazole reacting with zinc ion and copper ion, the complexe crystals were determined by IR, its thermal stabilities were studied by TG method. The results indicated that the thermal stability of 2-substituted benzimidazole-zinc is higher than the 2-substituted benzimidazole-copper,and the thermal decomposition mechanism was presented.

5:10 PM

Investigation on Extracting Boric Acid from Saline Brine by Boron Specific Chelating Resin: *Xiang Xiao*[1]; Baizhen Chen[1]; Xichang Shi[1]; Yunfeng Fu[1]; Hui Xu[1]; Xiyun Yang[1]; [1]Central South University School

Extracting boric acid from saline brine by boron specific ion-exchange resin was investigated. Effects of resin amount, temperature, time and stirring speed on boron adsorption rate were studied in the experiments. The results indicated that the XSC-700 resin showed excellent boron absorbability and it performed better property for elution. The maximum boron adsorption capacity was 6.16 mg.ml-1 in the optimum conditions: the resin concentration of 5ml/100ml(brine), the temperature of 60°C, the time of 5-6h and the stirring speed of 100r/min. Boron was eluted from the resin with HCl and boric acid was crystallized by evaporating the eluate. The product of boric acid was in pure form as confirmed by XRD and other analysis techniques.

5:25 PM
Low Temperature Carbothermic Reduction of Panzhihua Low Grade Ilmenite after Ball Milling: Ying Lei[1]; Yu Li[1]; *Jinhui Peng*[1]; Libo Zhang[1]; Shenghui Guo[1]; Wei Li[1]; [1]Key Laboratory of Unconventional metallurgy, Ministry of Education

In this work, low temperature carbothermic reduction of Panzhihua low grade ilmenite after ball milling were investigated. The ball milling of ilmenite and graphite were processed in a ball mill. The milling process can attenuate the particle of ilmenite, and the ilmenite characteristic peak were broadened and weakened. The reduction temperature and behaviors are studied on the thermo gravimetric apparatus; lower temperature and higher reaction rate were obtained for the reduction of ilmenite with graphite after milled. The reduction temperature and times of ilmenite decreased to 1000~1025°C and 40~60min via milled for 4 or 8h; Low down the reduction temperature can eliminate the solution of Mg2+ and FeTiO3 in a certain degree; the reduced ilmenite has an obvious loose structure, can favored the subsequent procedure such as leaching.

Chloride 2011: Practice and Theory of Chloride-Based Metallurgy: Pyrometallurgy and Acid Regeneration

Sponsored by: The Minerals, Metals and Materials Society, Canadian Institute of Metals, TMS Extraction and Processing Division, TMS: Magnesium Committee, TMS: Energy Committee
Program Organizers: Dirk Verhulst, Consultant, Extractive Metallurgy; V.I. (Lucky) Lakshmanan, Process Research Ortech, Inc.

Monday PM
February 28, 2011
Room: 19
Location: San Diego Conv. Ctr

Session Chairs: Khamal Adham, Hatch Ltd.; Lisa Helberg, E.I. DuPont de Nemours

2:00 PM
Chloride Metallurgy: Process Technology Development: Edgar Peek[1]; [1]Xstrata Process Support

The two dominant metallurgical problems in treating most base metal (sulphide) concentrates in both the chloride and sulphate system are iron removal and sulphur elimination, respectively. In this paper the opportunities, limitations and fundamentals of the chloride metallurgy system are described against this background. Only the treatment of oxide and sulphide ores is considered. Both hydrometallurgical and pyrometallurgical unit process operations are highlighted, since most successful base metal processes are based on a combination of the two. Hence, an overview of the technical and also some economical aspects of chloride metallurgy are presented. This overview summarizes the successful applications of chloride metallurgy, but it will not give exhaustive process and plant descriptions. It predominantly focuses on the essential technical features of the metallurgical unit process operations, while providing numerous references separately.

2:25 PM
Purification of Titanium Tetrachloride: A History: *Lisa Helberg*[1]; [1]E.I. DuPont de Nemours

Titanium tetrachloride is produced through the chlorination of a variety of titanium bearing ores, some occurring in nature, and others enriched in titanium content by a variety of "beneficiation" processes. The chlorination step can produce a variety of metal chloride impurities, such as iron chlorides and vanadium chlorides, which must be removed from the $TiCl_4$ before it can be used to produce pigmentary TiO_2, titanium metal or for any other commercial use. The requisite manipulations involve both physical and chemical separations in a multi-step process. The history of various purification strategies will be discussed.

2:50 PM
Zirconium Chlorination Behavior: A Literature Review: *Donald Voit*[1]; [1]Westinghouse

The reaction rate between zircon or zirconia, carbon, and chlorine is controlled by temperature, and the degree of contact intimacy between the carbon and the zirconium. Chlorination behavior observed in static beds (and pellets) is characterized by a thin reacting front that advances through the bed or pellet. Excess and unreacted material becomes an "ash layer" that limits chlorine access to the reacting front. The reaction rate for stoichiometric, well mixed, pelletized, sub-sieve zirconia is about 100 mmoles/cm2/hr at 800°C. The reaction rate for loosely packed, ~100µ carbon and zirconia is about 1 mmole/cm2/hr. Plug flow and fluid bed reactors, using much coarser zircon or zirconia and carbon particles, have a reaction rate of about 1 µmole/cm3/hr. The reaction rate is controlled by a slow reaction between carbon and zirconium species that results in an essentially zero order reaction rate dependency for the chlorine concentration.

3:15 PM
HCl Leaching and Acid Regeneration Using MgCl2 Brines and Molten Salt Hydrates: *Jan deBakker*[1]; Joshua LaMarre[1]; Vladimiros Papangelakis[2]; Boyd Davis[1]; [1]Queen's University; [2]University of Toronto

HCl leaching using molten salt hydrates has the potential to improve the extraction of nickel from laterite ores. However, as in other HCl leaching applications, recovery of HCl is problematic. Spray pyrohydrolysis, which is used in current chloride-based flowsheets, represents a high energy cost. The authors have developed a proposed molten salt hydrate laterite leach circuit using an alternative HCl recovery route. The leach circuit envisions a leach by HCl gas in aMgCl2 MSH medium, followed by Ni/Co recovery, iron hydrolysis, precipitation of magnesium from solution as magnesium hydroxychloride, and thermal decomposition of the hydroxychloride to produce HCl gas. The flowsheet will allow a chloride leach circuit to take place with simpler equipment requirements and lower energy cost than is currently available. The laboratory work investigates key aspects of the flowsheet, including precipitation of magnesium hydroxychlorides and their composition; magnesium hydroxychloride thermal decomposition; and iron control by selective pyrohydrolysis.

3:40 PM
Precipitation of Hematite and Recovery of Hydrochloric Acid from Concentrated Iron Chloride Solutions by a Novel Hydrolytic Decomposition Process: *Levente Becze*[1]; Sebastian Hock[1]; George Demopoulos[1]; [1]McGill University

Regeneration of HCl from chloride solutions is commercially practiced by high temperature (800-900°C) pyrohydrolysis associated with high energy consumption and capital cost. As an alternative to pyrohydrolysis a novel low temperature (<200°C) non-autoclave hydrolytic process has been studied in this research work, which comprises two steps. In the first step, ferrous chloride is oxidized by oxygen at T=150 °C, followed by hydrolytic decomposition of ferric chloride to coarse hematite particles and superazeotropic hydrochloric acid (8-9M) operated at approximately 180°C. This paper provides a summary of the laboratory results related to both oxidation and hydrolysis and presents a newly designed conceptual process flowsheet.

4:05 PM Break

4:20 PM
Application of Oxygen Enrichment for the Pyrohydrolysis of Metal Chlorides: *Kamal Adham*[1]; Cassandra Lee[1]; [1]Hatch Ltd.

Metal chlorides (e.g. iron and nickel) can be pyrohydrolysed at elevated temperatures to yield a HCl rich off-gas and an oxide product. Typically, the high energy requirement for pyrohydrolysis is provided by the combustion of a hydrocarbon fuel with air. Here, it is demonstrated that the use of oxygen-enriched air can result in lower fuel consumption and better HCl concentration. In addition, the oxygen enrichment can result in smaller quantity of off-gas and less expensive process equipment. This new concept can be applied to both the spray roaster and the fluidized bed reactor. Similar

to the air-combustion case, the O2 enriched process can use a direct contact venturi scrubber, to recover energy from the roaster off gas, but at a higher efficiency. The new process also uses an absorption tower for capturing HCl from the roaster off-gas, but generates a stronger HCl solution, than is currently possible with air roasting.

4:45 PM

Chlorine, Copper and "De Novo" Synthesis of Dioxins: Alfons Buekens[1]; Shao Ke[1]; [1]Zhejiang University, Institute for Thermal Power Engineering (ITPE)

The de novo synthesis of dioxins involves a low temperature catalytic conversion of carbonaceous substrates in a flow of air. The theory as developed by Dr. Stieglitz (FZ Karlsruhe) involves copper as most potent catalyst. Tests on various sources of carbon (activated carbon, three types of soot, and graphite) conducted at 300°C allowed comparing native samples (without) with cupric chloride doped ones. Steam addition to the combustion air raises the rate of oxidation, which in turn is severely suppressed by sulphur dioxide. This paper presents thermodynamic calculations on the system Cu-Cl-O-S at the temperature of the experiments (300°C), as well as in a wider range of potential copper catalysed dioxin generation. For the sake of comparison, the results are compared with those obtained while using other transition metals as a catalyst. Some preliminary computations are presented.

5:10 PM

Segregation Roasting of a Saprolitic Laterite Ore: An Experimental Investigation: Munyaradzi Kwatara[1]; Juzer Tayabally[1]; *Edgar Peek*[1]; Ron Schonewille[2]; [1]Xstrata Process Support; [2]Xstrata Nickel

The potential recovery of nickel from a South-American saprolitic laterite ore using the segregation roasting technique was investigated. The ore was heated to temperatures that ranged between 900 and 1000°C in a rotary kiln. In all the tests, the laterite ore was first mixed with a predetermined amount of calcium chloride and either coal or coke. By varying the temperature, calcium chloride and reductant in the different tests, a preliminary set of optimal conditions was established for the specific laterite ore that was tested. A combination of chemical analysis and electron-probe-micro-analysis (EPMA) were used to determine and evaluate the effectiveness of segregation roasting on the recovery of nickel. The present paper describes the test procedure and the results that were obtained.

5:35 PM

Mechanism of Selective Chlorination of Reduced Limonitic Nickel Laterite Using a Solid Chloride Agent: *Chuanlin Fan*[1]; Xiujing Zhai[1]; Yan Fu[1]; Yongfeng Chang[1]; Binchuan Li[1]; Ting-an Zhang[1]; [1]Northeastern University

The mechanism of the selective chlorination of nickel and cobalt in reduced limonitic laterite was investigated. Reduced ores were obtained from the reduction roasting of a limonitic nickel laterite. The thermal decomposition of related chlorides and the selective chlorination of the reduced ore were carefully studied using Differential Thermal analysis – Thermogravimetry (DTA-TG), X-Ray Diffraction and chemical leaching methods. In the temperature range of 300 - 500 \176C, aluminum chloride hexahydrate was chosen as chloride agent. During the selective chlorination, reduced ore was partly chlorinated by hydrogen chloride generated from the thermal decomposition of chloride agent. Then the oxydrolysis of ferrous chloride re-generated hydrogen chloride used to further the chlorination. Finally, iron was mostly rejected as hematite, while nickel and cobalt were selectively chlorinated. More than 91%Ni,90%Co and about 4%Fe in the chlorinated ore was extracted using a water leaching method.

Computational Thermodynamics and Kinetics: Brent Fultz Honorary Session I: Joint Session with Neutron and X-Ray Studies of Advanced Materials IV Symposium

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Alloy Phases Committee, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee, ASM: Alloy Phase Diagrams Committee
Program Organizers: Raymundo Arroyave, Texas A & M University; James Morris, Oak Ridge National Laboratory; Mikko Haataja, Princeton University; Jeff Hoyt, McMaster University; Vidvuds Ozolins, University of California, Los Angeles; Xun-Li Wang, Oak Ridge National Laboratory

Monday PM — Room: 9
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Xun-Li Wang, Oak Ridge National Laboratory; Michael Manley, Lawrence Livermore National Laboratory

2:00 PM Introductory Comments

2:05 PM Invited

Megawatts and Petaflops: *Ian S. Anderson*[1]; [1]Oak Ridge National Laboratory

A new generation of MW spallation neutron sources is providing unprecedented measurements of the dynamics of systems in, and away from, equilibrium. This ever-increasing experimental sophistication in synthesizing and interrogating matter at all levels is now taking place under the guidance of elaborate predictive models whose solution and simulation has been enabled by powerful cyber-capabilities and sophisticated mathematical and computational algorithms. Combining computational and experimental methods is increasingly important in materials science and engineering to extract critical information from data (e.g., intrinsic correlations between structure, processing, and properties/behavior), as well as being increasingly necessary for breakthroughs. Although numerous examples exist of successful combinations of modeling and experiment the question arises: how to make the most impact from the new capabilities?

2:35 PM Keynote

High Temperature Thermodynamics and Atom Vibrations: *Brent Fultz*[1]; [1]California Institute of Technology

The harmonic model often gives a good starting point for the vibrational thermodynamics of materials. The vibrational entropy is big, however, and at high temperatures relatively small non-harmonic effects can alter substantially the thermodynamics of alloy phases. One effect can be understood as standard "quasiharmonic" behavior, where vibrational frequencies are lowered as a crystal expands against its bulk modulus. Most materials show a second effect from phonon-phonon interactions (PPI), and sometimes a third from how electronic excitations interact with vibrations (electron-phonon interaction, EPI). New results are presented from a study on the phonon lifetimes in aluminum, where we show with perturbation theory that the PPI from cubic anharmonicity accounts well for the lifetimes of the individual phonons. In other metals, the EPI can be important thermodynamically to temperatures approaching 1000 K. Rules of thumb for these effects are emerging from chemical and structural trends of electronic and phonon dispersions.

3:05 PM Invited

Collective Behavior of Intrinsic Localized Modes Observed in the Vibrational Spectrum Of NaI: *Michael Manley*[1]; [1]Lawrence Livermore National Laboratory

Intrinsic localized modes (ILMs) form in the lattice vibrations of both metallic uranium and ionic sodium iodide. These dynamic nonlinear hot

spots have been found to impact a variety of technologically important properties including thermal expansion, diffusion, mechanical behavior, phase transformations, and thermal conductivity. In this talk I will present recent discoveries on the ILMs in NaI that are challenging the independent local mode picture. First, the polarization of the ILMs collectively switch between <111> and <011> with very small temperature changes. Second, once an ILM polarization locks in the parent mode fragments into several distinct dispersive modes of different symmetry. This suggests that the ILMs are organizing into arrays or patterns that modulate the parent mode. Pattern formation by ILMs in anharmonic crystals was predicted more than a decade ago but never observed. Invited talk for symposium in honor of Brent Fultz

3:35 PM Break

4:00 PM Invited
Ab Initio above Zero: Alloy Thermodynamics from First Principles: *Axel van de Walle*[1]; [1]Caltech

Although ab initio methods excel at calculating physical properties for any given atomic arrangements, proper modeling of the thermodynamic properties of solid-state alloys demands a sampling of the large number of states visited in thermal equilibrium, a task that is computationally intractable via a brute-force approach. This talk describes how this impasse can be resolved through a combination of automated computational techniques that are able to efficiently model electronic excitations, lattice vibrations and configurational disorder within a unified framework: the cluster expansion formalism in conjunction with a suitable coarse-graining of the partition function.

4:30 PM Invited
Studies on Vibrational Entropy in Alloys Using Inelastic Neutron Scattering at ORNL: *Jack Robertson*[1]; [1]Oak Ridge National Laboratory

Inelastic neutron scattering spectra and neutron diffraction patterns were measured for a verity of binary alloys using the triple-axis spectrometers at the High Flux Isotope Reactor (HFIR) at Oak Ridge NAtional Laboratory. Phonon density-of-states curves were calculated from the inelastic scattering spectra, allowing estimates of the vibrational entropy in the harmonic and quasiharmonic approximations. A survey of studies done in collaboration with Professor Fultz and his students will be presented.

5:00 PM Invited
Time-of-Flight Inelastic Neutron Scattering Studies of Phonons and Magnetism in Polycrystalline Samples: *Robert McQueeney*[1]; [1]Iowa State University

Neutron time-of-flight inelastic spectrometers with large area detectors provide complex data sets containing the wavevector and energy dependences of vibrational and magnetic excitations in solids. Extracting detailed information about the excitations continues to spur the development of more and more powerful calculations of the dynamics of solids. Over the last several years, our efforts have focused on the ability to analyze and interpret data from polycrystalline samples. In this talk, I summarize how the simultaneous development of new time-of-flight spectrometers and computation has furthered our understanding of the phonons and magnetism in several materials, most notably, our studies of cerium and plutonium metals and orthoferrite-based oxides. Although these materials appear quite dissimilar, in each case electronic valence fluctuations have profound effects on the structural phase diagrams and magnetic properties.

David Pope Honorary Symposium on Fundamentals of Deformation and Fracture of Advanced Metallic Materials: Intermetallics II and Ti Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: E. P. George, Oak Ridge National Laboratory; Haruyuki Inui, Kyoto University; C. T. Liu, The Hong Kong Polytechnic University

Monday PM
February 28, 2011
Room: 32A
Location: San Diego Conv. Ctr

Session Chairs: Kevin Hemker, Johns Hopkins University; Michael Smith, Federal Bureau of Investigation

2:00 PM Invited
Transformation-Induced Plasticity during Pseudo-Elastic Deformation in Ni-Ti Microcrystals: *Michael Mills*[1]; Peter Anderson[1]; Matthew Bowers[1]; Sivom Manchiraju[1]; Peter Sarosi[2]; Michael Uchic[3]; [1]The Ohio State University; [2]General Motors R & D Tech Center; [3]Air Force Research Labs RXLM

Micromechanical testing of single crystal Ni-Ti based shape memory alloys is being performed in an effort to understand the effects of specimen size and crystal orientation on martensite-induced pseudoelastic and shape memory response. The primary goal in this investigation is to determine the means by which the matrix accommodates the large strain associated with the martensitic transformation. This information is critical in extending the working life of components under cyclic loading and/or heating. Micron-scale pillars of several crystallographic orientations have been prepared by FIB and tested in compression. Post-mortem TEM analysis has revealed the plasticity induced by the transformation. We demonstrate through this method that it is possible to directly study the mechanical response and defect generation associated with individual, isolated martensitic transformations. The experimental results are correlated with the predicted transformations and local stresses due to the crystallographic theory of martensite.

2:30 PM Invited
Some Unusual Aspects of the Deformation of FeAl and Fe2MnAl: *I. Baker*[1]; [1]Dartmouth College

An overview of three unusual aspects of the behavior of the B2 compound FeAl and the L21derivative Fe2MnAl is presented. First, both compounds show a yield anomaly. A model for the anomaly in FeAl, based on vacancy hardening (E.P. George and I. Baker, Phil. Mag. A, 77 (1998) 737), is outlined along with both data supporting and predictions of the model. The applicability of the model to describe the yield anomaly in Fe2MnAl is discussed. Second, both the room-temperature ductility and yield strength of these compounds are affected by water vapor. The phenomenology associated with this behavior is discussed along with a possible fracture mechanism. Finally, both compounds show a paramagnetic to ferromagnetic transition upon room-temperature deformation, which is reversed upon annealing. A model for this behavior (Y. Yang, I. Baker, and P. Martin, Phil. Mag. B, 79 (1999) 449), based on the formation of anti-phase boundary tubes, is presented.

3:00 PM Invited
Recent Progress in High Temperature TiAl Alloys: *Guoliang Chen*[1]; Junpin Lin[2]; [1]Univ. of Science and Technology Beijing; [2]univ. of Science and Technology Beijing

Conventional cast TiAl alloys have been used for the engine GEnx. However, the conventional TiAl alloys are not strong enough for applications at higher temperatures. TiAl alloy have been developed into two categories: conventional TiAl and high temperature TiAl (high niobium containing)

alloys. These state-of-the-art alloys have great advantage that the service temperature is 60-100 oC higher than that of conventional TiAl alloys. The presentation discuss the relationships among composition, microstructure and properties for high temperature TiAl alloys, including the phase relationship with high Nb contents, the high temperature strengthening mechanisms of Nb, Al and other elements, excellent oxidation resistance at high temperature, especially focusing the new technology for the fabrication and processing of pilot ingots.

3:30 PM

Atomistic Simulations of Cross-Slip Nucleation in L12 Ni3Al: *Satish Rao*[1]; Dennis Dimiduk[2]; Jaafar El-Awady[3]; Triplicane Parthasarathy[1]; Michael Uchic[2]; Christopher Woodward[2]; [1]UES Inc.; [2]Air Force Research laboratory; [3]Johns Hopkins University

Using atomistic (molecular statics) simulations with embedded atom potentials, we evaluate the configurations for a dislocation to form the PPV lock by intersecting a forest dislocation in L12 Ni3Al. The activation energies were estimated by determining equilibrium configurations (energies) when variable pure tensile or compressive stresses are applied along the [111] direction on the intersection configurations. We show that the activation energy at the forest dislocation intersection is significantly lower than that for cross slip in bulk. These results suggest that cross-slip should be preferentially observed at selected screw dislocation intersections in L12 Ni3Al.

3:45 PM

Effect of Lamellar Microstructure on Impact Toughness and Fracture Behavior in Wrought Gamma TiAl Alloy: *Hirotoyo Nakashima*[1]; Toshikazu Kikugawa[1]; Shun Oinuma[2]; Masao Takeyama[1]; [1]Tokyo Institute of Technology; [2]Former graduate student (Currently Toshiba Corporation Power Systems Company)

Impact properties and fracture behavior of wrought \947-TiAl based alloys with various microstructures have been evaluated by means of an instrumented Charpy impact test and a specially designed three-point bend machine installed in SEM chamber, respectively. The alloy studied is Ti-42Al-8V (at.%) with a unique transformation pathway of \946-Ti-> \945-Ti -> \946+\947. The alloy can easily be hot free-forged by 70% reduction in height by one stroke in the \946+\945 two-phase region. The load-displacement curves of the impact test clearly detect the microstructure change, and the impact toughness increases with increasing the volume fraction of the lamellar grains. Further improvement was obtained in the specimens with fine \946 particles within lamellae. *In-situ* observation and the corresponding load-displacement curves of the bend test revealed that the higher the lamellar angle with respect to the loading axis the larger the crack propagation resistance, thereby increasing the fracture toughness.

4:00 PM Break

4:10 PM

The Role of Microtexture on Fatigue Lifetime Variability and Crack Initiation Mechanisms in Ti-6Al-4V: *Christopher Szczepanski*[1]; James Larsen[2]; S. Lee Semiatin[2]; [1]UTC/AFRL; [2]AFRL/RX

To quantify the effect of microtexture on the fatigue behavior of alpha+beta titanium alloys, Ti-6Al-4V material, initially containing bands of microtexture with a strong transverse texture was hot rolled to produce materials with varying levels of microtexture. These materials were heat treated to produce a range of microstructures: an equiaxed microstructure containing microtexture, a beta annealed structure containing microtexture and an equiaxed microstructure free of microtexture. The effect of test orientation on fatigue behavior for each of these microstructural variants was investigated by replicate testing of specimens along three different orientations; RD, TD, and 45° from the RD. Initial fatigue results indicate that specimens with their loading axis along the original plate TD tend to have the longest lifetimes. Orientation imaging microscopy and fractographic analysis were used to identify the mechanisms of fatigue crack initiation in these samples and to elucidate the role of microtexture.

4:25 PM

Understanding of the Fracture Behavior of Titanium Alloy Ti-6Al-4V: *Santhosh Koduri*[1]; Vikas Dixit[1]; Peter Collins[2]; Babu Viswanathan[1]; Hamish Fraser[1]; [1]The Ohio State University; [2]University of North Texas

A rigorous approach based upon integrated characterization and neural network modeling has resulted in the differentiation of the roles that microstructure, composition, and continuum variables (e.g., tensile properties, stress state at the crack tip) have on the fracture toughness for a+ß processed Ti-6A-l4V. This approach has been applied to a high-fidelity database containing composition, microstructure and the resulting tensile and fracture toughness properties. The modeling tools have been used to isolate a key microstructural variable, which have been subsequently characterized in great depth using a suite of advanced characterization techniques. The details of one particular type of interface associated with crack initiation ahead of the moving crack tip will be discussed.

4:40 PM

Extended Zonal Dislocations Mediating {11-22}<11-2-3> Twinning in Titanium: *Bin Li*[1]; [1]Center for Advanced Vehicular Systems

We present atomistic simulation results on the mechanism for the {11-22}<11-2-3> twin growth in titanium (Ti) by analyzing the interfacial structure at the twin boundary (TB). The simulation results reveal interesting twin growth controlled by interfacial dislocations at the TB. The elementary twinning dislocations (bt) nucleate and glide in pairs but separately and sequentially on two neighboring twinning planes, distinctly different from that of conventional zonal dislocations with the core spreading over two or more twinning planes and each plane comprising one Burgers vector of an elementary twinning dislocation. These two separate elementary twinning dislocations add up to a net Burgers vector 2bt~0.2 nm along the twinning vector, with the components in the in-plane direction perpendicular to the twinning vector canceled out. A reason for such an extended zonal dislocation will be discussed.

4:55 PM

Characterization of {11-21} Twinning in \945-Ti: *Leyun Wang*[1]; Rozaliya Barabash[2]; Yiyi Yang[1]; Thomas Bieler[1]; Martin Crimp[1]; Philip Eisenlohr[3]; Wenjun Liu[4]; Gene Ice[2]; [1]Michigan State University; [2]Oak Ridge National Laboratory; [3]Max-Planck-Institut für Eisenforschung GmbH; [4]Argonne National Laboratory

Activity of two tensile twinning systems, {10-12}<-1011>(T1) and {11-21}<-1-126>(T2), is observed in textured polycrystalline \945-Ti deformed by four-point bending. This work focuses on the relatively rare and less studied T2 twins. Owing to its large shear strain, T2 twins are often accompanied by accommodating T1 twins, surface ledges and in some cases, microcracks at grain boundaries. Using high resolution electron backscattering diffraction (EBSD), significant orientation gradients were observed in some T2 twins that were only several microns thick. The subsurface geometry of one T2 twin was examined by 3D X-ray diffraction. Streaking of Laue peaks indicates the presence of geometrically necessary dislocations (GNDs) in both the twin and the parent grain. Analysis of GND presence is used to identify a possible twin nucleation and propagation mechanism. This work is supported by NSF grant DMR-0710570 and DFG grant EI681/2-1.

Dynamic Behavior of Materials V: Dynamic Behavior of Reactive Materials

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

Program Organizers: Marc Meyers, UCSD; Naresh Thadhani, Georgia Institute of Technology; George Gray, Los Alamos National Laboratory

Monday PM
February 28, 2011

Room: 5A
Location: San Diego Conv. Ctr

Session Chair: Naresh Thadhani, Georgia Institute of Technology

2:00 PM Invited

Behavior of Laminate Reactive Materials under Dynamic Loading Conditions: *Timothy Weihs*[1]; Adam Stover[1]; David Lunking[1]; Jason Jouet[2]; Naresh Thadhani[3]; S Wu[3]; Kenneth Vecchio[4]; F Jiang[4]; Richard Lee[2]; [1]Johns Hopkins University; [2]Naval Surface Warfare Center; [3]Georgia Institute of Technology; [4]UC San Diego

Reactive materials are typically designed to produce rapid bursts of heat and light upon initiation of an exothermic reaction such as the formation of an intermetallic compound. The development of layered reactive materials offers the exciting opportunity to control the ignition threshold and propagation rates for these reactions. It also provides an ideal geometry, with uniform and consistent reactant spacing, for modeling material behavior. This presentation reviews results of dynamic studies of exothermic formation reactions in dense compacts of reactive laminate particles and it focuses on 3 separate dynamic impact studies performed using the same materials. This combination of studies provides information on ignition thresholds, pressure rises, degree of reaction and oxidation, and sample fragmentation following impact. Rapid thermal studies will also be reviewed to help explain the results of the dynamic impact experiments.

2:30 PM

Dynamic Pressure Sensing during Rapid Combustion of Metal Powders in Bomb Calorimetry: *Adam Stover*[1]; Nicholas Krywopusk[1]; David Lunking[1]; Timothy Weihs[1]; [1]The Johns Hopkins University

Reactive materials show promise for increasing the energy content of future weapon systems and many of these new materials rely on rapid and complete combustion of metal particles. However, many desirable metallic fuels such as aluminum, zirconium or titanium have potential drawbacks such as a high ignition temperatures or low stabilities. Bomb calorimetry coupled with dynamic pressure sensing is utilized to compare these metallic fuels to each other in various oxygen rich atmospheres to determine which produces the largest energy release and hence the largest pressure rise upon ignition. We will present results describing the ignition thresholds for each metallic fuel, the effects of average particle size and alloying on oxidation rates, and the use of reactive laminate particles to drive oxidation of the fuel particles in loose and dense compacts. In future applications the reactive laminate particles may serve as ignition sources for these fuels under dynamic loading conditions.

2:45 PM

Fabrication and Properties of Hot Explosive Consolidation of Ni-Al Composites: *Laszlo Kecskes*[1]; Akaki Peikrishvili[2]; Elguja Chagelishvili[2]; Zhiliang Pan[3]; Weihua Lin[3]; Qiuming Wei[3]; [1]US Army Research Laboratory; [2]Tsulukidze Institute of Mining and Technology; [3]University of North Carolina Charlotte

Clad and mechanical blends of Ni-Al powders were formed into cylindrical rods using a hot explosive consolidation process (HEC). A multi-stage loading of explosive consolidation was applied to fabricate highly dense billets having correct cylindrical geometry to form mostly Ni and Al with some intermetallic phases. The consolidation temperature was varied from ambient temperature up to 900°C; the shock loading intensity was as high as 10 GPa.The combination of high temperatures and explosive compression was beneficial to the consolidation of the both type of Ni-Al compositions, resulting in near theoretical densities and high hardness values. Right circular cylinders were cut from the HEC billets and were compressed at quasi-static and dynamic strain rates. Results show that there is a significant difference in the mechanical response of the Ni-Al specimens that were made from powder blends and those made from the Ni-coated Al particles. The results will be discussed.

3:00 PM

Investigating Reaction Mechanisms in Ni+Al Configurations Using Laser-Accelerated Thin Foil Impact: *Sean Kelly*[1]; Naresh Thadhani[1]; [1]Georgia Institute of Technology

The reaction mechanisms in various Ni+Al binary metal configurations are being investigated using laser-accelerated thin foil set-up. Dynamic deformation (such as in an impact event) can allow rapid mixing of the components and lead to chemical reaction and release of energy in the form of heat, even with little or no porosity. Reaction mechanisms in these material systems must be characterized as a function of reactant configuration in order to control the initiation and the extent of reaction at various temporal and spatial scales. Experiments conducted using laser-accelerated thin foil impact of various Ni+Al configurations have been performed at a range of loading conditions to determine the process of deformation, flow, and mixing occurring up to the point of complete reaction. The results based on time-resolved interferometry measurements and microstructural characterization of recovered materials are used to isolate the reaction mechanisms for the various Ni+Al configurations.

3:15 PM

Mesoscale Modeling of the Deconsolidative Burning in Polymer-Bonded Explosives: *H.Keo Springer*[1]; [1]Lawrence Livermore National Lab

Polymer-bonded explosives (PBXs) can transition from conductive burning to more violent deconsolidative burning and subsequently detonate under heavy confinement. Previous studies have demonstrated that relatively more brittle PBXs with lower binder content have an increased propensity for deconsolidative burning. The initial stage of deconsolidative burning is characterized by the pressure-dependent infiltration of cracks and pores by product gases at the burn-front. However, the mechanism underlying the formation of this damage in (initially) intact PBX is poorly understood. The objective of this study is to use mesoscale simulations to investigate the role of energetic volume fraction and burn rate characteristics, as well as damage and fracture properties, on deconsolidative burning. The key metric of deconsolidative burning in these studies is the formation of damage at the burn-front. These modeling studies are important because they can enable the design of PBXs that do not escalate to violent burning modes.

3:30 PM Break

3:50 PM Invited

Wave Propagation and Instabilities in "Soft" Heterogeneous Materials: *Vitali Nesterenko*[1]; [1]University of California, San Diego

The wave propagation and dynamic behavior of "soft" heterogeneous materials (granular, granular composites, laminates composed from granular layers, forest of carbon nanotubes) in a broad range of impact conditions and types of deformation (shock, shear) will be discussed. The current interest on these materials is due to their efficiency as mitigators of blast/impact loading. These materials also combine high compressive strength with the ability to bulk distribute fracture resulting in a small size reactive fragments and even possible reaction on later stages of dynamic deformation. The behavior of granular metamaterials (with specially assembled mesostructure, e.g., random and periodic one or two mass low dimensional structures) under plane shock loading will be analyzed. Energy absorption and inertia based dispersive properties of these materials, crucial for intense stress wave mitigation, will be addressed. The support for this project provided by ONR MURI N00014-07-1-0740 (Program Officer Dr. Clifford Bedford).

4:20 PM
Quasi-Static and Dynamic Response of Explosively Consolidated Reactive Mixtures: *Chung-Ting Wei*[1]; Vitali Efrem[2]; Fengchun Jiang[1]; David Benson[1]; Kenneth Vecchio[1]; Naresh Thadhani[3]; Marc Meyers[1]; [1]University of California, San Diego; [2]Lawrence Livermore National Lab; [3]Georgia Institute of Technology

Five different nearly fully dense (> 95% TMD) reactive mixtures, Nb+Al, Ni+Al, Mo+Al, W+Al, Ta+Al, were produced by using double tube explosive shock consolidation. It is found that the elastic modulus of the compacts are not fully consistent with the theoretical predictions due to the the soft matrix material (aluminum), imperfections of the compacts, and the different morphologies of the inclusions. The quasi-static behaviors are interpreted using Orowan and force chain mechanism and found that the differences of mechanical properties of the inclusions are not determinants. The dynamically mechanical and fracture behaviors are investigated using the split Hopkinson pressure bar, the high-speed camera (Phantom v12), and the simulation tool "RAVEN". It is found that the geometric properties of inclusions significantly influence the dynamic properties of the compacts. By conducting experimental and computational studies, it helps us understand the utmost importance of material factors to the explosively consolidated reactive mixtures.

4:35 PM
The Role of Plastic Deformation in Initiating Intermetallic Formation Reactions: *Brady Aydelotte*[1]; Naresh Thadhani[1]; [1]Georgia Tech

Rod-on-anvil experiments are conducted on uniaxially pressed and explosively compacted nickel+aluminum and tantalum+aluminum systems. The reactivity of these systems is compared and contrasted. Mesoscale simulations are conducted on real material microstructures to explore the role of plastic deformation in reaction initiation.

4:50 PM
Numerical Modeling of Dynamic Deformation of Al/W Granular Composites: *Karl Olney*[1]; Sophia Wang[1]; David Benson[1]; Vitali Nesterenko[1]; [1]UCSD

Aluminum/Tungsten granular composites are materials which combine high density and strength with bulk distributed fracture under impact or shock loading. They are processed using cold and hot isostatic pressing of W particles/rods in the matrix of Al powder. The presentation will describe modeling of these materials on the stage of cold isostatic pressing and under quasistatic and dynamic conditions. It will be demonstrated that morphology of W component and bonding between Al particles dramatically affects their strength and mode of fracture of Al matrix similar to observed in experiments. Funding was provided from the Grant: ONR MURI N00014-07-1-0740.

5:05 PM
Investigation of Al-Metal Reactions Induced by High-Rate Mechanical Loading: *Eric Herbold*[1]; Jennifer Jordan[2]; Naresh Thadhani[1]; [1]Georgia Tech; [2]AFRL

The reaction/combustion characteristics of compressed Al-metal powder combinations are presented in a modified rod-on-anvil Taylor impact-test set-up. The reaction threshold conditions for initiation of chemical reaction of these materials are discussed in terms of a critical energy and dynamic deformation requirement. These requirements depend on the pellet density, ratio and material properties of constituents, and level of pre-conditioning of the powders by ball-milling, which are shown to affect the sensitivity of the powders.

5:20 PM
Processing and Dynamic Testing of Al/W Granular Composites: *Po-Hsun Chiu*[1]; Vitali Nesterenko[1]; [1]UCSD

High density Al-W granular composites with an identical weight ratio between Al and W and with different porosities, size, shape and orientation of W component were investigated at strain rate 1000 1/s. Samples were fabricated by Cold Isostatic Pressing and also with subsequent Hot Isostatic Pressing. Size of particles and morphology of W inclusions had a strong effect on dynamic strength. Samples with W wires arranged in axial direction (diameter 100 microns) had a highest dynamic strength and exhibited bulk distributed fragmentation of Al matrix. The support for this project provided by the Office of Naval Research Multidisciplinary University Research Initiative Award N00014-07-1-0740 (Program Officer Dr. Clifford Bedford).

5:35 PM
Meso-Scale Simulations of Epoxy-Based Reactive Materials at High Strain-Rates: *Bradley White*[1]; H. Keo Springer[2]; Jonathan Spowart[3]; Jennifer Jordan[4]; Naresh Thadhani[1]; [1]Georgia Institute of Technology; [2]Lawrence Livermore National Laboratory; [3]AFRL/RXLMD; [4]AFRL/RWME

Particle reinforced polymer composites are increasingly being studied for use as structural energetic materials. This requires a greater understanding of the mechanisms of high strain-rate particle deformation and mixing, which influences chemical reactions occurring in reactive materials. To determine the effect that each composite constituent has on the bulk and particle level mechanical behavior at high strain-rates, meso-scale simulations were conducted on epoxy reinforced with nickel and aluminum particles. The composite was subjected to compressive strain-rates of 10^3–10^4 /s. The constituent phases were then examined to determine the effects that highly contrasting material properties have on the stress transfer and deformation response of the particles. Preliminary results show that the stress-strain response of epoxy is highly influenced by the presence of particles and consequently its effect on the mechanical behavior of the particles. In this presentation results from simulations and experiments will be presented, including representative microstructures from each.

Electrode Technology for Aluminium Production: Anode Baking

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Alan Tomsett, Rio Tinto Alcan; Ketil Rye, Alcoa Mosjøen; Barry Sadler, Net Carbon Consulting Pty Ltd

Monday PM
February 28, 2011
Room: 16B
Location: San Diego Conv. Ctr

Session Chair: Said Al Maawali, Sohar Aluminium

2:00 PM Introductory Comments

2:05 PM
Determination of Coke Calcination Level and Anode Baking Level – Application and Reproducibility of L_c Based Methods: *Stein Rørvik*[1]; Lorentz Petter Lossius[2]; Arne Petter Ratvik[3]; [1]SINTEF Materials & Chemistry; [2]Hydro Primary Metal Technology; [3]Norwegian University of Science and Technology (NTNU)

The average crystallite size (L_c) is an important property of carbon materials for aluminium electrolysis; L_c is used for characterizing the petroleum coke calcination level, and sometimes also to estimate the baking level of anodes. The paper discusses problems when comparing L_c results from different laboratories with examples from round robins. The main cause are peak broadening errors introduced by the XRD instrument and sample preparation. The L_c standards ISO 20203 and ASTM 5197 neglect these errors, and two ways are demonstrated to minimize the effect and improve the standards, 1) by using thin sample thickness and 2) by embedding the coke in a high absorptive medium. Additionally ISO 17499, the equivalent baking level method for anodes is discussed and compared to L_c measurement for anode baking level. The equivalent baking level method completely bypass the peak broadening problem and is suited for baking level comparisons between anode plants.

2:30 PM

Operation of an Open Type Anode Baking Furnace with a Temporary Crossover: *Esteban Cobo*[1]; Luis Beltramino[1]; Juan Artola[1]; Jorge Rey Boero[1]; Pierre-Jean Roy[2]; Jean Bigot[2]; [1]Aluar Aluminio Argentino; [2]Rio Tinto Alcan

As part of the Puerto Madryn smelter expansion project, Aluar Aluminio Argentino has built and now operates an open type furnace designed with Rio Tinto Alcan AP Technology™.The furnace was built and started in two stages, each of them with 34 sections and 2 fires. In order to allow the construction of the second stage and the connection between both halves of the furnace, a method was used that had prior success on other projects with a temporary crossover to connect two sections at the extremity of the first stage. This paper describes the experience gained while operating in this mode and the procedural and process control modifications that were necessary to maintain quality anode baking.Footnote: AP Technology™ is a trademark of Aluminium Pechiney.

2:55 PM

Recent Developments in Anode Baking Furnace Design: Dagoberto Severo[1]; Vanderlei Gusberti[1]; Peter Sulger[2]; Felix Keller[2]; *Markus Meier*[2]; [1]CAETE Engenharia Ltda.; [2]R&D Carbon Ltd.

Today, furnace design still proceeds mainly by extrapolation from existing furnaces. Investigating existing furnaces shows the dangers of underestimating the impact of apparently small modifications: e.g., larger pits to accommodate higher anodes can result in furnaces with substandard performance. Side effects such as soot creation have then to be accepted. This paper presents an approach to bake furnace design which completely eliminates extrapolation from existing furnaces. The same approach can be used to estimate the optimization potential for existing furnaces.

3:20 PM

Sohar Aluminium's Anode Baking Furnace Operation: *Said Al Hosni*[1]; Jim Chandler[1]; Olivier Forato[1]; François Morales[2]; Christian Jonville[2]; Jean Bigot[2]; [1]Sohar Aluminium; [2]Rio Tinto Alcan

The Sohar Aluminium anode baking furnace was commissioned in 2008 and the furnace performance since has excellent in terms of gas consumption, baking level, fire productivity and firing cycle range. With 10 fluewalls per section, the furnace has productivity levels of 70 kt per fire. Gas consumption of less 1.9 GJ/t for a baking level (Lc) of greater than 33 angstrom has been maintained. Operation has been demonstrated across a fire cycle range from 24 to 36 hours. Refractory condition is excellent and first generation refractory life is likely to be > 175 fire cycles due to a thorough maintenance program and a very low anode sodium concentration of less than 150 ppm. These results have been achieved through a combination of design, process control and operation which places it amongst the benchmark operations of its type today. Some of the challenges in achieving these results are discussed.

3:45 PM Break

3:55 PM

Meeting the Challenge of Increasing Anode Baking Furnace Productivity: *François Ordronneau*[1]; Magali Gendre[1]; Luc Pomerleau[1]; Nigel Backhouse[1]; Adam Berkovich[1]; Xin Huang[1]; [1]Rio Tinto Alcan

The need to support amperage creep in smelters requires an increase in anode baking furnace productivity. The furnace operation must be adapted to provide more anodes while maintaining adequate baking performance, minimizing energy consumption and assuring anode quality. To do so, the essential factors to optimize include the firing strategy to ensure the appropriate baking level for a given fire cycle, fuel combustion efficiency and adequate cooling capability even at accelerated fire cycles and high ambient temperatures. Through the operation of 28 baking furnaces of differing technologies, tools have been developed to support this process. Simulations of firing and baking as a function of the fire cycle and key furnace design parameters such as anode size, pit width and flue design; injector combustion efficiency and cooling capability are now routinely used. Industrial examples are shown from a number of sites using varying technology that demonstrate the gains achieved.

4:20 PM

Wireless Communication for Secured Firing and Control Systems of Anode Baking Furnaces: *Nicolas Fiot*[1]; Christian Coulaud[1]; [1]SOLIOS CARBONE

Anode baking requires firing and control systems which move every day as the fire progresses around the furnace. Wired connections between the moving equipment and the central control unit have always been an operation and maintenance concern. Wireless networks have become the modern solution with standard PLCs. However, modern furnace control requires new safety loops between the firing equipment. Wireless communication has encountered numerous drawbacks when used for secured control of the furnace via a safe communication between safety PLC's due to the nature of the safety communication protocol and interference with other wifi systems in the baking furnace area. Extensive development work has been completed with major PLC suppliers to find the right combination of modems and antenna and to fine tune the PLC's and wifi systems so that operational performance and safety requirements are fully met. Wifi is now available for the secured baking of anodes.

4:45 PM

Full Control of Pitch Burn during Baking: It's Impact on Anode Quality, Operational Safety, Maintenance and Operational Costs: *Detlef Maiwald*[1]; Domenico Di Lisa[1]; Peter Mnikoleiski[1]; [1]Innovatherm

Due to the physical design of the open top ring furnace the pitch burn development is not homogeneous in the individual flues of a section. As a primary result, the consistency of the anode quality will be affected. Secondary, other disadvantages will occur as well as there are unburned pitch fractions which are condensing downstream in the furnace and filter ducts, increase of emissions, higher operational costs, negatively impact on operational safety and a higher fuel consumption.The physics of the occurrence of the pitch burn is quite complex. Once the pitch burn process is started it behaves exothermal and is hard to control. This paper will describe the different control strategies which have been developed to gain full control of the pitch burn, and will show the results as remarkable improvements on anode quality, emissions, fuel consumption and running costs.

5:10 PM

High Performance Sealing for Anode Baking Furnaces: Pierre Mahieu[1]; Sébastien Neple[1]; Nicolas Fiot[1]; Ismael Ofico[2]; Manuel Eufrasio[2]; [1]Solios Carbone; [2]Mozal

Anode baking requires important volumes of air and fumes for combustion and thermal exchange. Efficient refractory port sealing is an important condition required to reach rated flows inside flue walls. This function, apparently simple, has been facing many problems with conventional sealing techniques which can lead to process inefficiency and degraded operating conditions. A reliable and proven technology has been developed to enhance this sealing function: it is based on the use of advanced materials that combine flexibility, resistance to the extreme conditions in the furnace and offers a new potential of process improvement in anode baking furnaces. This patented system held between two rigid plates and surrounding the inner refractory port reduces drastically the incursion of cold air into the fume exhaust ramp. The most favorable outcomes of this technology are the limited condensation of exhausted fumes and increased furnace thermal efficiency.

Fatigue and Corrosion Damage in Metallic Materials: Fundamentals, Modeling and Prevention: Fatigue Property-Microstructure Relationships and Crack Growth

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division
Program Organizers: Tongguang Zhai, University of Kentucky; Zhengdong Long, Kaiser Aluminum; Peter Liaw, University of Tennessee

Monday PM
February 28, 2011

Room: 31C
Location: San Diego Conv. Ctr

Session Chairs: Tongguang Zhai, University of Kentucky; Antonios Kontsos, Drexel University

2:00 PM
Effects of Materials Strength on Very-High-Cycle Fatigue Behavior for Low Alloy Steels: *Youshi Hong*[1]; Aiguo Zhao[1]; [1]Institute of Mechanics, Chinese Academy of Sciences

The researches on the behavior of very-high-cycle fatigue (VHCF) for low alloy steels have become a new branch in the field of metal fatigue since 1980s. The behavior of VHCF for low alloy steels is evidently dominated by the strength level (yield strength and ultimate strength), which is dependent on the composition and the microstructure of the steel. The first part of this paper is a short review, summarizing a large number of experimental data from literature, and the second part is our experimental investigation on the VHCF behavior of a low alloy steel with four kinds of strength levels. All of these attempt to show the effects of materials strength on VHCF behavior for low alloy steels, including the effects on fatigue limit, on the difference of fatigue strength, on crack origination and on S-N curve characteristics.

2:20 PM
Effect of Porosity on the Mechanical and High Cycle Fatigue Behavior of Casting AM60B Magnesium Alloy: *Kee-Ahn Lee*[1]; Kyu-Sik Kim[2]; Jung-Cheol Park[3]; Chang-Dong Im[4]; [1]Andong National University; [2]Hanyang University; [3]RIST; [4]KIMM

The effect of casting defects on the high cycle fatigue behavio of magnesium alloy was investigated. Two AM60B magnesium alloys were manufactured by high pressure die casting with different processing parameters such as die morphology, melt and die temperature. One of AM60B alloys with relatively high porosity was called Alloy A(4.06%) and the other was called Alloy B(0.07%). Microstructures of both Alloy A and B were composed of primary dendrite a-Mg, Al rich a-Mg and Mg17Al12 phases. Tensile properties of both alloys were showed that Alloy B had superior tensile, yield strength and elongation. Alloy A had significantly lower fatigue life than that of Alloy B. From the observation of the differences in porosities and fractography of two AM60B magnesium alloys, an attempt to explain the high cycle fatigue fracture behavior of casted magnesium alloys was made by using stress concentration factor Kmax. [supported by "the program for Materials Bank", Korea]

2:40 PM
The Effect of Precipitate State on the Cyclic Deformation Behaviour of Al Alloys: Experiments and Modelling: Weizhong Han[1]; Alexei Vinogradov[2]; *Christopher Hutchinson*[1]; [1]Monash University; [2]Osaka City University

Fatigue deformation involves the repeated moving backwards and forwards of dislocations. A degree of irreversibility is inherent in the dislocation motion and it is generally thought that the greater the degree of reversibility, the better the fatigue properties of the material. A measure of the reversibility of deformation can be calculated from the shape change of the hysteresis loop during cyclic loading. In this work, the evolution of the hysteresis loop shape as a function of precipitate state and plastic strain amplitude has been studied at room temperature in a model Al-4Cu alloy containing shear-resistant theta prime precipitates only. An expression for the slip reversibility is derived that explicitly includes the characteristics of the precipitate distribution. Coupling the expression for the slip reversibility to an expression for the strengthening response is shown to describe well the evolution of hysteresis loop shape with both microstructural changes and plastic strain amplitude.

3:00 PM Invited
Effect of Microstructure on Fatigue Behavior of Two Pipeline Steels: *Gongyao Wang*[1]; Muralidharan Govindarajan[2]; Douglas Stalheim[3]; Peter Liaw[1]; [1]University of Tennessee; [2]Oak Ridge National Laboratory; [3]DGS Metallurgical Solutions, Inc

In recent years, significant advances have been made in the microstructural design and inclusion control of advanced pipeline steels. A range of microstructures containing various amounts of polygonal ferrite, pearlite, bainite, and acicular ferrite can be achieved in pipeline steels with comparable tensile properties. Thus, there is a need to understand the effect of the presence/absence of these microstructural constituents on the fatigue and fracture behavior of these pipeline steels. In the current investigation, fatigue crack growth behavior in air and inert atmosphere of two X70/X80 grade pipeline steels with a mixture of polygonal ferrite/acicular ferrite microstructures has been evaluated. The effect of microstructures on the fatigue properties are compared and discussed.This work was funded by the US Department of Transportation Federal Aviation, under Grant No. USDOT-DTPH56-10-T-000001, with Dr. J. Merritt as contract monitor and by the Department of Energy, Office of Hydrogen, Fuel Cells and Infrastructure Technologies

3:20 PM
Fatigue and Fracture Performance of 500MPa Grade High Strength Bridge Steel: Huaxin Hou[1]; *Chengjia Shang*[2]; Yuling Zhang[3]; [1]Ansteel Company; [2]University of Science and Technology Beijing; [3]Institute of Railway Science and Technology

High strength bridge steel has a lot of advantages, not only with high strength, good toughness and weldability, but also with anti-weather resistance property. These years, a kind of low carbon low alloy steel is developing for bridge building. With the development of rail road and highway, tow long span bridges were built over Yangtze river with 420MPa grade low carbon bainitic steels. The thickness of plate has reached 80mm. Meanwhile, much higher strength bridge steel (yield strength 500MPa) is developing. In this paper, the performance such as fatigue, fracture, wide plate tensile properties of 500MPa grade plate steel and welding conjunction were invested. Moreover, a full size girder was also tested to invest the mechanical parameters of plastic deformation during loading.

3:40 PM Break

3:50 PM
Study the High-Cycle-Fatigue Behavior of a Nano-Precipitate Strengthened Alloy by In-Situ Neutron-Diffraction Experiments: *E-Wen Huang*[1]; Michael Hofmann[2]; Saurabh Kabra[3]; Sven Vogel[4]; Peter Liaw[5]; [1]National Central University; [2]Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II); [3]Australian Nuclear Science and Technology Organisation; [4]Los Alamos National Laboratory; [5]University of Tennessee

A tension-tension high-cycle-fatigue experiment was investigated to resolve the structural evolution of a nano-precipitate-strengthened alloy. A tension loading-unloading test was studied for comparison. Applying the in-situ neutron-diffraction experiments, the ensemble-averaged-diffraction results incorporating with the macroscopic stress-strain curves yield the deformation mechanisms of the alloy. In comparison with the fracture surfaces, the different deformation mechanisms of the alloy subjected to two types of loadings have been characterized by the evolution of the lattice-strain and peak-profile evolution. A complementary texture investigation was used to gauge its validity.

4:10 PM Invited
Fatigue Crack Tip Mechanics Following a Tensile Overload: *Soo Yeol Lee*[1]; Peter K. Liaw[2]; Hahn Choo[2]; Ronald B. Rogge[3]; Ke An[4]; Camden R. Hubbard[4]; [1]The University of British Columbia; [2]The University of Tennessee; [3]National Research Council Canada; [4]Oak Ridge National Laboratory

The improvement in lifetime of numerous engineering components exposed to fatigue is highly dependent on the accurate understanding of fundamental principles of the failure mechanism. One aspect that is still not completely understood is the overload-retardation micromechanism and crack-closure behavior in structural materials subjected to fatigue. Here we report in-situ neutron diffraction and electric potential investigations that allow us to observe the crack opening/closing processes and associated internal-stress distributions around the crack tip during real-time fatigue crack propagation following a tensile overload. The results provide insights into (i) the crack-tip deformation and fracture behaviors under applied loads, (ii) the overload-induced transient crack growth micromechanism, (iii) the applicability of the effective stress intensity factor range as the fatigue crack tip driving force, and (iv) the quantitative relationship between the crack tip driving force and crack growth behavior.

4:30 PM
A Modified LEFM Approach for the Prediction of the Notch Effect in Fatigue: *Masahiro Endo*[1]; Keiji Yanase[1]; Satoshi Ikeda[2]; Arthur McEvily[3]; [1]Fukuoka University; [2]Kawasaki Heavy industries, Ltd.; [3]University of Connecticut

Engineered designs usually contain stress-raising details such as holes, fillet, grooves and keyways which are known to have a deleterious effect on the fatigue limit and fatigue lifetimes because of the growth of fatigue cracks emanating from these details. A recent paper has dealt with the behavior of small cracks emanating from one type of stress-raiser, namely holes. In that paper, a modified LEFM approach was used and it involved consideration of elastic-plastic behavior, the Kitagawa effect, and the development of crack closure in the wake of a newly formed crack. The present paper extends this approach to deal with notches of elliptical shape. The proposed approach is validated by comparing predicted and experimental results.

4:50 PM
Resistivity Based Evaluation of the Fatigue Behaviour of Cast Iron: Holger Germann[1]; Peter Starke[1]; *Dietmar Eifler*[1]; [1]University of Kaiserslautern

Cast irons are used in particular for highly stressed components in the automotive and commercial vehicle industry, e.g. for crankcases and in the wind power industry, e.g. for rotor hubs. The microstructure of cast irons and consequently their mechanical properties are strongly influenced by graphite shape, micro-shrinkage cavities, micro-pinholes, micro-cracks etc. The measurement of the electrical resistance in the virgin state and its change during fatigue loading offers the possibility to get detailed information about the actual defect density and the cyclic deformation behaviour. In the scope of the present research work stress-controlled load increase tests and constant amplitude tests were carried out at ambient temperature with specimens of the perlitic cast irons EN-GJL-250 (ASTM A48 35B), EN-GJS-600 (ASTM 80-55-06) and EN-GJV-400. Beside electrical measurements scanning electron microscopy (SEM) was used to characterize the microstructure and to correlate microstructural details with cyclic properties.

5:10 PM
Microstructure-Sensitive Probabilistic Fatigue Modeling of Notched Components: *William Musinski*[1]; David McDowell[1]; [1]Georgia Institute of Technology

Microstructure-sensitive, simulation-based strategies for modeling fatigue life reduction in cyclically loaded notched components offer a means to augment costly experiments and facilitate consideration of microstructures not yet processed. Using computational polycrystal plasticity and probabilistic arguments, a strategy is presented that links notch root microstructure heterogeneity to scatter in fatigue response and notch size effects. The notch root stress gradient field, grain size distribution, and defect distribution (pores, nonmetallic inclusions) effects are considered for a polycrystalline PM processed Ni-base superalloy IN100. The probability of forming and propagating a crack from the grain scale to a transition crack length (LEFM applicable) is approximated using shear-based fatigue indicator parameters computed within the uncracked notched specimen. This model for crack formation and early growth is calibrated to existing smooth specimen experiments. Cumulative distribution functions and probabilistic strain life functions are constructed for various notch root radii and remote strain amplitudes.

5:30 PM
Metallurgical Aspects of Stress Corrosion Cracking of X100 High-Strength Pipeline Steel: *Frank Cheng*[1]; [1]University of Calgary

It is generally acknowledged that an elevated strength level of steels tends to decrease their resistance to stress corrosion cracking (SCC), particularly, hydrogen-induced SCC (HISCC). This work summarized the author's recent research in high-strength pipeline steel SCC phenomenon. The microstructure of X100 steel was characterized by scanning electron microscopy and energy-dispersive x-ray analysis, and various metallographic phases and inclusions were identified. Furthermore, micro-electrochemical measurement techniques and photo-electrochemical technique were combined to characterize the local activity of micro-defects, such as inclusions and grain boundaries, and the galvanic effect between the defect and the steel matrix. Micro-electrochemical measurements were also conducted on the X100 steel weld, where the typical microstructures and mechanical properties were introduced during welding. It is attempted to develop an essential insight into the effect of the welding metallurgy on local dissolution rate and the SCC susceptibility of X100 line pipe steel.

Federal Funding Workshop: Panel Discussion

Sponsored by: The Minerals, Metals and Materials Society, TMS: Public and Governmental Affairs Committee
Program Organizers: Robert Shull, National Institute of Standards and Technology; Jud Ready, Georgia Tech

Monday, 4:00-6:00 PM — Room: 6E
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Robert Shull, NIST; Jud Ready, Georgia Tech

4:00 PM Introductory Comments by Robert Shull

Panel Discussion with Panelists:

Zakya Kafafi, National Science Foundation (NSF), Director, Division of Materials Research

Michael Kassner, Office of Naval Research (ONR), Director of Research

Harriet Kung, Department of Energy (DOE), Director, Basic Energy Sciences

Jon Mogford, Defense Advanced Research Projects Agency(DARPA), Deputy Director, Defense Sciences Office

Thomas Russell, U.S. Air Force Office of Scientific Research (AFOSR), Director, Aerospace Chemical and Materials Division

David Stepp, U.S. Army Research Office (ARO), Chief, Materials Science Division

Summary Comments by Jud Ready

Networking Reception

Friction Stir Welding and Processing VI: High Temperature Materials 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; Murray Mahoney, Retired from Rockwell Scientific; Yutaka Sato, Tohoku University; Yuri Hovanski, Pacific Northwest National Laboratory; Ravi Verma, General Motors

Monday PM
February 28, 2011

Room: 5B
Location: San Diego Conv. Ctr

Session Chair: Yuri Hovanski, Pacific Northwest National Laboratory

2:00 PM Invited

Reliable Sealing of Copper Canisters through Cascaded Control of Power Input and Tool Temperature: *Lars Cederqvist*[1]; Olof Garpinger[2]; Tore Hagglund[3]; Anders Robertsson[3]; [1]SKB/Lund University; [2]XDIN; [3]Lund University

The Swedish Nuclear Fuel and Waste Management Company (SKB) will join at least 12,000 lids and bottoms to the copper tubes containing Sweden's nuclear waste using friction stir welding (FSW). The paper shows how the 45 minute long weld cycle, with variable thermal boundary conditions, can be reliably controlled by an inner regulator loop controlling the power input and a slower outer regulator loop controlling the tool temperature. The results from recent, automatically controlled, welds are compared to those of early, manually controlled, welds. It is also shown how relatively small changes in the control strategy, depending on sequence in the weld cycle, can improve the overall performance and repeatability of the process. In addition to this, the controller is also compared to manual control when it comes to suppressing torque disturbances. The authors most definitely believe that the chosen, cascaded, controller structure can be successfully used in other applications in which the temperature signal reacts relatively slow to fast and distinct torque disturbances.

2:25 PM Invited

Friction Stir Welding (FSW) of a Hardenable Alloy Steel in 'Dry' and 'Wet' Environments: Norman Overfield[1]; Murray Mahoney[2]; Russell Steel[3]; Jon Babb[3]; Sarath Menon[1]; *Terry McNelley*[1]; [1]Naval Postgraduate School; [2]Consultant, Brigham Young University; [3]MegaStir Technologies

Recent advances in FSW tool material and design will facilitate numerous applications of this solid-state joining technology to steels. A single W-Re-CBN tool was used to conduct a series of bead-on-plate FSW traverses, approximately 1.5m in total length, on 6.3mm thick plates of a hardenable alloy steel. The first series of traverses involved various RPM/IPM combinations and a dry plate. A second series was carried out while a plate was immersed in water in order to assess potential for inducing hydrogen assisted cracking (HAC) during FSW of susceptible alloys. All traverses were defect-free. The FSW nuggets exhibited refined microstructures and increased hardness relative to the base plate. The influence of FSW parameters on nugget microstructure and mechanical properties will be summarized.

2:50 PM Invited

Friction Stir Welding of Industrial Steels: Jonathan Perrett[1]; *Jonathan Martin*[1]; Jeremy Peterson[2]; Russell Steel[2]; Scott Packer[3]; [1]TWI; [2]MegaStir Technologies; [3]Advanced Metal Products

Friction Stir Welding of steel has been in development for over a decade, but only in recent years have the strength and wear characteristics of tool materials improved extensively. Tool materials have been developed that have excellent high-temperature wear resistance, and consistently achieve weld lengths in excess of 50 meters in steel. These newly developed material characteristics comprise of a fine balance of high-temperature strength, hardness and ductility. Shipbuilding, bridge decking, pipe seam welding, or applications which require long uninterrupted steel welds, are now attractive target markets for the friction stir process. It is the process's ability to create high-strength, low-distortion welds that make it potentially attractive to industry. This paper shows the results from welding and testing of five common industrial steels, as well tool wear profiles and demonstrative 6m length welds.

3:15 PM Invited

Localized Mechanical Behavior Of Ti-5111 Friction Stir Welds: *Jennifer Wolk*[1]; Marc Zupan[2]; Christopher Cheng[2]; [1]Naval Surface Warfare Center; [2]University of Maryland Baltimore County

Titanium (Ti) and titanium alloys have shown excellent mechanical, physical, and corrosion properties for marine applications. Friction stir welding (FSW) is being investigated as a possible joining alternative due to difficulties in conventional welding of Ti alloys. Due to the natural asymmetry of the FSW process, bulk mechanical properties may not be representative of the localized properties. This work focuses on direct measurement of the base metal, heat affected zone (HAZ), thermomechanically affected zone (TMAZ), and stir zone mechanical properties in friction stir welded Ti-5111. The localized mechanical properties are measured using micro scale tensile tests (~3 mm long by 1 mm wide with a cross-sectional gage section ~250 µm square) extracted directly from the transverse cross-section of the weld. After testing, the mechanical response is correlated to the underlying material microstructure and texture for the different regions of the Ti-5111 friction stir weld.

3:40 PM

Mechanical Properties of Thick Section Titanium 6Al-4V Friction Stir and Electron Beam Welds: *Paul Edwards*[1]; Gary Coleman[1]; Marc Petersen[1]; [1]The Boeing Company

In this study, Friction Stir Welding of Ti-6Al-4V was developed in 1.0 inch thickness material. The microstructure and mechanical properties such as static, fatigue, fracture toughness and crack growth of these thick section Friction Stir Welds were evaluated and compared to Electron Beam Welds produced in the same thickness material. It was found that the Friction Stir Welds did possess a more wrought type structure than the Electron Beam Welds, leading to better ductility, fatigue life and fracture toughness. The solidified structure of the Electron Beam Welds resulted in higher strengths and showed slower crack growth rates. This screening data will allow design engineers to down select these processes, considering both cost and performance, for advanced aircraft structures.

4:00 PM

Transformation and Deformation Texture Study in Friction Stir Processed API X80 Steel: *Majid Abbasi*[1]; Tracy Nelson[1]; Carl Sorensen[1]; [1]Brigham Young University

The nature of deformation in FSW is very complex. This complexity becomes even more challenging when allotropic phase transformations are present. In this research, Electron Backscattered Diffraction is used as a means to reconstruct the prior austenite texture and grain structure. Considering the fact that the room temperature microstructure contains very little or no residual PA, the prior austenite microstructure was reconstructed from the room temperature ferrite utilizing transformation orientation relationships. Analysis shows that there is little evidence of shear deformation texture in the reconstructed PA, however; room temperature ferrite exhibits strong shear deformation texture components. This implies that extended plasticity mechanisms are occurring in prior austenite which weaken the shear deformation texture. The observed shear deformation texture in the room temperature microstructure implies that FSW imposes deformation during and after the phase transformation. The evolution of both elevated and room temperature textures in FSW HSLA steel will be presented.

4:20 PM Break

4:30 PM Invited
The Mechanisms of Microstructure Control and Homogenization in Multi-Pass Friction Stir Processing (FSP) of NiAl Bronze: *Terry McNelley*[1]; Sarath Menon[1]; Carolyn England[1]; [1]Naval Postgraduate School

Multi-pass FSP of cast NiAl bronze provides a refined, homogeneous and isotropic surface layer in which both strength and ductility are doubled, relative to the as-cast condition. For large marine components this technology improves performance and reduces costs. The as-cast microstructure comprises primary and eutectoid constituents. The stir zone (SZ) microstructures reflect rapid transients and steep gradients in strain, strain rate and temperature. Field-emission scanning electron microscopy (FE-SEM) methods, including orientation imaging microscopy (OIM) have been applied to the evolution of thermomechanically affected zone (TMAZ) and SZ microstructures during single- and multi-pass FSP. A duplex a(fcc)/ß(bcc) microstructure develops in the TMAZ ahead of the tool and compatible deformation of these two phases enables estimation of strains and temperatures in this region. The development of the refined SZ microstructure in association with fragmentation of the duplex structure will be described.

4:55 PM
Wear Testing of Friction Stir Spot Welding Tools and Evaluation of Weld Performance as a Function of Tool Condition: Chris Ridges[1]; *Michael Miles*[1]; Yuri Hovanski[2]; Russell Steel[3]; [1]BYU; [2]Pacific Northwest National Lab; [3]Megastir Technologies

Friction stir spot welding has been shown to be a viable method of joining ultra high strength steel, both in terms of joint strength and process cycle time. However, the cost of tooling must be reasonable in order for this method to be adopted as an industrial process. Several tooling materials have been evaluated in prior studies, including silicon nitride and polycrystalline cubic boron nitride (PCBN). Recently a new tool alloy has been evaluated, where a blend of PCBN and tungsten rhenium (W-Re) was used in order to improve toughness while maintaining an acceptable level of hardness and wear resistance. Wear testing and weld strength results will be presented for a PCBN tool as well as for tools that contain a blend of PCBN and W-Re, in the proportions of 60% PCBN, 70% PCBN, and 80% PCBN, where the balance in each case is W-Re.

5:15 PM
Tungsten Based Tool Material Development for the Friction Stir Welding of Hard Metals: *Brian Thompson*[1]; [1]EWI

The Friction Stir Welding (FSW) of hard metals such as steel and titanium requires unique tool materials capable of achieving long linear lengths while minimizing tool degradation. W-based materials have demonstrated success as a FSW tool in resisting degradation while maximizing tool life. This presentation will review the development of current and future W-based tool materials for the FSW of hard metals. Results from the characterization of tool material microstructures identifying tool degradation mechanisms will be presented along with the evaluation of cutting edge tool materials for their performance in X-70 High Strength Steel and Titanium 6-4. In addition, resulting weld mechanical properties from these welds with corresponding stir zone microstructural characterization will be presented. This on-going work has resulted in a greater understanding of W-based tool materials and the development of improved W-based tool material compositions capable of achieving tool life in the hundreds of feet.

5:35 PM
Improving Heat-Affected Zone Liquation Cracking Resistance of Nickel-Base Alloys by Friction Stir Processing: *James Rule*[1]; Jeffrey Rodelas[1]; John Lippold[1]; [1]Welding and Joining Metallurgy Group, The Ohio State University

Friction stir processing (FSP) was evaluated as a means to reduce the susceptibility of two solid-solution strengthened Ni-Base alloys, Alloy 625 and Hastelloy X, and a Ni-Base superalloy, Inconel 718, to heat-affected zone (HAZ) liquation cracking. FSP of the base metal resulted in severe reduction in grain size (average grain diameter of less than 10μm). These grains contained highly tortuous grain boundaries with a very fine distribution of second phases. Cracking resistance was evaluated using the spot-varestraint test. Testing showed a reduction in cracking susceptibility relative to the baseline due to the reduction in HAZ grain size. Optical microscopy evaluation revealed that HAZ liquation cracking resistance is enhanced due to a higher density of grain boundaries resulting in finer, more discontinuous networks of low melting eutectic and second phases.

5:55 PM
Using Electron Backscatter Diffraction to Characterize the Texture and Microstructure of Friction Stir Welded AISI 304L Stainless Steel: *Benjamin Nelson*[1]; Carl Sorensen[1]; [1]Brigham Young University

The crystallographic texture of friction stir welded (FSW) metals has been shown to resemble the torsional simple shear deformation (TSSD) texture of metals. In this study, electron backscatter diffraction (EBSD) was used to analyze the texture and microstructure of FSW AISI 304L stainless steel (304L). Locations at the retreating side, weld center, advancing side, and TMAZ were scanned and analyzed. For texture analysis, EBSD data was rotated to match the sample coordinate system of TSSD. Quantitative analysis of the texture showed that over 90% of the non-random textures correspond to TSSD; the remainder corresponds to a discontinuous dynamic recrystallization texture. EBSD microstructure analysis shows that dislocation cells are formed in the final microstructure of FSW 304L. Texture and microstructure analysis results suggest that dynamic recovery and continuous dynamic recrystallization (recovery phenomena) are active in the final microstructure of FSW 304L, while discontinuous dynamic recrystallization (nucleation phenomenon) is not.

Frontiers in Solidification Science: Nucleation and Related Phenomena

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS: Solidification Committee
Program Organizers: Jeffrey Hoyt, McMaster University; Daniel Lewis, Rensselaer Polytechnic Institute

Monday PM — Room: 6E
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chair: To Be Announced

2:00 PM Invited
Heterogeneous Nucleation of Anisotropic Solids: Aurèle Mariaux[1]; *Michel Rappaz*[1]; [1]EPFL

Surprisingly, the well-known theory of heterogeneous nucleation of a solid from a liquid phase on a foreign substrate did not consider anisotropic situations. Taking the case of zinc grains forming in thin coatings deposited by hot-dipping on steel sheets as a motivation to this study, the xsi-vector formalism of Cahn and Hoffman was first used to handle anisotropic nucleation situations. It can be shown that the ratio of the heterogeneous and homogeneous nucleation energy barriers is still given by the ratio of the corresponding volumes of the heterogeneous and homogeneous anisotropic nuclei. However, the function describing this ratio, which only depends on the ratio (gamma_lf - gamma_sf)/gamma_sl0 for the isotropic case, where gamma_lf and gamma_sf are the liquid-foreign substrate and solid-foreign substrate interfacial energies, respectively, becomes a function of the orientation of the nucleus with respect to the substrate. In the case of zinc, it is shown that good wetting conditions, i.e., (gamma_lf - gamma_sf) > 0, favor a basal orientation of the nucleus on the foreign substrate. Experimental evidence from several zinc-based systems will be presented to support this finding.

2:30 PM Invited
Phase-Field Simulations of Peritectic Growth in Ti-Al-B Including TiB_2 Inoculation: *Markus Apel*[1]; Janin Eiken[1]; Ulrike Hecht[1]; [1]Access e. V.

By varying the B content in a peritectic Ti45at%AlxB alloy the formation temperature of boride particles can be adjusted. For x>1.47at% TiB_2 is the first phase which forms during solidification. For 0.58at%< x <1.47at% TiB_2 becomes stable after β-Ti but before α-Ti. In the first case boride particles can inoculate β-Ti, whereas in the latter case α-Ti. We elucidate the effect of borides on microstructure formation by phase-field simulations coupled to a thermodynamic database. Nucleation and growth of small boride particles are explicitly taken into account on a scale below the phase-field interface thickness by using semi-analytical growth models.The simulation results show a variety of effects: grain refinement due to heterogeneous nucleation on TiB_2 particles either of the β or α phase, interactions between the growing Ti phases and the borides, as well as a pronounced dependency of the phase transformation rates on the dominant nucleation sites.

3:00 PM
Dispersion of Nanoparticles in Magnesium Using Contactless Electromagnetic Acoustic Transmission (EMAT) under High Magnetic Fields: *Zachary Bryan*[1]; Peiru Chen[1]; Hunter Henderson[1]; Orlando Rios[2]; Gail Mackiewicz-Ludtka[2]; Gerard Ludtka[2]; Michele Manuel[1]; [1]University of Florida; [2]Oak Ridge National Laboratory

Magnesium and its alloys have recently drawn significant attention due to their high specific strength. However, magnesium's inherently low ductility has prevented its wide spread integration into major engineering applications. It has been shown that the addition of ceramic nanoparticles to magnesium alloys can lead to dramatic increases in hardness, strength, and possibly ductility. This collaborative study focuses on the application of a novel contactless electromagnetic acoustical transmission (EMAT) technique to disperse dysprosium oxide nano-spheres and nano-rods in molten magnesium and throughout solidification. The magnesium and nano-particle composites, prepared at the University of Florida's Materials Design & Prototyping Laboratory, were induction melted using a custom apparatus developed at Oak Ridge National Laboratory (ORNL) in a 20 Tesla high magnetic field under controlled atmosphere at the National High Magnetic Field Laboratory (NHMFL). A detailed microstructural analysis characterizing the level and degree of nano-particle dispersion is presented and inferences will be provided regarding the nature of the solidification process. The authors acknowledge financial support from the US National Science Foundation award number DMR-0845868, Department of Energy DOE-EERE-ITP and Office of Science Graduate Fellowship Program administered by the Oak Ridge Institute for Science and Education, and General Motors Corporation.

3:20 PM
In-Situ Investigation of the Growth Dynamics of Floating and Locked Eutectic Grains during Thin-Sample Directional Solidification Using a Rotating Stage: *Melis Serefoglu*[1]; Sabine Bottin-Rousseau[1]; Silvère Akamatsu[1]; Gabriel Faivre[1]; [1]UPMC / CNRS

We investigated the directional-solidification dynamics of "floating" and "locked" eutectic grains (EGs) in a 12-µm thick metallic samples (In-In2Bi) using a rotating stage. Our setup permits us to change the in-plane orientation of the crystals with respect to the thermal gradient continuously, while keeping the relative orientation of the two phases constant, and observing the solidification front in real time by videomicroscopy. We show that floating EGs correspond to the so-called regular eutectics generally observed in nearly isotropic systems with nonfaceted solid-liquid interfaces, i.e. the lamellae grow locally perpendicular to the front over the whole angular range. In contrast, in locked EGs, the lamellae keep, on average, a fixed orientation in the sample (i.e. crystal) reference frame during rotation. We usually observed a few locking orientations in a given EG. Assuming that they correspond to singular orientations, one can obtain valuable information on the gamma-plot of the interphase boundaries.

Hume-Rothery Symposium Thermodynamics and Diffusion Coupling in Alloys - Application Driven Science: Modeling of Atomic Mobility

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Zi-Kui Liu, The Pennsylvania State University; Larry Kaufman, CALPHAD, Inc.; Annika Borgenstam, Royal Institute of Technology; Carelyn Campbell, NIST

Monday PM — Room: 31A
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: David Andersson, Los Alamos National Laboratory; Annika Borgenstam, Royal Institute of Technology

2:00 PM Invited
Assessment of Mobilities: *Lars Hoglund*[1]; [1]KTH

Simulations of diffusion controlled transformations have over the recent years become increasingly important for understanding the behaviour materials both in manufacturing and use. This applies both to new advanced material as well as materials that been long available. These simulations take advantage of the large availability of existing thermodynamic databases and software that with great accuracy can predict phase equilibria in a number of alloying systems. In order to perform accurate simulations of diffusion controlled processes, accurate descriptions of diffusion coefficients in the system must assessed and stored in databases.The most convenient way in which to store this type of information is to assess and store the mobilities of the different diffusing components in databases. An overview on the methods used and the type of data that can be used to perform this task will be presented

2:30 PM Invited
Evaluation of the Uncertainty of Extrapolating Thermodynamic and Mobility Data in Temperature and Composition: *Annika Borgenstam*[1]; [1]KTH

It has for a very long time been debated if the bainitic transformation is formed by a diffusionless process or if it is controlled by carbon diffusion. If the transformation is controlled by carbon diffusion it should be possible to calculate the growth rate as controlled by carbon diffusion under paraequilibrium conditions. This has been done previously but must be done again for certain cases. When calculating the growth rate it is very important to have a proper thermodynamic description as well as correct mobility data. Since the bainitic transformation in steel takes place at rather low temperatures it is important that the extrapolations of both the thermodynamic and mobility data are correct. In the present work an attempt to evaluate the uncertainty in these extrapolations of temperature and composition are presented. It will be discussed from a general perspective and exemplified using new thermodynamic descriptions of Fe-C and Fe-Cr.

3:00 PM Invited
Development of a Reference Self-Diffusion Mobility Database: *Carelyn Campbell*[1]; [1]National Institute of Standards and Technology

Twenty years ago, John Ågren initiated the development of a CALPHAD-based approach to diffusion mobility databases. Since then, several multicomponent diffusion mobility databases have been developed for steels and Ni-base superalloys; however, the development of multicomponent diffusion mobility databases for other alloy systems has been slowed by the lack of standard references for the self-diffusion of the pure elements. In 2009, an international group began meeting at NIST to develop a set of standard reference diffusion mobilities. An overview of this group's work, including a set of initial recommendations for the self-diffusion in Al, Cu, Ni and Fe, will be presented. The recommendations are based on evaluation of the experimental data using a statistical meta-analysis, first principle calculations, and the use of previous assessments.

3:30 PM Break

3:50 PM Invited

Atomistic and Meso-Scale Simulations of Diffusion in $UO_{2\pm x}$: *David Andersson*[1]; Pankaj Nerikar[1]; Blas Uberuaga[1]; Michael Buksas[1]; Neil Carlson[1]; Cetin Unal[1]; Chris Stanek[1]; [1]Los Alamos National Laboratory

Species transport is a critical process in nuclear fuels and due to fission gas redistribution and evolution of the $UO_{2\pm x}$ stoichiometry it is an important performance characteristic. Xe transport is studied by calculating the activation energy using density functional theory (DFT) techniques. By analyzing the Xe solution thermodynamics, the relevant migration barriers and the interaction of dissolved Xe atoms with U vacancies we demonstrate that Xe diffusion occurs via a vacancy-mediated ring-like mechanism. The interaction of Xe with grain boundaries and dislocations has previously been investigated via atomistic simulations and we apply these results to formulate a thermodynamic and kinetic model for Xe segregation that account for the unique boundary characteristics. The resulting transport model for bulk and boundaries is then solved for both idealized and realistic microstructures within a finite element code. Finally, using DFT and kinetic Monte Carlo simulations we study the diffusion of O in non-stoichiometric UO_{2+x}.

4:20 PM

Ab Initio Determination of Point Defect Kinetics in Fe-Based Alloys: Tilmann Hickel[1]; Roman Nazarov[1]; *Niko Sandschneider*[1]; Joerg Neugebauer[1]; [1]Max-Planck-Institut fuer Eisenforschung GmbH

Aiming at a weight reduction of Fe-based products, there is currently a great interest in various alloying concepts such as Fe-Mn or Fe-Al. However, in order to reduce failure mechanisms in these materials, the understanding of their defect kinetics needs to be significantly improved. We therefore extensively studied the diffusion of hydrogen, a prominent interstitial atom connected to brittle fracture. In addition we considered vacancy driven self- and impurity diffusion, which is often responsible for strength reduction and creep. Density functional theory has been used to determine the energetics and structures of the involved defects (including the complex interplay with the local magnetic environment) and to obtain the complete information on the relevant transition states. These data are combined with kinetic Monte-Carlo simulations, in order to achieve temperature dependent diffusion constants. We observe characteristic dependencies on the concentration of alloying elements, their spatial distribution and the resulting local strain.

4:50 PM

Oxygen Diffusion in α-Titanium Alloys: *Henry Wu*[1]; Dallas Trinkle[1]; [1]University of Illinois at Urbana-Champaign

Titanium alloys are limited in high temperature applications by oxidation. To understand and inhibit oxygen penetration, we determine the mechanisms of oxygen diffusion through α-titanium with *ab initio* calculations. Diffusion in both the c-axis and basal directions proceeds between octahedral interstitial sites, through metastable hexahedral and crowdion configurations. From the mechanisms and energy barriers, we calculate the diffusion coefficient and anisotropy in the c-axis and basal directions that match well to experimental measurements. We use DFT to compute the interaction energy between oxygen in α-titanium and substitutional solutes such as Al, V, and Sc. Solutes reduce oxygen diffusion: attractive solutes act as traps, and repulsive solutes induce the labyrinth effect for non-dilute concentrations. We use the diffusion model to compare the effect of solutes on oxygen diffusion through pure Ti and Ti-6Al-4V.

5:20 PM

Phase Stability of Mg2X Intermetallic Phases in the Mg-X-X' (X,X': Si, Ge, Sn) Systems: *Jean Claude Tedenac*[1]; Isabelle Martin[1]; Elodie Ruiz[1]; Abel Haidoux[1]; [1]ICG

The materials based on the intermetallic compounds Mg2X (X = Si, Ge, Sn) are widely studied for applications in thermoelectric energy conversion. The ternary systems from which they derive are not very well known. It appears that the stabilities of the solid solutions are not as obvious as it sounds. We present in this communication an experimental study on the sections Mg2Si-Mg2Ge and Mg2Si-Mg2Sn of the ternary systems. The stability of alloys of composition between 10 and 30 at% are studied by the way of the solid state methods. A thermodynamic study is given and the kinetic of decomposition is described. This study concerns an experimental approach which willl be included a Calphad procedure. First results on the modelling of the system Mg-Ge-Si are given.

Hydrogen Storage in Materials: Theory and Experiment: Session II

Sponsored by: The Minerals, Metals and Materials Society, ASM Materials Science Critical Technology Sector, TMS: Energy Conversion and Storage Committee
Program Organizer: Louis Hector Jr, GM R&D Center

Monday PM
February 28, 2011

Room: 13
Location: San Diego Conv. Ctr

Session Chair: Dallas Trinkle, University of Illinois, Urbana-Champaign

2:00 PM

Controlling the Kinetics and Decomposition Pathway of LiBH4 and Mg(BH4)2 via Confinement in Highly Ordered Nanoporous Carbon: *Xiangfeng Liu*[1]; David Peaslee[1]; Christopher Jost[1]; Eric Majzoub[1]; [1]University of Missouri-St. Louis

In addition to conventional strategies for improving kinetics and thermodynamics of metal hydrides, including the use of catalyst dopants or destabilization, the effect of nanoscale size, through confinement in templates or frameworks, has attracted increasing attention. The decomposition thermodynamics and kinetics of LiBH4 and Mg(BH4)2 has been investigated in the presence of highly ordered nanoporous carbon (NPC) with a 2D-hexagonal geometry, a narrow size distribution, and different pore sizes. LiBH4 confined within 2 nm highly-ordered columnar pores becomes amorphous. The nano-confinement results in the disappearance of the structural phase transition and the melting transition, a significant decrease of the onset desorption temperature, and suppresses diborane release. The reaction pathway is likely altered through a strong interaction with the carbon template. We illustrate the wetting behavior of LiBH4 on infiltration NPC using differential scanning calorimetry, and discuss the implications of the wetting behavior on the decomposition pathways.

2:20 PM

Direct Line-of-Site Gas Desorption Study of $LiBH_4$ in Nanoporous Carbons: The Influence of Surface Chemistry and the Size Effect: *David Peaslee*[1]; Xiangfeng Liu[1]; E. Majzoub[1]; T. Baumann[2]; [1]University of Missouri - St. Louis; [2]Lawrence Livermore National Laboratory

Thermodynamic and kinetic properties of metal hydrides may differ between bulk and nano-sized particles. Studies of nano-structured carbon materials infiltrated with $LiBH_4$ reveal differences in the decomposition pathway that depend on the nano-pore size and the surface chemistry. We present results of gas desorption using a direct line-of-site residual gas analyzer mass-spectrometer (RGAMS) to characterize gas desorption species, and decomposition temperatures, and differences in reaction pathways for a variety of $LiBH_4$, $Ca(BH_4)_2$, and $Mg(BH_4)_2$ infiltrated carbons. We present results for 9-14 nm carbon aerogels and 2-4 nm highly ordered nano-porous carbons consisting of columnar pores packed in a hexagonal geometry. RGAMS results, together with XRD, FTIR, and calorimetry studies, indicate that pore size, non-crystallinity, and surface chemistry all play a role in formation of diborane (B_2H_6) during the infiltration process and also during desorption of melt-infiltrated materials.

2:40 PM

Energetics and Microstructures in Mg/Nb Multilayers: *Anchalee Junkaew*[1]; Byoungsoo Ham[1]; Xinghang Zhang[1]; Raymundo Arroyave[1]; [1]Texas A&M University

Nb has been proposed for improving the hydrogen sorption kinetics in Mg thin films. Microscopy studies show the formation of metastable phases

at interfaces. In this presentation, we examine the microstructure of Mg/Nb multilayers through first-principles calculations based on Density Functional Theory. The interfacial energies of different interface configurations are reported and the effect of chemistry and strain energy on the interfacial energies is quantified. The resulting interfacial structures are then compared to experimental investigations. The structures of bulk distorted bcc/hcp lattices will also be compared to the structures resulting from interfacial relaxations, and the underlying physical basis for the formation of these metastable interfacial phases will be discussed. Finally, the electronic structure of these interfacial structures will be compared to that of hydrogen-carrying bulk phases in order to determine likely sites for hydrogen occupation in Mg/Nb multilayer films.

3:00 PM

Exploration of Mg and Ca Based Laves Phases for Hydrogen Storage: *Beau Billet*[1]; Yuwen Cui[1]; J.-C. Zhao[1]; Leonid Bendersky[2]; William Boettinger[2]; [1]Ohio State University; [2]National Institute of Standards and Technology

Laves phases are well-known compounds for forming interstitial hydrides. Lightweight Laves phase based on Mg and Ca are attractive since lighter elements may improve the gravimetric density of the hydrides, which is key for hydrogen storage. For transition metal based Laves phases, empirical rules have been developed to correlate the hydriding and dehydriding properties to the interstitial site sizes. We are studying the mutual solubility and the dependence of lattice parameters with compositions using a high-efficiency diffusion-multiple approach. Two diffusion multiples made up of CaMg2, CaAl2, CaZn2, MgNi2 and MgCu2 are studied to determine the stability and composition space for the C14, C15 and C36 Laves phases of the pseudo binary and pseudo ternary systems. Results on the phase equilibria, interstitial sizes and hydrogen storage properties of selected compositions will be reported.

3:20 PM

Hydrogen Absorption/Desorption Behavior of the MgH_2-Ni/Al_2O_3 Composite Prepared by High Energy Ball Milling: *Natsuki Yamasaki*[1]; Manshi Ohyanagi[1]; [1]Ryukoku University

The effect of Ni/Al_2O_3 composite additive on the hydrogen absorption/desorption kinetics of the magnesium hydride (MgH_2) was studied. The Ni/Al_2O_3 composite was prepared by the oxygen reduction of the NiO/Al_2O_3 composite using hydrogen. The MgH_2 and 50wt% Ni/Al_2O_3 were mechanically milled using a gear driven planetary ball mill for 10 min. In the case of the specimen heated with 1 °C/min under helium flow, the onset temperature of hydrogen desorption of the MgH_2-Ni/Al_2O_3 was 180 °C, which is 40 °C lower than that of MgH_2-Al_2O_3. From the results of the hydrogen absorption measurement at 150°C for 10 s, the Mg-Ni/Al_2O_3 absorbed approximately 60% of the relative hydrogen desorption capacity, when the Mg-Al_2O_3 absorbed only 10%.

3:40 PM Break

4:00 PM

Improved Thermodynamics and Kinetics of Mg-based Hydrogen-Storage Materials Produced via Controlled Devitrification of a Metallic Glass: *Eric Lass*[1]; [1]NIST

Mg-based hydrogen-storage materials are attractive because of their high gravimetric capacity and low cost. However, they exhibit poor kinetics and high absorption/desorption temperatures due to slow diffusion and a high formation enthalpy of the hydride. Ultra-fine grained materials show improved hydriding characteristics because of an increase in the density of short-circuit diffusion paths (i.e. grain/particle surfaces); and can destabilize the hydride phase when particle size is sufficiently reduced. In this work, nanocrystalline Mg-Ni based alloys for hydrogen-storage are produced via controlled devitrification of a metallic glass. The kinetics of hydriding/dehydriding are much improved compared to other bulk Mg-based materials. By adding small amounts of a ternary element, phase equilibria can be modified so that 4.5 to 5 wt. % H can be fully absorbed and desorbed at 473 K, about 100 K lower than other bulk Mg-based alloys.

4:20 PM

Li-Mg Solid Solution Alloy for Reversible Hydrogen Storage: *Bo Liu*[1]; Zhigang Fang[1]; Jun Lu[1]; Peng Fan[1]; [1]University of Utah

The search for onboard solid hydrogen storage materials have focused on complex metal hydride in recent years. The present work examines a new concept of hydrogen storage material based light metal solid solution alloys. Results of the experimental investigation of Li-Mg solid solution, which is a model system, are presented. Li-based solid solution can be prepared by using non-equilibrium processes such as rapid solidification and high energy mechanical alloying. The results showed that the temperature of dehydriding of the hydrogenated Li-Mg solid solution is much lower than that of monolithic LiH. The hydriding and dehydriding of the solid solution alloy of Li-Mg is reversible.

4:40 PM

Nano-Chemo-Mechanics of Hydrogen Embrittlement of Metals under Extreme Conditions: Shan Huang[1]; David McDowell[1]; *Ting Zhu*[1]; [1]Georgia Institute of Technology

Challenges associated with a hydrogen economy are substantial, ranging from hydrogen generation, storage and transportation. Hydrogen in high-pressure metallic containment systems causes the degradation that can result in sudden catastrophic fracture. A wide range of hydrogen embrittlement phenomena was attributed to the loss of cohesion of interfaces due to interstitially dissolved hydrogen. However, this concept has not been made sufficiently predictive, due to a lack of fundamental understanding of the chemo-mechanical processes of embrittlement. Here, by combining the atomistic simulation, thermodynamic theory of interfacial embrittlement, and electron theory of alloying, we analyze how the structure of grain boundaries influences the propensity of hydrogen chemisorption and consequent effects on embrittlement. Our results highlight the collective effects of multiple segregation sites in grain boundary embrittlement. Implications for efficient hydrogen storage are discussed by linking to a recent experiment showing that hydrogen embrittlement can be markedly reduced through grain boundary engineering.

5:00 PM

Production and Characterization of Supported Transition Metal Nanoparticles on Multi-Walled Carbon Nanotubes Functionalized by Gamma Irradiation and Chemical Process: *Jessika Rojas*[1]; Carlos Castano[1]; [1]Missouri University of Science and Technology

Recently, hydrogen has been the source of intense research and diverse methodologies to store it are being investigated. Single and multi-walled carbon nanotubes show promise as good options to achieve high storage. Although the current hydrogen storage capacity of CNTs does not fulfill the DoE requirements, further research is proposed about modifying the nanostructures to affect its properties. The main objective of this work is to evaluate mechanism of functionalization and purification by chemical and irradiation techniques to create functional groups in the surface and remove the caps at the end of the nanotubes to enhance the hydrogen storage capacity. Functional groups serve as a medium to attach metal nanoparticles to carbon nanotubes.SEM, STEM and XPS were used for morphological, chemical and structural characterization of the nanostructure.

5:20 PM Invited

Thermodynamics of Nano-Cluster Complex Hydrides Using First-Principles Density Functional Theory: *Eric Majzoub*[1]; Vidvuds Ozolins[2]; Fei Zhou[2]; [1]University of Missouri - St. Louis; [2]UCLA

While recent interest has focused on complex metal hydrides such as $NaAlH_4$ and $Ca(BH_4)_2$, these compounds are not as easily tunable as the interstitial metallic hydrides through alloying with other metal atoms. We investigate these materials at the nano-scale, where the ratio of surface to bulk atoms impacts the energetics. We present theoretical results for desorption energetics of nano-clusters of $NaAlH_4$. Prototype geometries for the clusters were generated using a well-validated electrostatic ground state approach to a global optimization of the cluster total energy using a recently developed non-conventional Monte Carlo random walk in energy space. First-principles density functional theory applied to the prototype clusters

was used for full free energy calculations of the clusters and decomposition products. Results will be discussed with relation to recent experimental work on incorporation of complex hydrides in nanoporous framework materials, and the importance of the surface chemistry of the frameworks.

ICME: Overcoming Barriers and Streamlining the Transition of Advanced Technologies to Engineering Practice -- The 12th MPMD Global Innovations Symposium: Modeling and Simulation Tools

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division
Program Organizers: Paul Mason, Thermo-Calc Software Inc; Mei Li, Ford Motor Company; James Warren, National Institute of Standards and Technology; Jeff Simmons, AFRL

Monday PM
February 28, 2011
Room: 7A
Location: San Diego Conv. Ctr

Session Chairs: Jeff Simmons, AFRL; David McDowell, Georgia Institute of Technology

2:00 PM Invited
Open Source Calphad Software – Friend or Foe?: *Ursula Kattner*[1]; [1]National Institute of Standards and Technology

In the early days of Calphad software was freely available, such as the Lukas' suite of programs or SOLGASMIX. Development of free packages has virtually stopped with Lukas' retirement and incorporation of SOLGASMIX into commercial software. Commercial software packages are constantly being extended and improved and have the benefit of the availability of support. Recently, Gibbs, a suite for the calculation and visualization, was introduced as open source software for thermodynamic calculations. A potential problem for developers of commercial Calphad software is the fairly small market size and that new products may have a negative impact on the sustainability of existing products. On the other hand, the advance cost of a license may deter newcomers from trying the method and/or restrict coupling of Calphad calculations with other calculations or simulations. The potential impact of open source software will be discussed using experience from thermodynamic databases for which similar issues apply.

2:25 PM Invited
FiPy: PDE Performance in Python: *Jonathan Guyer*[1]; Daniel Wheeler[1]; James Warren[1]; [1]NIST

FiPy is an open source, object oriented, partial differential equation (PDE) solver, written in Python. FiPy is particularly tailored to phase transformation simulations, focusing on the phase field and level set methods. The solution of coupled sets of PDEs is ubiquitous to the numerical simulation of science problems. Numerous PDE solvers exist, using a variety of languages and numerical approaches. Many are proprietary and difficult to customize. As a result, scientists spend considerable resources repeatedly developing limited tools for specific problems. FiPy takes advantage of Python's existing suite of open source tools for array calculations, sparse matrices and data rendering to provide a tool that is extensible, powerful and freely available. This talk focuses on our efforts to allow users to easily exploit parallel computing environments, sophisticated linear solvers, and advanced matrix preconditioners to obtain maximum performance without distracting them from their science.

2:50 PM
Assessing Data Completeness and Predictive Potential in Magnesium Alloy Databases: *Kim Ferris*[1]; Dumont Jones[2]; [1]Pacific Northwest National Laboratory; [2]Proximate Technologies, LLC.

Effective materials property databases need data completeness, which considers the range of property values and sufficiency of system definitions. Property coverage and system definition are prerequisites for predictive structure-processing/property models. We have examined data completeness of Mg-alloy databases, finding current repositories do not exhibit full data completeness; however, their predictive capability can be substantially enhanced. Data completeness issues: 1) repositories do not completely specify individual alloy systems (e.g. processing); 2) reported Mg-alloy systems focus strongly on AZ series; and 3) measured properties show correlation in design and property spaces. Current information is sufficient to draw some conclusions for Mg-alloy design, and suggest design hypotheses. Improving predictive capability of repositories involves 1)better system definition (more detailed processing and post-treatment information), extracting basic mechanical measurements for "new" Mg-alloys, and testing hypotheses identified from current information. Authors acknowledge support from U.S. Department of Energy, Energy Efficiency and Renewable Energy, Office of Vehicle Technologies.

3:10 PM Invited
Accelerated Insertion of Materials with Incomplete or Uncertain Information: *Krishna Rajan*[1]; [1]Iowa State University

One of the critical barriers in transitioning new materials development into engineering practice is the uncertainty associated with the data that is used in the materials design process. While constitutive modeling strategies still form the foundations for computational materials design, uncertainty and incomplete information still pervades. Often intuition and/or heuristics experimentation is used to fill the gaps in evolving data needs. In this presentation we show how informatics methods helps to explore and analyze data where the information content and the taxonomy is not well defined. The talk aims to show that a formalism based on robust mathematical foundations, linked to materials engineering principles can be used to help in the design of materials in the context of their use in engineering practice. Suggestions on how this impacts the organization and use of databases to promote the philosophy of integrated computational materials engineering (ICME) are also discussed.

3:35 PM Break

3:50 PM Invited
Code Validation and Qualification – The YMP Case: *Patrice Turchi*[1]; [1]Lawrence Livermore National Laboratory

Having to tackle very challenging practical issues in an engineering-driven environment requires not only a quick solution but also in most cases code validation and qualification. For the purpose of illustration we will consider a series of studies conducted for the Yucca Mountain Project (YMP), although old and put on hold. One of the problems was to study the thermal integrity of the waste disposal canisters as a function of time with a combination of thermodynamic and kinetic modeling applied to complex multi-component alloys. After a brief review of the context of this project and the output of the materials study we will discuss the importance of code and results validation and qualification, and how it was done within this project. Then some general rules on how to qualify codes will be drawn.Work performed under the auspices of the U.S. DOE by LLNL under contract DE-AC52-07NA27344.

4:15 PM Invited
A Phase Field Study on Static Strain Aging Kinetics of Dual Phase Linepipe Steel: *Ning Ma*[1]; Neeraj Thirumalai[1]; Nathan Nissley[2]; Rick Noecker[3]; William Lamberti[1]; Raghavan Ayer[1]; [1]Corporate Strategic Research, ExxonMobil Research & Engineering Company; [2]ExxonMobil Upstream Research Company; [3]ExxonMobil Development Company

The increase of yield strength of steel during static strain aging is generally investigated following Cottrell's theory on Cottrell atmosphere formation. In the current study, we found that the time scale for strain aging of a dual-phase linepipe steel is several orders of magnitude longer than that needed for formation of "classical" Cottrell atmosphere. A Phase Field model was developed to study the kinetics of static strain aging considering the long range carbon diffusion. The observed strain aging kinetics can be rationalized by carbon diffusion across the ferrite grain from either trapped carbon in grain boundaries or neighboring carbon rich grains. The model was

extended to predict the increase in yield strength as a function of aging time and temperature. The predicted temperature dependence of yield strength produced by prolonged aging matches well with the experimental trend. Preliminary SIMS analysis in both aged and unaged samples will also be discussed.

4:40 PM

Determination of Microstructure-Property Correlations Using Phase Field Method: *Saswata Bhattacharya*[1]; Tae Wook Heo[1]; Kunok Chang[1]; Ricardo Lebensohn[2]; Long-Qing Chen[1]; [1]Pennsylvania State University; [2]Los Alamos National Laboratory

Properties of materials, such as elastic stiffness, yield strength, dielectric and piezoelectric constants, diffusivity, thermal conductivity, etc. are affected by their microstructures. Therefore, establishment of microstructure-property relation is crucial for designing new materials and improving their performance. We developed an integrated computational framework based on phase field methodology to compute the effective properties of structurally inhomogeneous solids. We applied our technique to compute the effective properties of materials containing cavities or inclusions, composites, multiphase and polycrystalline microstructures. Moreover, our method can be used to calculate residual field distribution and the response behavior in inhomogeneous microstructures. As an example, we employed our method to computing the residual stress distribution and determining stress-strain relation in elastically and plastically anisotropic polycrystals undergoing deformation. Further, we studied grain growth and texture evolution under applied load and structural and diffusional phase transformations under applied field to demonstrate how the microstructural evolution affects the microstructure-property correlations.

5:00 PM

The Hierarchy of Fatigue Damage Accumulation: *Sushant Jha*[1]; Christopher Szczepanski[1]; Craig Przybyla[2]; James Larsen[2]; [1]Universal Technology Corporation/Air Force Research Laboratory; [2]Air Force Research Laboratory

Fatigue damage is proposed to accumulate in an array of microstructural arrangements, which can be ranked in terms of the magnitude of deformation heterogeneity. These arrangements vary in size, combination of phases, and crystallographic orientations. This hypothesis rationalizes the dominance of crack growth lifetime (i.e., almost instantaneous crack-initiation) in the minimum fatigue behavior, which is often associated with an extreme microstructural arrangement. A computational study was conducted to explore the relationship between the occurrences of fatigue-critical microstructural arrangements and their rank, with the goal of predicting the probability of the life-limiting fatigue mechanisms. The analysis employed microstructures of a titanium alloy and a Ni-base superalloy simulated by an ellipsoid packing method. The computational study was complemented by detailed characterization of life-limiting microstructural configurations in both materials. The study also provided insights into the effect of microstructure, microtexture, and specimen volume on the probability of life-limiting failures in these materials.

5:20 PM

In-Situ Microscale Fatigue Study to Evaluate the Role of Microstructural Neighborhoods: *Christopher Szczepanski*[1]; Sushant Jha[1]; Paul Shade[1]; Robert Wheeler[2]; James Larsen[3]; [1]UTC/AFRL; [2]UES/AFRL; [3]Air Force Research Laboratory/RX

A recent study of fatigue crack-initiation mechanisms and their relationship to lifetime variability in bulk samples of Ti-6Al-2Sn-4Zr-6Mo has revealed that crack initiation can occur in a variety of characteristic microstructural neighborhoods. These microstructural neighborhoods, which vary in size and frequency of occurrence, are observed at fatigue crack initiation sites through fractography. However, fractographic investigations do not allow one to identify the underlying nature of the fatigue damage accumulation and cracking mechanisms. To characterize these mechanisms under controlled conditions in a scanning electron microscope, an in-situ micro-scale tensile testing technique has been adapted to complete fatigue tests on micro-specimens extracted from representative fatigue-critical microstructural neighborhoods. The results of these experiments will be presented, with an emphasis on identifying microstructurally driven cyclic damage accumulation processes leading to fatigue crack initiation. Additionally, the potential correlation between deformation severity and specific local microstructural configurations will be investigated to validate current mechanistic models.

Magnesium Technology 2011: Casting and Solidification

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Wim Sillekens, TNO Science and Industry; Sean Agnew, University of Virginia; Suveen Mathaudhu, US Army Research Laboratory; Neale Neelameggham, US Magnesium LLC

Monday PM
February 28, 2011

Room: 10
Location: San Diego Conv. Ctr

Session Chairs: Baicheng Liu, Tsinghua University; Elhachmi Essadiqi, CANMET

2:00 PM

Simulation of Porosity and Hot Tears in a Squeeze Cast Magnesium Control Arm: *Christoph Beckermann*[1]; Kent Carlson[1]; John Jekl[2]; R. Berkmortel[2]; [1]University of Iowa; [2]Meridian

Simulations are performed of the squeeze casting of AM60 and AZ91 automotive control arms. Advanced feeding flow and stress models are used within the casting simulation software to predict shrinkage porosity and hot tears. The simulations are validated through comparisons with observations made on experimental castings. Generally good agreement is obtained between the measured and predicted defect locations and extents. Design and process changes are introduced to mitigate the shrinkage and hot tear problems in these castings. The comparisons of the present study establish considerable confidence in the ability of casting simulation to predict shrinkage and hot tears in squeeze casting of magnesium alloys.

2:20 PM

Dendritic Microstructure in Directional Solidification of Magnesium Alloys: *Morteza Amoorezaei*[1]; Sebastian Gurevich[1]; Nikolas Provatas[1]; [1]McMaster University

We demonstrate morphological transitions in Mg-Al alloy dendritic microstructure as the cooling conditions change during steady state and transient directional solidification. The effect of temperature gradient on the transition is investigated numerically using two-dimensional phase field simulations. The six-fold symmetry of Mg alloys leads to very different dendrite morphologies than those encountered in alloys exhibiting four-fold surface tension anisotropy. In particular, we find that at high temperature gradients primary dendrites become columnar in the direction of thermal gradient. In contrast, in the regions where surface energy anisotropy is dominant, primary stalks cross at 60-degree angles that characterize hexagonal crystal structure. Our modelling observations are compared to new Mg-Al experiments.

2:40 PM

Effect of Fraction Solid and Injection Speed on Microstructures and Casting Defects of Magnesium Alloy in New Type Semi-solid Injection Process: *Yuichiro Murakami*[1]; Naoki Omura[1]; Mingjun Li[1]; Takuya Tamura[1]; Shuji Tada[1]; Kenji Miwa[1]; [1]Advanced Industrial Science and Technology

We have developed a new type semi-solid injection process, which can obtain high material yield of about 90% for magnesium alloy. In this process, generic magnesium billets are heated to the semi-solid temperature range in the injection cylinder without cover gasses and are injected into a mold. In this study, several billets were heated precisely in the cylinder to obtain desired fraction solid and the plate-like specimens were produced by injecting them under different injection speeds. The microstructures were observed by optical microscopy, and the casting defects were detected by

X-ray CT scanner. As increasing the injection speed, the shape and the size of alpha phase solid particles became more spherical, and smaller, and volume fraction of defects increased. Contrary, with higher fraction solid, volume fraction of defects decreased.Spheronization and miniaturization of solid particles was attributed to shear stress, and defects were affected by viscosity.

3:00 PM

Macrostructure Evolution in Directionally Solidified Mg-RE Alloys: Mario Salgado-Ordorica[1]; Willi Punessen[1]; Sangbong Yi[1]; Jan Bohlen[1]; Karl-Ulrich Kainer[1]; *Norbert Hort*[1]; [1]GKSS-Forschungszentrum

The mechanical properties of Mg-RE alloys, in particular their high strength at high temperatures, are well-known facts that make them promising materials for transport applications. However, since the quality of a specimen or the success of subsequent processing operations depends on the initial cast microstructure, an effort should be made on trying to understand and control the evolution of these alloy microstructures. To this end, Mg-RE alloys (where RE = Gd, Nd or Y) were cast by permanent mould direct chill casting. This process was performed in a specially optimized installation to ensure the obtention of ingots with homogeneous composition and free of porosity and inclusions. Different conditions were evaluated in order to better control the final grain size, orientation and distribution. The selection mechanisms operating during solidification, namely texturization and Columnar to Equiaxed Transition, were characterized and put into relation with the composition of the alloy and cooling conditions.

3:20 PM

Microstructure and Mechanical Behavior of Cast Mg AZ31-B Alloy Produced by Magnetic Suspension Melting Process: *Nathan Rimkus*[1]; Mark Weaver[1]; Nagy El-Kaddah[1]; [1]Univ of Alabama

Magnesium is the lightest of all structural metals and offers significant weight savings compared to traditional automotive materials. This paper describes macro/microstructure of Mg AZ31-B alloy produced via the Magnetic Suspension Melting (MSM) technique at a low superheat of 5°C. It was found that casting at this low superheat produced a fine cellular grain structure in comparison to a dendritic structure in conventionally cast alloys. The intermetallic phases were analyzed in detail and compared with the conventionally cast alloy. In the MSM cast alloy, the γMg17AL12 phase formed mainly at the grain boundaries, in contrast to typical dendritic entrapment of this phase within the grains in conventional castings. The formation of the Al-rich secondary-α phase during solidification was investigated. The effects of this morphology change on mechanical and fracture behavior of this material will be presented. These results will be discussed relative to conventionally cast Mg alloys.

3:40 PM

Investigations on Hot Tearing of Mg-Zn-(Al) Alloys: Le Zhou[1]; Yuanding Huang[1]; Pingli Mao[2]; Karl Ulrich Kainer[1]; Zheng Liu[2]; *Norbert Hort*[1]; [1]MagIC-Magnesium Innovation Centre, GKSS Research Centre; [2]Shenyang University of Technology

Mg-Zn alloys are widely used as wrought alloys such as ZM, ZK and ZE. It already had been reported that they are prone to hot tearing. The present work evaluates the hot tearing susceptibility (HTS) of binary Mg-Zn alloys using quantitative experimental methods and thermodynamic simulations based on Clyne's model. The results show that the curve of the HTS vs. the content of Zn has a typical "λ" shape. With increasing the content of Zn, the HTS increases firstly, reaches the maximum at 1.5% Zn and then decreases again. The addition of Al in Mg-Zn alloys influences the HTS. In the Mg-Zn-Al ternary system, the HTS decreases with the increase of Al content. The effect of Zn content on HTS in ternary Mg-Zn-Al systems is similar to that observed in binary Mg-Zn alloys.

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Proportional Strength-Ductility Relationship of Non-SF6 Diecast AZ91D Eco-Mg Alloys: *Shae K. Kim*[1]; [1]Korea Institute of Industrial Technology

SF6 gas has been generally used for Mg alloys during melting and casting as a cover gas. The use of SF6 gas, however, will be limited owing to its significant impact on global warming. Non-SF6 process during melting and casting in diecasting industry has been proved with Eco-Mg alloys, simple addition of small amount of CaO into AZ91D and AM60B Mg alloys. This paper will show non-SF6 diecasting procedures for AZ91D Eco-Mg alloys. Cold-chamber and hot-chamber diecasting mass productions were performed by using a Toshiba 135-ton cold chamber and a Frech 200-ton hot chamber diecasting machines under CO2 atmosphere without SF6 gas. An emphasis will be on proportional strength and ductility relationship of Eco-Mg alloys, in part, due to high-quality melt, refined grain size and Al2Ca second phase strengthening. Microstructures and mechanical properties of AZ91D Eco-Mg alloys will be evaluated in comparison with those of conventional AZ91D Mg alloy.

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Estimation of Heat Transfer Coefficient in Squeeze Casting of Magnesium Alloy AM60 by Experimental Polynomial Extrapolation Method: *Zhizhong Sun*[1]; Xiaoping Niu[2]; Henry Hu[1]; [1]University of Windsor; [2]Promatek Research Centre, Cosma International

In this work, a different wall-thickness 5-step (with thicknesses as 3, 5, 8, 12, 20 mm) casting mold was designed and squeeze casting of magnesium alloy AM60 was performed in a hydraulic press. The casting-die interfacial heat transfer coefficient(IHTC) in 5-step casting was determined accurately based on thermal histories throughout the die and inside the casting which were recorded by fine type-K thermocouples. With measured temperatures, heat flux and IHTC were evaluated using the polynomial curve fitting method and numerical inverse method. For numerical inverse method, a solution algorithm is developed based on the function specification method to solve the inverse heat conduction equations. The IHTCs computed with these two techniques were compared. The results show that the peak values of IHTC at the thin steps are higher than those at the thick steps. However, the duration for IHTC remaining at the peak value increases as the step thickness increases.

5:00 PM

Wide Strip Casting Technology of Magnesium Alloys: *Woo-Jin Park*[1]; Jae-Joong Kim[1]; In-Joon Kim[1]; Dong-Kyun Choo[1]; [1]RIST

Extensive investigations relative to producing high performance and low cost magnesium sheet by strip casting have been performed for the applications to automotive parts and electronic devices. Research in order to develop magnesium sheet production technology has been begun from 2004 by RIST with support of POSCO. The POSCO has completed the plant to manufacture magnesium coil in the world's first. Big project in order to develop wide magnesium strip casting technology was started in succession. In this study, the recent results for wide magnesium strip casting are going to be presented. Wide magnesium coils of ~16 tons with 2,000mm width, 2,500mm diameter and 4~8mm thickness were successfully manufactured by strip casting process. Surface defects like inverse segregations could be minimized in the wide magnesium sheets by controlling adequately process parameters. As-strip cast microstructure consists of surface chill zone, dendritic columnar structure and central region.

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Microstructural Analysis of Segregated Area in Twin Roll Cast Mg Alloy Sheet: *Jae Joong Kim*[1]; Woo-Jin Park[1]; Dong Kyun Choo[1]; [1]RIST

Twin roll cast of magnesium alloy has been studied by various institutes since POSTECH started at the world first. RIST has done the R&D program of twin roll cast and reverse warm mill of magnesium sheet with 600 mm width. Presently, RIST is working on twin roll cast of magnesium sheet with 2,000 mm width. Twin roll cast magnesium alloy includes segregated area which is important due to controllable quality of final product. Here we present the microstructural analysis of segregated area of twin roll cast magnesium alloy to understand segregation phenomena. Especially, we studied on phase identification in segregated area, comparing the theoretical simulation. Our result shows that two kinds of phase morphologies are observed in the segregated area, not depending upon the position in the sheet. Phase identification shows that segregated area includes densely continuous or dispersed beta and phi phase, and dispersed Al_8Mn_5 and Mg_2Si.

5:40 PM

Development of the Electromagnetic Continuous Casting Technology for Mg Alloys: *Joonpyo Park*[1]; Myounggyun Kim[1]; Jong-Ho Kim[1]; Gyu Chang Lee[1]; U-Sok Yoon[1]; Woojin Kim[1]; [1]Research Institute of Industrial Science and Technology

Continuous casting technology for high-quality surface billets with fine-grained and homogeneous microstructure can be a solution for the cost barrier breakthrough. The latent heat of fusion per weight (J/g) of magnesium is similar to that of other metals. However, considering the heat emitted to the mould surface during continuous casting in meniscus region and converting it to the latent heat of fusion per volume, magnesium will be rapidly solidified in the mould during continuous casting, which induces subsequent surface defect formation. In this study, electromagnetic casting and stirring (EMC and EMS) techniques is proposed to control solidification process conveniently by compensating the low latent heat of solidification by volume and to fabricate magnesium billets of high quality surface. This technique was extended to large scale billets up to 300 mm diameter and continuous casting was successfully conducted. Then magnesium billet was used for the fabrication of prototype automobile pulley.

Magnesium Technology 2011: Primary Production; Characterization and Mechanical Performance

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Wim Sillekens, TNO Science and Industry; Sean Agnew, University of Virginia; Suveen Mathaudhu, US Army Research Laboratory; Neale Neelameggham, US Magnesium LLC

Monday PM
February 28, 2011

Room: 6F
Location: San Diego Conv. Ctr

Session Chairs: Neale Neelameggham, US Magnesium LLC; Adam Powell, Metal Oxygen Separation Technologies Inc.

2:00 PM

The Development of the Multipolar Magnesium Cell: A Case History of International Cooperation in a Competitive World: *Olivo Sivilotti*[1]; [1]Sigma Tau Technologies

The author conceived the first multipolar magnesium electrolytic cell in the late 1970s, in order to offset the impact of an 80kA monopolar cell being developed in cooperation with Osaka Titanium Company (OTC) being licenced by Alcan to a major competitor of OTC. During the 80s the commercial value of the multipolar magnesium cell technology was established. The 90s saw significant further progress in OTC, while attempts by Alcan to commercialize it in magnesium production plants met with technical difficulties and failure, due primarily to the lack of magnesium chloride feed of adequate quality. Now the author plans to commercialize a new multipolar cell design with a target productivity of 8.5-10 tons/day and a unit power consumption of 8.5-10 KWHR/kg of magnesium.

2:20 PM

Effect of KCl on Liquidus of LiF-MgF2 Molten Salts: *Sh Yang*[1]; Fengli Yang[1]; Xianwei Hu[2]; Zhaowen Wang[2]; Zhingning Shi[2]; Bingliang Gao[2]; [1]Jiangxi University of Science and Technology; [2]School of Materials and Metallurgy117#, Northeastern University

Liquidus temperature of KCl-LiF-MgF2 molten salts was determined based on cooling curve. The results proved the method of cooling curve was reliable and accurate by comparison liquidus temperatures of KCl. The measured value of KCl liquidus temperatures 769.27°C, and the documental value was 769.5°C. The different value was 0.23%, and the error ratio was 0.03%. The results were shown that effect of KCl on liquidus of molten salt was great for MgF2-LiF electrolyte. The liquidus temperatures were different with increasing of content of KCl in electrolyte. The liquidus temperature could be reduced 20°C with content of KCl in electrolyte from 0wt% to 50wt%. The experiments showed that the compound addition of KCl into LiF-MgF2 electrolyte not only reduced the liquidus temperature of electrolyte effectively but also decreased the aluminum-magnesium production cost and increased the economic benefits.

2:40 PM

Efficiency and Stability of Solid Oxide Membrane Electrolyzers for Magnesium Production: *Eric Gratz*[1]; Soobhankar Pati[1]; Jarrod Milshtein[1]; Adam Powell[2]; Uday Pal[1]; [1]Boston University; [2]Metal Oxygen Separation Technologies, Inc.

Solid oxide membrane (SOM) process has been successfully employed for the production of magnesium directly from its oxide. The process involves dissolving MgO in a fluoride based ionic flux and electrochemically pumping out the oxygen ions from the flux via an oxygen-ion-conducting SOM to the anode where they are oxidized, while reducing magnesium ions at the cathode. Understanding the long-term stability of the SOM in the flux is critical for the commercial success of this technology. This study utilizes electrochemical techniques such as impedance spectroscopy and linear sweep voltammetry to investigate key concepts related to MgO dissociation and current efficiency. Results show that the dissociation potential of MgO is dependent on the partial pressures at which magnesium is generated and the membrane stability is likely related to the current efficiency.

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Magnesium Production by Vacuum Aluminothemic Reduction of a Mixture of Calcined Dolomite and Calcined Magnesite: *Wenxin Hu*[1]; Naixiang Feng[1]; Yaowu Wang[1]; Zhihui Wang[1]; [1]Northeastern University

A new method of magnesium production was proposed that using a mixture of calcined dolomite and calcined magnesite with the molar ratio of MgO to CaO 6:1 as raw materials by vacuum aluminothermic reduction and extracted alumina from CaO•2Al2O3, the main phase of reduction slag analyzed by X-ray diffraction analysis. The results show that the reduction ratio of MgO was 86.18% at the conditions of 1414 K, 150 mins and was increased while reduction temperature and reduction time extended. The alumina leaching ratio of reduction slag reached 88% at the conditions of leaching temperature 95 ºC and the concentration ratio of Na2CO3 to NaOH was 100:75 in leaching Solution.

3:20 PM

Multiphase Diffusion Study for Mg-Al Binary Alloy System: *Young-Min Kim*[1]; Sazol Das[1]; Manas Paliwal[1]; In-Ho Jung[1]; [1]McGill University

Multiphase diffusion simulation and experimental studies are performed for Mg-Al binary alloy system at various temperatures. All simulations are based on the assumption that the inter-diffusion coefficients are composition independent and the interface always lies at equilibrium. Finite difference method (FDM) is used to solve diffusion equation and program is coded in FORTRAN with the evaluated self and inter diffusivity coefficients of Mg and Al. Annealing experiments of Mg-3wt% Al and Mg-6wt% Al were carried out at 330oC and 400oC for various times and the concentration profiles of Al were measured by Electron Probe Micro Analyzer (EPMA). The diffusion simulation results are in good agreement with both diffusion couple as well as annealing experiments.

3:40 PM

Experiments and Modeling of Fatigue of an Extruded Mg AZ61 Alloy: *J Jordon*[1]; J. Gibson[1]; M. Horstemeyer[1]; [1]Mississippi State University

In this study, structure-property relations with respect to fatigue of an extruded AZ61 magnesium alloy were experimentally quantified. Strain-life experiments were conducted in the extruded and transverse orientations under low and high cycle conditions. The cyclic behavior of this alloy displayed varying degrees of cyclic hardening depending on the strain amplitude and the specimen orientation. The fracture surfaces of the fatigued specimens were analyzed using a scanning electron microscope in order to quantify structure-property relations with respect to number of cycles to failure. Intermetallic particles were found to be the source of fatigue initiation on a majority of fracture surfaces. Finally, a multistage fatigue model based on the relative microstructural sensitive features quantified in this study was employed to capture the anisotropic fatigue damage of the AZ61 magnesium alloy.

4:00 PM Break

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Low-Cycle Fatigue Behavior of Die-Cast Mg Alloy AZ91: *Luke Rettberg*[1]; Warwick Anderson[1]; J. Wayne Jones[1]; [1]University of Michigan

An investigation has been conducted on the influence of microstructure and artificial aging response (T6) on the low-cycle fatigue behavior of super vacuum die-cast (SVDC) AZ91. Fatigue lifetimes were determined from total strain-controlled fatigue tests for strain amplitudes of 0.2%, 0.4% and 0.6%, under fully reversed loading at a frequency of 5 Hz. Cyclic stress-strain behavior was determined using incremental step test (IST) methods. Two locations in a prototype casting with different thicknesses and, therefore, solidification rates, microstructure and porosity, were examined. In general, at all total strain amplitudes fatigue life was unaffected by microstructure refinement and was attributed to significant levels of porosity. Cyclic softening and a subsequent increased cyclic hardening rate, compared to monotonic tests, were observed, independent of microstructure. These results, fractography and damage accumulation processes, determined from metallographic sectioning, are discussed.

4:40 PM
Small Fatigue Crack Growth Observations in an Extruded Magnesium Alloy: *J. Bernard*[1]; J. Jordon[1]; M. Horstemeyer[1]; [1]Center for Advanced Vehicular Systems- Mississippi State University

The purpose of this paper is to quantify the microstructurally small/physically small crack growth behavior in an extruded AZ61 magnesium alloy. Fully-reversed, interrupted load control tests were conducted on notched specimens that were taken from the transverse direction with respect to the extrusion direction. In order to measure crack growth, replicas of the notch surface were taken at periodic intervals. Scanning electron microscopy analysis of the replica surfaces revealed multi site crack initiation and subsequent crack coalescence. The crack growth behavior of the small fatigue cracks was shown to have a strong dependence on the material microstructure as the crack was submitted to a tortuous growth path along grain boundaries and crystallographic slip planes. A multi-stage microstructurally dependent crack growth model that was previously calibrated for FCC metals was further developed by this research for HCP materials.

5:00 PM
Applicability of Mg-Zn-(Y, Gd) Alloys for Engine Pistons: *Kazutaka Okamoto*[1]; Masato Sasaki[2]; Norikazu Takahashi[2]; Qudong Wang[3]; Yan Gao[3]; Dongdi Yin[3]; Changjiang Chen[3]; [1]Hitachi, R&D; [2]Hitachi Automotive Systems; [3]Shanghai Jiao Tong Univ

Commercial magnesium alloys have a great potential for structural applications in automotive due to their significant weight saving. However, they have poor creep resistance at temperature over 125°C, thus making them inadequate for power train applications such as engine pistons, which are operated at temperature up to 300°C. Recently, creep resistant magnesium alloys with rare earth elements and Zn have been developed, hence the applicability of Mg-Zn-(Y, Gd) alloys for engine pistons was investigated in this paper. Gravity casting was performed with Mg-Zn-(Y, Gd)-Zr ingot, followed by T6 treatment. High cycle fatigue tests were carried out and results were compared to the current aluminum cast alloy of A336 (JIS AC8A) for pistons. At room temperature, the fatigue strength is 27% lower than A336, while it is 35% higher at 300°C. The effects of microstructure on the mechanical properties will be discussed in the presentation.

5:20 PM
Compressive Creep Behaviour of Extruded Mg Alloys at 150°C: *Michelle Fletcher*[1]; Lukas Bichler[1]; Dimitry Sediako[2]; Robert Klassen[3]; [1]University of British Columbia; [2]NRC-Canadian Neutron Beam Centre; [3]University of Western Ontario

Wrought Mg alloy bars, sections and tubes have been extensively used in the aerospace, nuclear and automotive industries, where component weight is of concern. The operating temperature of these components is typically limited to below 100°C, since appreciable creep relaxation of the alloys takes place above this temperature. This study was focused on a selected wrought magnesium alloys developed for high temperature applications. Compressive creep behaviour of AE, ZE, AJ, AX and EZ rods was studied at room temperature and at 150°C using the microindentation creep technique. Measurements were obtained in the radial and transverse directions to observe the effect of texture on creep strain, hardness and elastic modulus. Microscopic examination of the alloys revealed that the distribution of second phases was critical to the alloy's creep resistance. Selected results were also related to in-situ compressive creep experiments carried out using neutron diffraction techniques.

5:40 PM
The Effect of Thermomechanical Processing on the Creep Behavior and Fracture Toughness of Thixomolded® AM60 Alloy: *Zhe Chen*[1]; Amit Shyam[2]; Jane Howe[2]; Jack Huang[3]; Ray Decker[3]; Steve LeBeau[3]; Carl Boehlert[1]; [1]Michigan State University; [2]Oak Ridge National Laboratory; [3]Thixomat Inc.

The effect of thermomechanical processing and subsequent heat treatment on the small fatigue crack growth behavior of a nanoMAG AM60 magnesium alloy will be reported. The investigated alloys have a finer grain structure and higher levels of yield strength compared to similar alloys with conventional processing treatment. The effect of maximum stress and load ratio on the growth rate of cracks emanating from artificial notches will be characterized and modeled. The notches were of the same size scale as natural defects in these materials and were fabricated by electro-discharge machining. Microstructure and fracture surface characterization will be performed by electron microscopy to develop the microstructure-fatigue property relationships in this class of lightweight alloys.

Magnetic Materials for Energy Applications: Magnetocaloric Materials

Sponsored by: TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Energy Conversion and Storage Committee, TMS: Magnetic Materials Committee; JSPS 147th Committee on Amorphous and Nanocrystalline Materials; Lake Shore Cyrotronics, Inc.; AMT&C
Program Organizers: Victorino Franco, Sevilla University; Oliver Gutfleisch, IFW Dresden; Kazuhiro Hono, National Institute for Materials Science; Paul Ohodnicki, National Energy Technology Laboratory

Monday PM
February 28, 2011
Room: 11A
Location: San Diego Conv. Ctr

Session Chair: Oliver Gutfleisch, IFW Dresden

2:00 PM Keynote
Towards Better Magnetocaloric Materials: *Vitalij Pecharsky*[1]; Karl Gschneidner[1]; [1]Iowa State University

Recent achievements in the design of robust near room temperature magnetic cooling devices signify paradigm shift in refrigeration, liquefaction and freezing technologies, and call for a much broader base of advanced magnetocaloric materials to support the quick realization of this environmentally friendly, energy efficient technology in a variety of markets. The latest materials discoveries and current trends in design of advanced magnetocaloric compounds will be reviewed. Work at the Ames Laboratory is supported by the U.S. Department of Energy – Basic Energy Sciences under contract No. DE-AC02-07CH11358.

2:40 PM Invited
Interplay between Structure and Magnetism in $Gd_5(Si_xGe_{1-x})_4$: *Daniel Haskel*[1]; [1]Argonne National Laboratory

The connection between atomic structure and magnetism in giant magneto-caloric effect (MCE) material $Gd_5(Si_xGe_{1-x})_4$ is explored using synchrotron radiation techniques. The element selectivity of x-ray absorption fine structure (XAFS) and additional orbital selectivity of x-ray magnetic circular

dichroism (XMCD) allow probing the magnetic polarization of conduction states responsible for mediating exchange interactions between localized Gd 4*f* electrons in the context of the magneto-structural phase transition. Furthermore, extension of these measurements to the diamond anvil cell allows a detailed comparison of the effects of chemical pressure (Si doping) and physical pressure upon the strength of magnetic interactions providing valuable insight into the role that Si/Ge *p*- Gd 5*d* hybridization plays in regulating the ferromagnetic ordering temperature of these materials. Work at Argonne is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

3:05 PM Invited

Novel Design of Magnetic Refrigerant Materials towards High Refrigeration Performance: *Julia Lyubina*[1]; [1]Imperial College London

Materials showing a large magnetocaloric effect around room temperature are of increasing interest due to their possible application in magnetic refrigeration. Very attractive in this respect are materials with a first order transition, since the latter results in a large (giant) entropy change. However, these materials usually possess magnetic and thermal hysteresis and are prone to fracture due to crystal symmetry and/or volume change accompanying the transition that reduces the efficiency or even makes the refrigeration cycle impossible. Here, we discuss how the magnetic and mechanical properties can be improved by special processing. It is shown that the design of porous microstructure in La(Fe,Si)13 materials is advantageous for the reduction of hysteresis and substantially improves mechanical stability. Practical implementation issues for the integration of La(Fe,Si)13 in cooling engines are discussed. This research is in part supported by a Marie Curie Intra European Fellowship within the 7th FP of EC.

3:30 PM

Adiabatic Temperature Change, Entropy Change and Magneto-Volume Effect in La-Fe-Si Alloys: A Study of Thermal and Magnetic Hystereses: *Konstantin Skokov*[1]; James Moore[1]; Jian Liu[1]; Maria Krautz[1]; Oliver Gutfleisch[1]; [1]Leibniz Institute for Solid State and Materials Research

Compounds with the first-order ferromagnetic-paramagnetic transitions seem to be very promising for magneto magneto-cooling technique due to very high values of the magnetic entropy change. However in comparison with the second-order transition, the first-order transition is unavoidably accompanied by field- and temperature-hystereses as well as latent heat transfer. During operational field or temperature cycling this irreversibility appears as an undesirable heating of the magnetocaloric material. Thus, the study on the issue of hysteresis is extremely important for development of an efficient magnetic refrigerator prototype. A comparative study of the magnetic hysteresis from isothermal M-H loops, the magnetic entropy change, the adiabatic temperature change and the volume change of $LaFe_{13-x}Si_x$ alloys undergoing the first-order magnetic transition has been carried out. Also, effects of temperature and field cycling on the magnetocaloric effect were investigated. By building the S-T diagram, a thermodynamic analysis of the first-order transition in $LaFe_{13-x}Si_x$ alloys has been performed.

3:45 PM

Magnetocaloric Study of Mechanically Alloyed LaFeSi: Mathieu Phejar[1]; *Lotfi Bessais*[1]; Valérie Paul-Boncour[1]; [1]CNRS

The room temperature magnetic refrigeration based on the magnetocaloric effect (MCE) is an alternative to conventional cooling. Much interest have been carried on La(Fe,Si)13 compounds since the discovery of their giant MCE associated with low cost. These compounds is hardly formed by a peritectic reaction between the liquid La and the solid solution of Fe,Si resulting in a inhomogeneous microstructure. We have synthesized those compounds by ballmilling which until now haven't been used for this kind of samples. While a heat treatment of one month is necessary to obtain the crystallization of the NaZn13 type structure for bulk alloys only 30 minutes is sufficient for ballmilled samples because of their finer microstructure. The magnetic properties of these compounds have been improved by substitution of Fe for Si and by insertion of hydrogen or carbon.

4:00 PM Break

4:15 PM

Field Effect on Martensitic Transition and Magnetocaloric Effect in $Ni_{50}Mn_{35.5}In_{14.5}$ and $Ni_{45.5}Mn_{43}In_{11.5}$ Ribbons: Wagner Rosa[1]; Tatiana Sánchez[1]; Javier García[1]; Victor Vega[1]; Victor Prida[1]; Lluisa Escoda[2]; Joan Suñol[2]; *Blanca Hernando*[1]; [1]Universidad de Oviedo; [2]Universidad de Girona

Ferromagnetic shape memory Ni-Mn-In Heusler alloys undergo a thermoelastic martensitic transformation (MT) from parent austenitic phase to a martensitic one on cooling [1]. The sensitivity of MT to the applied magnetic field becomes this material very suitable for alternative energy technologies due to magnetocaloric effect (MCE) [2]. We report on structural and magnetic properties for Ni-Mn-In melt-spun ribbons. Samples microstructure was checked by XRD and SEM together with their chemical composition by EDS. Magnetic properties were measured with a VSM in the temperature range of 50K-400K and up to 3 T. MT was characterized from the thermomagnetic measurements (ZFC-FC-FH) in different applied fields. MT behaves highly sensitive to the applied field and alloy composition. Direct and inverse MCE near Curie point and MT point, have been clearly revealed. [1] Z.D. Han, et al, Solid State Communications 146 (2008) 124. [2] B. Hernando, et al, Appl. Phys. Lett. 94 (2009) 222502.

4:30 PM

In-Situ High-Energy X-Ray Studies of Magnetic-Field-Induced Functional Behaviors in Ferromagnetic Shape-Memory Alloys: *Zhihua Nie*[1]; Yandong Wang[1]; Yang Ren[2]; [1]Beijing Institute of Technology; [2]Argonne National Laboratory

A large magnetic-field-induced strain (MFIS) found in ferromagnetic shape-memory alloys (FSMAs) has stimulated much attentions. The large MFIS can be achieved through (1) the rearrangement of martensite variants via twin boundary motion, such as NiMnGa, or (2) the phase transition from a phase with lower magnetization to a phase with higher magnetization, such as NiCoMnIn. Synchrotron-based high-energy X-ray diffraction (HEXRD) techniques provide an in-situ tool for tracing the microscopic evolution of structures under various environments. This talk will present our in-situ HEXRD studies of magnetic-field-induced functional behaviors in FSMAs. An internal-stress of ~8 MPa was uncovered in a NiMnGa single crystal after thermo-magnetic training, showing a new training mechanism by introducing internal-stress in FSMAs which aims to lower the twinning stress. In polymer-bonded NiCoMnIn composites, the magnetic-field not only induced an inverse martensitic phase transformation accompanied with a MFIS of ~1.76%, but also affected the phase fraction at low temperatures.

4:45 PM

Room Temperature Magneto-caloric Effect In Fe Substituted Ni-Mn-Sn Alloy: *Rahul Das*[1]; Perumal Alagarsamy[1]; Ananthakrishnan Srinivasan[1]; [1]Indian Institute of Technology Guwahati

Ferromagnetic shape memory alloys exhibiting high magnetic entropy change (ΔS_M) near room temperature are being developed for magnetic refrigeration application. $Ni_{49}Mn_{37}Fe_1Sn_{13}$ ($Ni_{47}Mn_{37}Fe_3Sn_{13}$) exhibit highest ΔS_M of ~6 $Jkg^{-1}K^{-1}$ (11 $Jkg^{-1}K^{-1}$) for 2 T field change at 241 K (177 K) [1]. In this work, we altered the composition of Ni-Mn-Sn-Fe system and obtained ΔS_M of 5.2 $Jkg^{-1}K^{-1}$ for 2 T field change in $Ni_{50}Mn_{36}Sn_{13}Fe_1$ alloy at 304 K. ΔS_M obtained is larger than those reported for Fe substituted Ni-Mn-Sn alloys near room temperature for the same field change. Since the properties of these alloys are strongly influenced by the composition and processing conditions, it is possible to improve the ΔS_M of these alloys further. Details of experiments and results obtained would be presented.[1] T. Krenke *et al. J. Appl. Phys.* 102 (2007) 033903.

5:00 PM

The Magnetocaloric Effect in Ni-Mn-Ga Heusler Alloys: *Mikhail Drobosyuk*[1]; Vasiliy Buchelnikov[1]; Sergey Taskaev[1]; [1]Chelyabinsk State University

In this work we study experimentally the positive magnetocaloric effect (MCE) in $Ni_{2+x}Mn_{1-x}Ga$ (x = 0.02, 0.03, 0.04, 0.07, 0.09) Heusler alloys. The MCE have been measured by the direct method. For the magnetic field change

$\Delta H = 2$ T, the maximal adiabatic temperature change ΔT_{ad} in $Ni_{2+x}Mn_{1-x}Ga$ alloys is larger than 0.6 K. The results show that the studied Heusler alloys may be interesting from the practical application point of view.

5:15 PM

Calorimetric and Magnetic Measurements of Magnetic Entropy Change in Pr0.5Sr0.49Ca0.01MnO3: *Ramanathan Mahendiran*[1]; [1]National University of Singapore

Perovskite manganites are considered to be one of the likely candidates for magnetic refrigeration due to significant magnetic entropy change exhibited by them. Majority of the existing work focussed on extracting magnetic entropy change from magnetization isotherms using Maxwell's relation. However, application of Maxwell's equation overestimates magnetic entropy in first order transition and independent verification of magnetic enetropy by thermal method is essential. We investigated magnetocaloric effect in Pr0.5Sr0.5-xCaxMnO3 series (x = 0 to 1) using differential scanning caloriemeter and magnetization isotherms. While magnetic measurement gave a large magnetic entropy change (= +10.9 J/kg K for a field variation of 5 T, calorimetric data gave smaller value (= +6.5 J/kg K) around the Neel temperature (TN = 140 K) but comparable values (= 4 J/kg K) around the Curie temperature (TC = 260 K). The large magnetic entropy around TN found by calorimetric method is promising for application.

5:30 PM

Influence of Heat Treatment on the Structure and Magnetic Properties of Gd_5Sn_4 Alloy for Magnetic Refrigeration: *Xichun Zhong*[1]; H Zhang[2]; M Zou[2]; Zhongwu Liu[1]; Dechang Zeng[1]; K.A. Gschneidner Jr.[2]; V.K. Pecharsky[2]; [1]South China University of Technology; [2]The Ames Laboratory, Iowa State University

The effects of heat treatment on the structure and magnetic properties of Gd5Sn4 alloy were investigated. In as-cast alloy, the main phase is orthorhombic Gd5Sn4, and two secondary phases are monoclinic Gd5Sn4 and hexagonal Gd5Sn3. After 1300°C annealing, the content of main phase increased from 85.39% to 98.47% and the monoclinic Gd5Sn4 phase disappeared. For as-cast alloy, an antiferromagnetism-ferromagnetism transition near 60K caused by Gd5Sn3 phase was evident. Two magnetic transitions near 82K and 130K are caused by the Gd5Sn4 main phase and monoclinic phase, respectively. For the annealed sample, the phase transitions near 60 and 130K are not significant due to the reduced secondary phases and the Tc of Gd5Sn4 phase decreases from ~82 to ~80K. The maximum magnetic entropy change in a low field of □H=20kOe increases from 21.5 to 24.65J/kgK after heat treatment. The results demonstrated that Gd5Sn4 alloy is a good magneticaloric material for refrigeration.

5:45 PM

Bonded Magnetocaloric Powders for the Refrigeration Application: *Spomenka Kobe*[1]; Benjamin Podmiljšak[1]; Marko Soderžnik[1]; Boris Saje[1]; Paul McGuiness[1]; [1]Jožef Stefan Institute

Magnetocaloric materials (MCM) based on Gd5Si2Ge2 with Fe as an additive show that the refrigeration capacity (RC) can be optimized with processing methods and substitution of various atoms in the lattice. Modified alloy can be used at room temperature. The hysteresis losses are drastically decreased with small decrease of net refrigeration capacity (NRC).The final product for refrigeration application should not be in the powder form and the idea is to use the discs, which will rotate in the applied magnetic field. The discs should be mechanically hard, but the amount of the bonding material should be as low as possible to prevent the dilution of the active magnetocaloric material. We used MCM with the best properties achieved, and various bonding materials. With the systematic experimental work we determined the composition, which enabled us the highest density with simultaneously good mechanical strength and magnetocaloric properties.

Massively Parallel Simulations of Materials Response: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: High Temperature Alloys Committee
Program Organizers: Diana Farkas, Virginia Tech; Susan Sinnott, University of Florida

Monday PM
February 28, 2011

Room: 1A
Location: San Diego Conv. Ctr

Session Chair: To Be Announced

2:00 PM Invited

Status of the ReaxFF Reactive Force Field: Development and Applications: *Adri van Duin*[1]; [1]Penn State

The ReaxFF method enables large-scale (>>1000 atoms) molecular dynamics simulations on chemically reactive systems. The method combines a bond order/bond distance concept with a polarizable charge method and employs relatively long-range bond orders, enabling a accurate description of transition state energies. While initially developed for hydrocarbons and first-row element chemistry, the method has currently been applied to a significant section of the periodic system, including covalent, metallic, ionic and ceramic materials. Furthermore, the ReaxFF program has been distributed to over 125 academic groups. In this presentation we will provide a status-report on the currently available ReaxFF force fields, the program environments that currently support ReaxFF, including a number of parallel options, and the plans for further method development. This includes recent extensions of ReaxFF to aqueous-phase chemistry, enabling applications to biochemical reactions, extensions and applications to combustion chemistry, applications to material failure and simulations on catalytic materials.

2:25 PM Invited

LIGGGHTS – Open Source Discrete Element Simulations of Granular Materials Based on LAMMPS: *Christoph Kloss*[1]; [1]JKU Linz

In this work, we present the development of an open-source software for modeling granular material by means of the Discrete Element Method. LIGGGHTS (www.liggghts.com) stands for 'LAMMPS Improved for General Granular and Granular Heat Transfer Simulations' and is based on LAMMPS ('Large Atomic and Molecular Massively Parallel Simulator'), a successful open source Molecular Dynamics code written and distributed by Sandia National Laboratories for massively parallel computing on distributed memory machines. We first give a brief overview of implemented models and features. These comprise CAD geometry import, features for particle insertion and packing, contact models, wallstress analysis, moving mesh capability, a 6 DOF feature and non-spherical particle handling. Finally, we would like to focus on the simulation of coupled granular-fluid systems with the CFD-DEM approach.

2:50 PM Invited

Parallel Parameterization of Models Using Noisy Simulation Data: *Steven Stuart*[1]; Dheeraj Chahal[1]; Sebastien Goasguen[1]; [1]Clemson University

Although massively parallel simulations of materials are becoming increasingly routine, parallelization of the optimization of the potentials needed in those simulations generally lags far behind. This talk describes some advances in automated parameterization methods that make efficient use of large-scale parallel processing environments. Rather than performing a deterministic minimization of a noise-free objective function, as with most automated parameterization methods, this approach acknowledges the stochastic nature of the data obtained from finite-duration dynamics and sampling simulations. Consequently, potentials can be successfully fit to physically relevant thermodynamic data, rather than relying too heavily on zero-temperature quantum mechanical energetic and structural data. Each stage in the parameterization process terminates as soon as it can be

determined that the parameters are non-optimal, but continues sampling as long as the parameters remain viable, rather than terminating at an arbitrary cutoff. The parameterization approach interfaces easily with massively parallel simulation engines, including LAMMMPS.

3:15 PM Invited

Effect of the Stress Field of an Edge Dislocation on Carbon Diffusion in α-Iron: Coupling Molecular Statics and Atomistic Kinetic Monte Carlo: *Michel Perez*[1]; [1]Université de Lyon - INSA de Lyon

Carbon diffusion near the core of a [111](I 01) edge dislocation in α-iron has been investigated by means of an atomistic model that brings together molecular statics and atomistic kinetic Monte Carlo (AKMC). Molecular statics simulations with a recently developed embedded atom method potential have been carried out in order to obtain atomic configurations, carbon-dislocation binding energies, and the activation energies required for carbon hops in the neighborhood of the line defect. Using information gathered from molecular statics, on-lattice AKMC simulations have been performed for temperatures in the 300–600 K range, so as to study the behavior of a carbon atom as it interacts with the edge dislocation stress field. This model can be seen as a very first step toward the modeling of the kinetics of carbon Cottrell atmosphere formation in iron during the static aging process.

3:40 PM Break

3:55 PM

Molecular Dynamics Simulation of Cavitation in Metallic Glass: *Michael Falk*[1]; Shuo Lu[2]; Pavan Valavala[1]; Michael Spector[1]; [1]Johns Hopkins University; [2]Beihang University

Experimental investigations indicate that cavitation may play a critical role in determining the fracture toughness of amorphous solids such as metallic glasses. We have undertaken a series of molecular dynamics simulations of cavitation under hydrostatic tension in a binary metallic glass analog using pair-wise potentials. We compare the rate of cavity nucleation directly to homogeneous nucleation theory to extract the role of irreversible deformation in the cavitation process. In doing so we are able to estimate the effective free energy for cavitation. Our simulations indicate that the barrier to cavitation is several orders of magnitude lower than that expected in the liquid. The implication of these measurements for the observed fracture behavior in metallic glasses will be discussed.

4:15 PM

Large Scale Simulation of 3D Nanocrystalline Mg by Molecular Dynamics: *Dong-Hyun Kim*[1]; M. V. Manuel[1]; F. Ebrahimi[1]; S. R. Phillpot[1]; [1]Department of Materials Science and Engineering, University of Florida

So far, comparing with f.c.c metals, very little has been done in large scale simulation of mechanical behaviors of h.c.p. metals. To understand nucleation and interaction of dislocations by slip and twinning, here we take Mg as a prototypical nanocrystalline h.c.p. metal. Atomistic process of slip and twinning created by a tensile stress was observed in fully densed 3D structure with nano-grain sizes. We address a length-scale dependence of twinning and slip in plastic deformation of nc-Mg. This work was supported by DOE NERI contract DE-FC07-07ID14833 and by the DOE-BES Computational Materials Science Network (CMSN).

4:35 PM

Large-Scale Molecular Dynamics Simulations of Tribology in Metallic Contacts: *Michael Chandross*[1]; [1]Sandia National Laboratories

Materials that perform well in electrical contacts usually exhibit high adhesion during frictional contacts. An excellent example of this phenomenon is pure gold, which has extremely low electrical contact resistance, but generally has a high friction coefficient. The exception to this, however, is nanocrystalline gold alloyed with minute amounts of Ni or Co, which in addition to its low contact resistance can also show low friction. The mechanism for this remains poorly understood. We will present the results of large scale molecular dynamics (MD) simulations which study the tribological response of both single crystal and nanocrystalline gold films in contact with curved probe tips under a variety of sliding conditions. Results from simulations of adhesion and friction in metallic alloys will also be presented.

4:55 PM

Molecular Dynamics Simulations of Nanoindentation of Si/SiO2 Systems using the Charge Optimized Many-Body (COMB) Potential: *Tzu-Ray Shan*[1]; Simon Phillpot[1]; Susan Sinnott[1]; [1]University of Florida

Oxides such as SiO2, Al2O3 and HfO2, are widely used together with Si in many high-performance electronic devices, including metal-oxide-semiconductor (MOS) devices/junctions and gate stacks. Since nanoindentation has been established as a primary tool for investigating the mechanical behavior of small volumes of materials, classical molecular dynamics simulation is used to examine the nanoindentation of Si/SiO2 interfacial systems. Because these systems consist of heterogeneous interface with significant changes in bonding as one crosses from one side of the interface to the other, the empirical charge optimized many-body (COMB) potential as implemented in LAMMPS is used to model the structural evolution, mechanical response and charge transfer of Si/SiO2 systems under the influence of a nanometer-scale spherical indenter. The SiO2 layers considered include a-quartz and amorphous silica with various thicknesses. Aspects of the Si/SiO2 interface during nanoindentation, including dislocation formation and the mechanisms by which fracture occurs, will also be addressed.

5:15 PM Invited

Shear Transformation Zone Dynamics Modeling of Metallic Glasses: *Christopher Schuh*[1]; Eric Homer[1]; [1]MIT

A thorough understanding of deformation in metallic glasses has proven elusive as a result of the disparate time and length-scales that span between the unit processes of deformation and the macroscopic flow localization events that are experimentally observed. We attempt to model the deformation of metallic glasses using a meso-scale model that treats the "shear transformation zone" (STZ) as a fundamental unit of deformation. STZ dynamics combines finite element mechanics, to solve for the stress redistribution when STZs operate, with a kinetic Monte Carlo algorithm, to model the kinetics of their activation sequence. The method allows processing and deformation to be simulated on larger length-scales and longer time-scales than, e.g., atomistic simulations. The method can be implemented in two or three dimensions, and can provide insight on deformation in complex experimental situations, such as in the formation of shear bands or the development of microplasticity around stress concentrators.

5:40 PM Invited

Modeling Shear Band Formation and Propagation in Bulk Metallic Glasses and Nanoglasses: Daniel Sopu[1]; Yvonne Ritter[1]; *Karsten Albe*[1]; [1]TU Darmstadt

Plastic deformation in metallic glasses is highly localized in shear bands and there is a broad consensus that shear transformation zones (STZs) are the basic units of plastic deformation. In this work, we study plastic deformation of bulk metallic glasses and nanoglasses by molecular dynamics simulation using LAMMPS. We investigate homogeneous shear band nucleation by simulating tensile deformation of a 3-dimensional-periodic metallic glass sample and compare it to shear band nucleation in the presence of free surfaces and interfaces as present in nanoglasses. In contrast to earlier studies, our results show only little influence of surfaces and interfaces on the shear band nucleation process and the stress-strain-behavior. We conclude that shear band formation involves the condensation of a critical number of STZs along a feasible shear path. Finally, we compare structural features present in shear bands to those found in internal interfaces present in nanoglasses.

Materials for the Nuclear Renaissance II: Irradiation Effects

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS/ASM: Nuclear Materials Committee

Program Organizers: Raul Rebak, GE Global Research; Brian Cockeram, Bechtel-Bettis; Peter Chou, Electric Power Research Institute; Micah Hackett, TerraPower, LLC

Monday PM
February 28, 2011

Room: 4
Location: San Diego Conv. Ctr

Session Chair: Micah Hackett, KAPL

2:00 PM Introductory Comments

2:05 PM

Evaluation of Scale Bridging Methodology for Performance Prediction: *Dongsheng Li*[1]; Yulan Li[1]; Fei Gao[1]; Ram Devanathan; Xin Sun[1]; Moe Kahleel[1]; [1]Pacific Northwest National Laboratory

Effective property prediction is the cornerstone for performance evaluation of materials under irradiation condition. Molecular dynamics method is used to calculate atomic-level properties of materials, while phase field model is used to predict the microstructure evolution. Different methods have been developed to bridge low scale models with polycrystalline level models. Finite Element Analysis (FEA), microstructure informed FEA, self consistent models, Taylor models, Sachs models and statistical continuum mechanics are evaluated. Cost, efficiency and accuracy were compared for these methods. Pros and cons, depending on the problems studied, are discussed. While open to discussion, general recommendation on bridging methodology is presented based on different scenarios.

2:25 PM

Localized Deformation in Proton and Heavy Ion Irradiated Austenitic Stainless Steels: *Zhijie Jiao*[1]; Gary Was[1]; Terumitsu Miura[2]; Koji Fukuya[2]; [1]University of Michigan; [2]Institute of Nuclear Safety System

Recent studies have shown that among the various features of the irradiated microstructure, localized deformation correlates most strongly with irradiation-assisted stress corrosion cracking (IASCC). The degree of localized deformation is influenced by irradiated microstructure and possibly by the irradiation depth if that depth is less than the grain size. In this study, two austenitic stainless steels, 304SS and 316SS, were irradiated to 5 dpa using 1.2 and 2 MeV protons and to 100 dpa using 2.8 MeV Fe++ at 300\176C. Constant extension rate tensile tests were conducted on the irradiated samples to 2% stain in both argon and water environments. The degree of localized deformation (the weighted average dislocation channel height) was characterized using atom force microscopy and crack initiation was examined using scanning electron microscopy. The effect of the degree of localized deformation on IASCC and the influence of irradiation depth on localized deformation will be presented and discussed.

2:45 PM

Toward a Better Understanding of Heat-to-Heat Variations in Irradiation-Assisted Stress Corrosion Cracking: *Peter Chou*[1]; Raj Pathania[1]; Bob Carter[1]; [1]Electric Power Research Institute

There are marked heat-to-heat variations in the susceptibility of materials to environmentally-assisted cracking in LWR environments. Although, in time, all heats will be affected by EAC, it is the "early" failures of a minority of heats in the field that consume and attention and resources of the industry. Heat-to-heat variations imply a gap in knowledge between microstructure/microchemistry and materials performance; and better understanding will influence how the industry should approach testing, screening, and development of existing and new materials. This presentation will describe ongoing EPRI-sponsored research on the application of atom-probe tomography to irradiated stainless steels and discuss how the observations serve as motivation for future research to support existing and new plants.

3:05 PM

Multiscale Simulation of the Effect of Irradiation-Induced Microstructure Evolution on Reactor Fuel Performance: *Michael Tonks*[1]; Paul Millett[1]; Derek Gaston[1]; Bulent Biner[1]; Anter El-Azab[2]; [1]Idaho National Laboratory; [2]Florida State University

Fuel performance in nuclear reactors is highly dependent on irradiation-induced microstructure evolution. Therefore, a fuel performance code must consider atomistic and mesoscale effects in order to provide a predictive capability. In this work, we present the multiscale fuel performance modeling approach currently being employed at Idaho National Laboratory (INL). Atomistically-informed mesoscale phase field simulations are used to determine the effect of irradiation-induced microstructure evolution on bulk properties, such as thermal conductivity and density. Continuum expressions describing the effect of irradiation on the bulk properties as a function of temperature are then determined from the mesoscale simulations. Finally, these expressions are used in INL's BISON fuel performance code to model fuel pellet behavior.

3:25 PM Break

3:35 PM

Mechanical Properties of Fresh and Neutron Irradiated U-Mo Fuels for the RERTR Application: *Ramprashad Prabhakaran*[1]; D Burkes[1]; A Robinson[1]; J-F Jue[1]; A DeMint[2]; J Gooch[2]; D Keiser[1]; D Wachs[1]; I Charit[3]; [1]Idaho National Laboratory; [2]Y-12 National Security Complex; [3]University of Idaho

The Reduced Enrichment for Research and Test Reactors (RERTR) program was initiated to develop new nuclear fuels to enable the research and test reactors to use low-enriched uranium fuels instead of high-enriched uranium fuels, without significant loss in performance. Hence, a new monolithic fuel type that possesses the greatest possible uranium density in the fuel region is being developed, where the fuel region consists of a single foil encased inside an aluminum alloy cladding. Currently, efforts are ongoing to evaluate the mechanical properties and microstructure of fresh and irradiated fuels as a function of molybdenum content, carbon content, burn up and fabrication method (Friction Bond and Hot Isostatic Pressing). Small-scale specimen testing techniques, such as sub-size tensile testing, microindentation hardness testing and shear punch testing are being performed. Other materials characterization techniques, such as optical microscopy, XRD and SEM are being used in conjunction with the small-scale mechanical test methods.

3:55 PM

Micro-Displacement Measurement for Small Samples in the Advanced Test Reactor (ATR) National Scientific User Facility (NSUF): *Anthony Santo Domingo*[1]; Mitchell Meyer[2]; Denis Beller[1]; [1]Univ. of Nevada, Las Vegas; [2]ATR National Scientific User Facility

The ATR NSUF at the Idaho National Laboratory (INL) provides a high-flux environment for long-term irradiation experiments. Battelle Energy Alliance (BEA) and the Electric Power Research Institute (EPRI) are fielding an experiment that will obtain data on the growth rate of zirconium alloys versus hydrogen content. This project will be conducted to investigate a postulated hydrogen-assisted irradiation growth rate mechanism that may be operative in zirconium alloys and that is associated with channel bowing in BWRs. In the project described herein, University of Nevada, Las Vegas faculty and students and INL personnel collaborated in an ATR NSUF Faculty-Student Research Project to design, build, and test a device that will accurately measure the micro-displacement of pre-irradiated and post-irradiated specimens of zirconium alloya. The design, fabrication, and operation of the micro-displacement measurement device will be described.

4:15 PM
MC Calculation of Optimal Gd and B Containing Composition for Effective Protection from Gamma and Neutron Irradiation: *Nikoloz Chikhradze*[1]; Leri Kurdadze[2]; Guram Abashidze[1]; [1]Mining Institute/ Georgian Technical University; [2]G. Tsulukidze Mining Institute

The radiation materials have different applications, including: working in nuclear reactors, localization of uncontrolled radiation sources, safety storage of radiation wastes and etc.Accordingly, it is actual to create composite materials having the appropriate ratio mixtures of constituents, which can produce stable physical-chemical and radiation-resistant properties and characterized with abilities of inactivated absorption of various types of neutron and gamma irradiation. In the paper the results of calculation of sorption properties of Me–Gd and Me-B composite materials with various ratio of B and Gd are discussed. Calculations was performed by the Monte-Carlo method with a program package GEANT3 including a special package GCALOR for the simulation of the interaction of thermal and fast neutrons within the sample. During the neutron passage through the sample all main processes of the interaction of neutrons with matter are considered including: elastic and inelastic scattering, neutron fission of nuclei, radiation capture, etc.

4:35 PM
Microstructure and Mechanical Properties of Irradiated Friction Stir Welded ODS Alloys: *Ramprashad Prabhakaran*[1]; J Wang[2]; I Charit[3]; J Cole[1]; KL Murty[4]; R Mishra[2]; [1]Idaho National Laboratory; [2]Missouri University of Science and Technology; [3]University of Idaho; [4]North Carolina State University

Conventional fusion welding of oxide dispersion strengthened (ODS) alloys causes various undesirable effects, such as coalescence of oxide dispersoids and significant porosity. In this study, MA956 and MA754 alloys were friction stir welded in a bead-on-plate configuration. Microhardness, shear punch and mini-tensile testing of the base and processed materials were carried out to evaluate the mechanical properties. Optical microscopy and transmission electron microscopy were used to study the changes in the grain size and particle characteristics across the weld zone. Efforts are ongoing to study the effect of neutron irradiation (at the Advanced Test Reactor, Idaho Falls) on the friction stir welded MA956 and MA754 alloys. Microhardness and shear punch tests are being carried out on the irradiated samples (1 and 2 dpa), and will be compared with the properties of the unirradiated materials.

Materials in Clean Power Systems VI: Clean Coal-, Hydrogen Based-Technologies, and Fuel Cells: Materials for Gasification and Turbines I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Conversion and Storage Committee, TMS: High Temperature Alloys Committee
Program Organizers: Xingbo Liu, West Virginia University; Zhenguo "Gary" Yang, Pacific Northwest National Laboratory; Jeffrey Hawk, U.S. Department of Energy, National Energy Technology Laboratory; Teruhisa Horita, AIST; Zi-Kui Liu, The Pennsylvania State University

Monday PM
February 28, 2011

Room: 33C
Location: San Diego Conv. Ctr

Session Chair: Xingbo Liu, West Virginia University

2:00 PM Keynote
Meeting the Materials Challenges to Enable Clean Coal Technologies: *Cynthia Powell*[1]; [1]Office of Research and Development, National Energy Technology Laboratory, US Department of Energy

Realization that the environmental impact of energy production must be reduced on a global scale, combined with an increased desire in this country to reduce dependence on foreign energy sources, is driving significant change in the way the United States will derive and produce power in the future. While renewable energy resources will continue to grow in importance, environmentally responsible fossil energy production will be necessary to provide a bridge to the next energy revolution. This drive to increase efficiencies and reduce environmental impact in fossil-based energy production will lead to increased operating temperatures and pressures and increasingly aggressive operating environments. The practical result is a requirement for affordable and reliable high-performance materials and materials systems to enable these next-generation fossil energy systems. This talk will focus on the research being performed at the National Energy Technology Laboratory to meet this requirement for high performance yet affordable materials.

2:30 PM Keynote
Mechanistic Understanding of Materials Degradation Processes in Solid Oxide Fuel Cell (SOFC) Power Generation Systems: From Bulk to Interfacial Reactions: *Prabhakar Singh*[1]; [1]University of Connecticut

High temperature operation of solid oxide fuel cells (600-1000C), exposure of cell components to dual atmosphere, and the presence of complex gas environment have been associated with severe surface and bulk corrosion and structural degradation conditions in cell and the balance of plant (BOP) components. This presentation provides an overview of materials degradation and corrosion processes operating in cell stack and BOP. Role of bi-polar exposure condition on accelerated corrosion has been investigated. Oxide evaporation in oxidizing and reducing atmospheres has also been investigated and its influence on the catalytic and electrocatalytic poisoning will be reported. Pertinent electrode structural changes and interfacial compound formation will be examined and discussed. Current research trend will be presented.

3:00 PM Invited
The Impact of Mixed Carbon Feedstock on High Cr2O3 Refractory Liners Used in Commercial Gasifiers: *James Bennett*[1]; Kyei-Sing Kwong[1]; Seetharaman Sridhar[2]; Jinichiro Nakano[3]; [1]NETL, US DOE; [2]Carnegie Mellon University; [3]URS Corp.

Gasifiers are high temperature/pressure reaction vessels used to contain interactions between a carbon feedstock, water, and oxygen (reducing environment); producing CO and H2 used in power generation or chemical production. Carbon feedstock includes coal and/or petcoke, with biomass considered a candidate to produce a carbon neutral process. Impurities in carbon feedstock materials vary from about 1-10 pct and liquefy during gasification (1325-1575°C, 10-8 O2 partial pressure), forming slags that interact with the high chrome oxide refractory liner by chemical corrosion and spalling, causing unpredictable and premature shutdown of a gasifier. Mixtures of carbon feedstock that include biomass will have an unknown impact on slag properties and refractory liners, and are the focus of ongoing research. Slag properties and slag/refractory interactions are being evaluated by post mortem analysis of commercial gasifier samples, in laboratory simulations, and by thermodynamic modeling. Results of these studies to data will be presented.

3:25 PM Break

3:40 PM
Stability of Mullite and V2O3 in Synthetic Slags Based on Molten Coal/ Petcoke Ash Mixtures: *Jinichiro Nakano*[1]; Seetharaman Sridhar[2]; Kyei-Sing Kwong[1]; James Bennett[1]; Thomas Lam[1]; [1]NETL; [2]Carnegie Mellon University

In gasifiers, non-volatiles such as SiO2 in coal and petcoke carbon feedstock form slags that can impact gasifier efficiency and increase refractory corrosion. In this study, synthetic slags simulating coal-petcoke ash mixtures were evaluated for phases existing in a gasification environment at 1500°C and Po2=10-8atm. VxOy-bearing phases are of particular interest because their stabilities are governed by Po2. Slag mixtures of SiO2-Al2O3-Fe2O3-CaO-V2O5 containing 0-10wt.% of V2O5 were equilibrated at 1500oC and rapidly quenched to freeze high temperature phases. XRD analyses identified mullite (in Al2O3-rich slags) and V2O3 (in V2O5-rich slags) as stable

phases. TEM and EPMA analyses indicated that the mullite contained V and V2O3 contained Al. Concentration depended on starting slag composition. XRD indicated that lattice parameters of the two crystalline phases were found to vary with elemental substitution. The presence of the solid phases is discussed in light of its impacts on slag fluidity and refractory erosion.

4:00 PM

Effect of Temperature Gradient on Slag Infiltration into Porous Refractory Material: *Tetsuya Kaneko*[1]; Jinichiro Nakano[2]; Seetharaman Sridhar[1]; Kyei-Sing Kwong[2]; Rick Krabbe[2]; Hugh Thomas[2]; James Bennett[2]; [1]Carnegie Mellon University; [2]US Department of Energy National Energy Technology Laboratory

Entrained-flow slagging gasifiers produce syngas (CO and H2) using coal, petcoke, and/or biomass as a carbon feedstock. The high operating temperatures (1325-1575oC) of these gasifiers, along with corrosive gases and slags from the carbon feedstock pose material challenges in the gasifier. Slags originating from mineral impurities in the carbon feedstock liquefy, corrode, and penetrate the porous refractory liner. The penetration characteristics of coal slag into alumina refractory have been studied using a temperature gradient along the slag's penetration direction, which are compared to those obtained under isothermal conditions. The studies were conducted with a hot face temperature of 1450°C and a CO/CO2 ratio of 1.8 (corresponding to an oxygen partial pressure of approximately 10-8 atm). Slag-infiltrated refractory samples were studied using SEM-EDS to identify primary phases and to track the slag's changing chemical composition. Primary phases and slag compositions were compared to those calculated in using FactSageTM software.

4:20 PM Invited

Creep Strength Property of Advanced Ferritic Creep Resistant Steels for Fossil Energy Applications: *Kazuhiro Kimura*[1]; Kota Sawada[1]; [1]National Institute for Materials Science

An overestimation of long-term creep strength was pointed out on creep strength enhanced ferritic (CSEF) steels and premature failure due to Type IV cracking and remarkable drop in creep rupture ductility were also recognized. Microstructural evolution is influenced by stress and that is different in the high- and low-stress regimes divided by a macroscopic elastic limit where successive plastic deformation of polycrystalline material initiates which corresponds to 50% of 0.2% offset yield stress. A region splitting analysis method was proposed for a reliable life prediction of CSEF steels and allowable stress was reduced. Creep rupture ductility drop is caused by a combination of high creep strength and stress dependence of creep deformation. In order to ensure strength and soundness, upper limit of aluminum was reduced from 0.04mass% to 0.02mass% and maximum level of 0.01mass% was specified on titanium and zirconium, as well as weld strength reduction factor.

4:45 PM

A Comparison of Creep-Rupture Tested Cast Alloys HR282, IN740 and 263 for Possible Application in Advanced Ultra-supercritical Steam Turbine and Boiler Components: *Neal Evans*[1]; Philip Maziasz[2]; Yukinori Yamamoto[2]; Paul Jablonski[3]; [1]University of Tennessee, Knoxville; [2]Oak Ridge National Laboratory; [3]National Energy Technology Laboratory

Cast forms of traditionally wrought Ni-base precipitation-strengthened superalloys are being considered for service in the ultra-supercritical conditions (760°C, 35MPa) of next-generation steam boilers and turbines. After casting and homogenization, these alloys were given heat-treatments typical for each in the wrought condition to develop the gamma-prime phase. Specimens machined from castings were creep-rupture tested in air at 800°C. In their wrought forms, alloy 282 is expected to precipitate $M_{23}C_6$ within grain boundaries, alloy 740 is expected to precipitate several grain boundary phases including $M_{23}C_6$, G Phase, and η phase, and alloy 263 has $M_{23}C6$ and MC within its grain boundaries. This presentation will correlate the observed creep-life of these cast alloys with the microstructures developed during creep-rupture tests, with an emphasis on the phase identification and chemistry of precipitated grain boundary phases. The suitability of these cast forms of traditionally wrought alloys for turbine and boiler components will also be discussed.

5:05 PM

Mesoscopic Finite Element Analysis of Interfacial Failure in High Temperature Alloys: Changsoo Kim[1]; *Keeyoung Jung*[2]; Frederick Pettit[2]; Gerald Meier[2]; [1]University of Wisconsin-Milwaukee; [2]University of Pittsburgh

A numerical model has been developed employing mesoscopic finite element analysis (FEA) to study interfacial failure in high temperature alloys and coatings. Using this model, the impacts of mesoscopic details on the thermo-mechanical performance such as different oxide compositions, types, sizes and shapes of encapsulated particles, and geometries of scales can be quantitatively estimated. The developed algorithm was applied to the scale buckling phenomena in an outward-growing thermal oxide that may occur in syngas-fired gas turbines assuming that brittle interfacial fracture occurs in Mode II. Experimentally observed microstructures with measured thermo-mechanical parameters were incorporated in the model. The presentation will discuss and elucidate the potential scale decohesion mechanism observed in experiments. This FEA model can be readily expanded to thermo-mechanical issues in other high temperature material applications that are integrated with clean energy systems such as scale spallation from metallic SOFC interconnects.

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 Organizers: Prince Anyalebechi, Grand Valley State University; Srikanth Bontha, Temple University

Monday PM — Room: 12
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chair: Prince Anyalebechi, Grand Valley State University

2:00 PM

Computational Modeling of Field-Assisted Sintering: *Cristina Garcia Cardona*[1]; Eugene Olevsky[1]; Veena Tikare[2]; [1]San Diego State University; [2]Sandia National Laboratories

A three-dimensional multi-scale model of Field-Assisted Sintering is presented. The model captures the complexity of the multiphysics phenomena involved in field-assisted sintering by coupling: electrical, thermal, stress-strain and densification components. The calculations are based on the finite-element (macroscopic scale) and kinetic Monte-Carlo (mesoscopic scale) codes. Specimen's macroscopic behavior is described through a non-linear viscous constitutive relation. The simulation of the densification is based on local conditions, and micro-scale factors as grain growth are also taken into account. Thus not only distributions of current density, temperature and strain fields can be obtained but also a spatial density and grain size evolution can be computed. The sintering constitutive parameters are determined through Monte-Carlo simulations of the grain-pore structure evolution. This corresponds to a true 3D, multi-scale, multiphysics modeling of sintering. The modeling predictions are discussed via several examples and calculation results are compared with experimental data available.

2:15 PM

Simulation of Macrosegregation Due to Melt Convection and Grain Sedimentation in Steel Ingots Using a Mixture Model: Wensheng Li[1]; *Houfa Shen*[1]; Baicheng Liu[1]; [1]Tsinghua University

In order to understand the formation of macrosegregation in steel ingots, a mixture solidification model has been developed which accounts for coupled mass, momentum, species, and energy transport and notably describes the sedimentation of free equiaxed grains. In this model, the mushy zone

is divided into the slurry and porous regimes according to a solid packing fraction. Settling velocity of equiaxed grains in the slurry regime is given by an explicit expression. The solidification of a binary steel ingot (Fe-0.36 wt pct C) with a two-dimensional geometry is numerically simulated. It is demonstrated that the predicted macrosegregation patterns agree to some extent with classical experimental results. Further refinement of this model is still necessary for realistically simulating steel ingot solidification.

2:30 PM

Effect of Casting Speed on Temperature Difference between Copper Plate and Solidifying Shell in Meniscus of Slab Continuous Casting Mold: *Xiangning Meng*[1]; [1]Northeastern University

A three-dimensional finite-element heat-transfer model was established to predict temperature of hot copper plate in a slab continuous casting mold and effect of casting speed on temperature difference between copper plate and solidifying shell was simulated in combination with process data. The results show that centre temperature of hot copper surface at casting speed 1.8 m•min-1 and 2.0 m•min-1 are higher than that of 1.6 m•min-1 casting speed 4.7-5.2 °C and 11.2-12.2 °C respectively and temperature is not increased linearly with casting speed. Temperature difference in meniscus between mold wall and shell surface is influenced obviously by casting speed and increased 4.0-6.0 °C with increment of casting speed 0.2 m•min-1. Fluctuation of temperature difference in meniscus is a main reason to deteriorate casting effectiveness as increasing casting speed.

2:45 PM

Numerical Simulation of Decarburization Reaction on the Surface of Liquid Iron: *Hyunjin Cho*[1]; Sangjoon Kim[1]; Haegeon Lee[1]; [1]POSTECH

Decarburization phenomena on the surface of liquid iron have been simulated by numerical method and the results were compared to the experimental data. During the DeC process in a converter, a large amount of splashed droplets are generated from the top surface of the bath and consequently the reaction surface area is increasing tremendously. Since the decarburization mainly occurs at the interface, it is important to understand the reactions on the liquid iron droplets.The flow and species concentration fields around liquid droplets taking into account the reaction of carbon with oxygen at the interface were calculated by a commercial CFD code FLUENT which solving multiphase Navier-Stokes equation with Volume Of Fluid method. The initial carbon concentration was fixed to 4 wt% and the concentrations of oxygen in the atmosphere were varied from 5 to 50 wt%. This numerical approach could allow predicting the decarburization rate of liquid iron.

3:00 PM

3D Simulation of the Melting during an Electro-Slag Remelting Process: *Abdellah Kharicha*[1]; [1]University of Leoben

The droplet formation during the melting of a 400 mm diameter electrode is simulated with a multiphase-MHD approach. The computational model includes a layer of slag and a layer of liquid steel. A VOF approach is used for the interface tracking, and a potential formulation is used for the electric and the magnetic field. The Lorentz force and the Joule heating is recalculated at each time step in function of the phase distribution. The first results provided by this model are presented.

3:15 PM Break

3:30 PM

An Analysis of Electromagnetic Field and Joule Heating of Electroslag Remelting Processes with Two Series-Connected Electrodes: Fang Wang[1]; *Baokuan Li*[1]; [1]Northeastern University

Comparing with single electrode in electroslag remelting processes, two series-connected electrodes have the advantage of small inductive, low power consumption and high efficiency, which was widely used to produce large scale steel. In the present work, a 3D finite element model was developed for the magnetic field, electromagnetic force, current density and Joule heating field of electroslag remelting processes (electrode, slag and ingot) with two series-connected electrodes using Maxwell equation, Lorentz law and Joule law. It was found through comparison that the modeling results is in accord with the experimental results, thus verifying the reliability of the model developed and relevant computation program. The results show that the current distribute on internal surfaces of electrode is smaller than on external surfaces; the maximum electromagnetic force is at the upper surface of slag nearby the electrode and the maximum joule heating is at the middle bottom of two electrodes.

3:45 PM

Modeling of Copper Converter Foamover and Operational Improvements: *Pengfu Tan*[1]; [1]Xstrata Copper

The foamed converter charges were up to 38 per month in Xstrata Copper Smelter at Mount Isa. The issue of high converter foamover frequency had remained unsolved for many decades in Mount Isa. A thermodynamic model of copper P-S converter and a viscosity model of converter slag have been developed to simulate the slag blow and copper blow, in order to understand the mechanism of converter foamover. The model predicts the chemistry and viscosity of slag during slag blow and copper blow. The effects of oxygen potential, charge of dirty reverts, slag carry-over, under-blowing, over-blowing, and temperature on the foamover and viscosity of slag in copper blow have been discussed. A theory has been developed to understand the mechanism of converter foamover. The practical solutions have also been developed and implemented in Mount Isa Copper Smelter. Some improvements of the industrial operations have been presented. After a series of improvements, the number of foamed charges per month in the converter operations has been decreased significantly from 35 in Aug 2008 to only one per month in 2009/2010.

4:00 PM

Modeling and Control of Copper Loss in Smelting Slag: Part I and Part II: *Pengfu Tan*[1]; [1]Xstrata Copper

Xstrata Copper Smelter at Mount Isa in Australia had experienced the technical issue of high copper losses in smelting slag (or RHF slag) before 2008. The copper losses in RHF slag was more than 3% in 2006 and 2007. The thermodynamic models, viscosity model have been developed to simulate the process control, slag chemistry, slag viscosity, and matt-slag separations. The theory of RHF KPIs has also been developed to reduce the copper losses in RHF slag. The RHF KPIs Theory has been applied in Mount Isa Copper Smelter. The copper losses in RHF slag have been dropped from 3.1% in 2007 to 0.76% in Apr 2009. The average copper loss in RHF slag in 2009 and 2010 is about 0.9%.

Microstructural Processes in Irradiated Materials: Microstructure Evolution: Experimental

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee

Program Organizers: Gary Was, University of Michigan; Thak Sang Byun, Oak Ridge National Laboratory; Shenyang Hu, Pacific Northwest National Laboratory; Dane Morgan, UW Madison; Yasuyoshi Nagai, Tohoku University

Monday PM
February 28, 2011

Room: 3
Location: San Diego Conv. Ctr

Session Chairs: Dane Morgan, University of Wisconsin; Thak Byun, Oak Ridge National Laboratory

2:00 PM Invited

In-Situ TEM Study on Elastic Interaction between a Prismatic Dislocation Loop and a Gliding Dislocation: *Yoshi Matsukawa*[1]; Grace Liu[1]; Ian Robertson[1]; [1]University of Illinois

Due to its glissile character the prismatic dislocation loop is expected to be either swept or arrested by the strain-field of gliding dislocations, and the latter is believed to be a key mechanism whereby locking of dislocation source occurs in neutron-irradiated metals. In this talk we show the first successful in-situ TEM observation capturing trap and de-trap processes

of the prismatic loop to/from a gliding dislocation. The critical capture distance measured on a micrograph was ~8 nm, which was smaller than the loop diameter (~10 nm). The gliding dislocation was originally a mixed dislocation; however, its line direction changed to a screw configuration upon interaction. The dislocation eventually bypassed the loop via cross-slip to a plane with a resolved shear stress ~40% smaller than that of the original slip plane. The prismatic loop was certainly an obstacle for dislocation-slip though a single loop was insufficient to completely lock the dislocation.

2:40 PM

In-Situ Investigation of Microstructure Evolution in NF616 and HCM12A Alloys under Heavy Ion Irradiation: *Cem Topbasi*[1]; Arthur Motta[1]; Mark Kirk[2]; [1]Pennsylvania State University; [2]Argonne National Laboratory

The NF616 and HCM12A ferritic-martensitic alloys are candidate cladding and duct materials for Generation IV nuclear energy systems. In-situ investigation of radiation damage was conducted for these alloys using the IVEM-Tandem at Argonne National Laboratory, which is a transmission electron microscope interfaced with ion accelerators allowing observing the development of irradiation damage as it occurs. The alloys were irradiated in situ to 15 dpa with 1 MeV Kr++ ions at temperatures between 50K and 673K. The number density and size distribution of irradiation induced defects were determined as a function of dose and temperature. Nano-sized defects (~1-2 nm) first appeared as white dots in dark-field TEM images between 0.5 to 1 dpa and later develop into resolvable loops. Dynamic observations show that black dots constantly appear and disappear under irradiation indicating a steady state of defect density. At high doses, complex defect superstructures including loop alignment were observed.

3:00 PM

Thermal Annealing Recovery Behavior of HT-9 Steel Irradiated to High Doses in FFTF: *Thak Sang Byun*[1]; William Lewis[1]; Stuart Maloy[2]; [1]Oak Ridge National Laboratory; [2]Los Alamos National Laboratory

The Fuel Cycle R&D Program is investigating methods of burning minor actinides in a transmutation fuel, for which the fast reactor core materials must be able to withstand very high doses (>200 dpa). To investigate operating strategies to reduce the buildup of radiation-induced defects, thermal annealing behavior has been studied for irradiated HT-9 steel. Samples were taken from representative locations of the ACO-3 duct of Fast Flux Test Facility: their irradiation doses were in the range of 3 – 148 dpa and irradiation temperatures in the range of 379 – 503 ºC. The samples were tested after each isochronal anneal for 1 hour at 300 – 650 ºC or after each isothermal anneal at 510 or 550 ºC. The radiation-induced hardening was nearly completely recovered by one-hour isochronal annealing above 550 ºC or by isothermal annealing at 510 ºC for about 20 hours or at 550 ºC for 2 hours.

3:20 PM

Microstructural Study of Pure Iron and Fe-Cr Model Alloys Ion Irradiated within the Jannus Platform (In- and Ex-Situ Mode): *Daniel Brimbal*[1]; Estelle Meslin[1]; Brigitte Décamps[2]; Jean Henry[1]; Alain Barbu[1]; [1]C.E.A. Saclay; [2]CNRS

Damage displacement cascades and helium production by transmutation reactions will result from the intense neutron irradiation in structural materials of future Fusion and GenIV reactors. In order to predict in-service properties of such materials, the microstructural evolution under irradiation of model materials (high purity iron and Fe-Cr alloys) has been studied using the JANNuS platform. Single- and dual-beam irradiations (Fe and Fe/He) have been performed ex-situ and in-situ (TEM) at high temperature. The TEM analysis leads to the following results: - In both materials irradiated ex-situ at 500°C up to 6 dpa, a coarse microstructure of large <100> dislocation loops, located in {100} planes, was formed. - Single-beam in-situ irradiations show a difference between iron and Fe-5at.%Cr : dislocation loops formed during irradiation display back-and-forth motion in iron but not in the alloy. They interact with dislocations in the alloy. - Dual-beam in-situ and ex-situ experiments are being analysed.

3:40 PM

Multiple-Beam Irradiation Effects in Ferritic Steels: *Naoyuki Hashimoto*[1]; Norihito Yamaguchi[1]; Hiroshi Oka; Hiroshi Kinoshita[1]; Somei Ohnuki[1]; [1]Hokkaido University

Reduced-activation ferritic/martensitic steel F82H has been developed as one of prime candidate materials for experimental fusion reactors. To estimate irradiation effects in fusion reactor components, multiple-scale modeling has been studied. Modeling activities for irradiation induced microstructural change is quite effective to enhance the capability to predict mechanical properties of the materials during irradiation. Defect activation energies such as vacancy and interstitial migration energies should be estimated to obtain fundamental parameters for the modeling. And also, some of the key issues are the effects of helium and hydrogen on the microstructure evolutions such as swelling, and on the mechanical properties such as fracture toughness or embrittlement. In this study, TEM specimens of F82H, Fe-8Cr model alloy, and Pure Fe have been irradiated by electron and helium ion beams using a High Voltage Electron Microscope (HVEM) as the experimental evaluation of the modeling and simulations.

4:00 PM Break

4:20 PM

The Effects of Mn on Microstructure and Hardness in A533B and Model Alloys: *Hideo Watanabe*[1]; Naoaki Yoshida[1]; [1]RIAM, Kyushu University

It is well known that radiation hardening and embrittlement of A533B steels are very sensitive to minor solutes (namely, P, Cu, Ni, Si etc.) containing in the steels. To investigate the effect of these solutes on microstructural evolution and hardness changes due to irradiation, A533B steels and some model alloys were irradiated by neutron, Fe ion and electron at 290°C. After the irradiation, the microstructure of the specimens was observed by transmission electron microcopy. The recovery behavior of the hardness by post-irradiation annealing at 450°C was related with the disappearance of these dislocation loops. The study revealed that the formation of interstitial type dislocation loops, which can be seen by conventional weak-beam technique, was essential for irradiation hardening of these samples. In this study, microstructure and hardness of these samples were compared with the results of Fe ion and electron irradiation at room temperature and 290°C.

4:40 PM

Microstructural Changes by Thermal Aging and Neutron Irradiation in Stainless Steel Weld Overlay Cladding of Nuclear Reactor Pressure Vessels: *Tomoaki Takeuchi*[1]; Jun Kameda[2]; Yasuyoshi Nagai[3]; Takeshi Toyama[3]; Yutaka Nishiyama[1]; Kunio Onizawa[1]; [1]Japan Atomic Energy Agency; [2]National Institute for Materials Science; [3]The Oarai Center, Institute for Materials Research, Tohoku University

Microstructural changes in thermally aged and neutron-irradiated stainless steel weld overlay cladding applied to the inner surface of nuclear reactor pressure vessels were investigated using three-dimensional atom probe (3DAP). The cladding material was composed mainly of austenitic phase and partly of ferritic phase. In the as-received cladding material, a slight fluctuation of Cr concentration probably due to spinodal decomposition was observed only in the ferritic phase. Moreover in the ferritic phase, thermal aging at 400°C for 10,000 h caused the precipitation of G phase with chemical composition of Ni:Si:Mn = 16:7:6 as well as increased spinodal decomposition. Significant hardening of the ferritic phase was induced by the thermal aging. A correlation between hardening and the microstructural changes will be discussed. The neutron irradiation effects will be also shown in this presentation.

5:00 PM

Dependence of Radiation Induced Segregation on Grain Boundary Structure in a 9 wt.% Cr Model Ferritic/Martensitic Steel: *Kevin Field*[1]; James Bentley[2]; Chad Parish[2]; Jeremy Busby[2]; Todd Allen[1]; [1]University of Wisconsin - Madison; [2]Oak Ridge National Laboratory

Ferritic/Martensitic (F/M) steels containing 9 wt.% Cr, are candidate materials for structural components in the next generation of advanced nuclear and fusion reactors. Although it is known that these alloys exhibit

radiation induced segregation (RIS) at grain boundaries (GBs) while in-service, little is known about the mechanism behind RIS in F/M steels. Current understanding of RIS in F/M steels presents a model of point defect migration to GBs, which are considered perfect sinks. Changes in grain boundary character may influence the sink nature and migration processes. Proton irradiated 9 wt.% Cr model steel was investigated using STEM/EDS spectrum imaging and GB misorientation analysis to determine the role of GB structure on RIS at different GBs. This preliminary investigation found Cr preferentially segregates to specific GB structures. The preferential segregation to specific GB structures suggests a need to alter the understanding of how point defects interact at GBs in F/M steels.

5:20 PM

Kinetic Lattice Monte Carlo Simulations of Radiation Induced Segregation of Chromium in Ferritic-Martensitic Steels: *Brian Frisbie*[1]; Brian Wirth[1]; [1]University of California at Berkeley

Ferritic-martensitic steels with 8-12% Cr are leading candidates for advanced nuclear applications because of their superb thermal properties and swelling resistance. Irradiation of these steels introduces microstructural and microchemical changes in the material that can lead to degradation of their structural properties through processes such as radiation-induced embrittlement. In this work, we present a kinetic lattice Monte Carlo model intended to simulate the segregation behavior of Cr to grain boundaries in these ferritic-martensitic steels while under irradiation. The simulations confirm that the enrichment or depletion of Cr at grain boundaries is dependent on its diffusivity relative to that of Fe. While variations in input parameters including the temperature and local energy barriers influence the direction and rate of Cr enrichment, all simulation results display a tendency towards saturation in Cr content at the grain boundaries after a sufficiently high dose.

5:40 PM

Application of Modified Inverse Kirkendall Model of Radiation-Induced Segregation to Ferritic-Martensitic Alloys: *Janelle Wharry*[1]; Zhijie Jiao[1]; Gary Was[1]; [1]University of Michigan

Ferritic-Martensitic (F-M) alloys are leading candidates for fast reactor cladding and structural materials. This study is a combined experiment-modeling effort to understand radiation-induced segregation (RIS) of Cr in these alloys. Alloys T91 (9wt%Cr), HT9 (12wt%Cr), and HCM12A (11wt%Cr) were irradiated with 2.0 MeV protons at 400°C to 3, 7, and 10 dpa, and with 5.0 MeV Fe2+ ions to 100 dpa at an equivalent temperature. Prior austenite grain boundary (PAGB) compositions were measured with scanning transmission electron microscopy using energy-dispersive X-ray spectroscopy (STEM/EDS). Results show Cr depletion at PAGBs in HT9 and HCM12A, and enrichment in T91. The modified inverse Kirkendall (MIK) model, which has successfully predicted RIS in austenitic alloys, was modified for the Fe-Cr system and used to predict the direction and magnitude of RIS in F-M alloys. Model results are compared to experimental results and are discussed in the context of the RIS mechanism in F-M alloys.

6:00 PM

Ab Initio-Based Rate Theory Modeling of Radiation Induced Segregation in Ni-Cr Alloys: *Leland Barnard*[1]; Samrat Choudhury[2]; Dane Morgan[1]; [1]University of Wisconsin-Madison; [2]Los Alamos National Lab

The fcc Ni-Cr alloy system forms the basis of an important class of structural materials in the nuclear power industry, both in existing reactors and for future reactor designs. Point defect diffusion under irradiation in this alloy results in a phenomenon known as radiation induced segregation (RIS), whereby Cr is depleted near grain boundaries, voids, and surfaces. Cr is essential to the corrosion resistance of these alloys, and depletion near these critical microstructural features potentially results in a vulnerability to corrosion damage. In this study, we construct a rate theory model to simulate RIS in Ni-Cr alloys parameterized entirely with data obtained through ab initio calculations. Our results indicate that details pertaining to the fate of radiation produced defects that are neglected in conventional RIS modeling, such as defect sink bias, production bias, and energetic defect trapping, may play a critical role in the evolution of Cr RIS.

Neutron and X-Ray Studies of Advanced Materials IV Symposium: Brent Fultz Honorary Session I: Joint Session with Computational Thermodynamics and Kinetics Symposium

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Xun-Li Wang, Oak Ridge National Laboratory; Jaimie Tiley, Air Force Research Laboratory; Peter Liaw, The University of Tennessee; Erica Lilleodden, GKSS Research Center; Brent Fultz, California Institute of Technology; Y-D Wang, Northeastern University

Monday PM — Room: 9
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Xun-Li Wang, Oak Ridge National Laboratory; Michael Manley, Lawrence Livermore National Laboratory

See page 133 for program.

Pb-Free Solders and Other Materials for Emerging Interconnect and Packaging Technologies: Whisker Growth

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

Program Organizers: Indranath Dutta, Washington State University; Darrel Frear, Freescale Semiconductor; Sung Kang, IBM; Eric Cotts, SUNY Binghamton; Laura Turbini, Research in Motion; Rajen Sidhu, Intel Corporation; John Osenbach, LSI Corporation; Albert Wu, National Central Univ, Taiwan; Tae-Kyu Lee, Cisco Systems

Monday PM — Room: 7B
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Eric Cotts, Binghamton University; Peter Borgesen, Binghamton University

2:00 PM Keynote

The Metallurgy of Sn Whisker and Hillock Growth: *William Boettinger*[1]; [1]National Institute of Standards and Technology

Pb-free pre-tinned electronic components are subject to the spontaneous growth of Sn whiskers at room temperature. These whiskers are known to have caused shorts in various high reliability circuits. The metallurgical factors affecting whisker growth will be described. The columnar grain structure of Sn deposits combined with biaxial in-plane compressive stress cause either whiskers or hillocks to grow due to a grain boundary creep-type mechanism. Hillocks appear to grow when the grain boundaries of the Sn in the substrate are mobile whereas whiskers grow when the Sn grain boundaries in the substrate are pinned. The biaxial stress is due to the plating process as well as the growth of Cu_6Sn_5 intermetallic particles. Remaining questions related to the location of nucleation sites for hillocks and whiskers as well as the details of the stress generation mechanisms will be discussed.

2:40 PM Invited

Kinetic Mechanisms Controlling IMC Growth, Stress Evolution and Whisker Formation: *Eric Chason*[1]; Nitin Jadhav[1]; Eric Buchovecky[1]; Fei Pei[1]; Allan Bower[1]; Sharvan Kumar[1]; [1]Brown University

Understanding the mechanisms controlling tin whisker formation is necessary for developing reliable mitigation strategies and for developing predictive models. Although stress is widely accepted as the driving force

for whisker growth, there are still many questions about how the stress is generated, what controls its evolution and how it leads to whisker formation. To address these issues, we present results from real-time studies that explore the correlation between the kinetics of IMC growth, stress evolution and whisker nucleation in films with different microstructures and layer morphologies. Comparisons among different measurements (e.g. varying layer structures, grain sizes, surface oxide layers and Pb content) help identify the different kinetic processes that play a role in their evolution. These mechanisms are used to develop a finite element analysis (FEA) model combining IMC growth, plastic deformation and grain boundary diffusion to simulate how the stress evolves and how it leads to whisker growth.

3:05 PM Invited

Role of Stress and Oxidation on Metallic Whisker Growth: *Elizabeth Hoffman*[1]; Yong Sun[2]; Poh-Sang Lam[1]; Xiaodong Li[2]; [1]Savannah River National Laboratory; [2]University of South Carolina

Preventing metallic whisker growth leading to electronic failures is particularly important in microelectronic circuits where devices are separated by spacings as small as tens of nanometers. Historically, lead has been added to solders to suppress whisker growth and to improve the manufacturing processes. With the understanding of lead's harmful effects on the environments and human health, this is no longer an acceptable solution. It is generally acknowledged that internal stresses arising in low melting point metals, such as Sn, promote whisker growth based on a creep-like process. Compressive stress resulting from metal oxidation is one of the theories that have been suggested to play a role in the growth of whiskers. To investigate the influence of oxidation on whisker formation and growth, a series of experiments are underway to correlate the oxygen uptake, the measured internal stress, and the whisker density that forms on specimens with particular thin film/substrate combinations.

3:30 PM

Evidence of Plastic Deformation Adjacent to Sn Whiskers: *John Osenbach*[1]; Robert Hilty[2]; [1]LSI Corporation; [2]Tyco

In this paper the results of indentation induced plastic deformation and Sn whisker growth is reported. Sn plating on a number of different substrate materials were subjected to indentation stresses. Post indentation, the microstructure and presence or absence of whiskers was evaluated using scanning and transmission electron microcopy. In all cases, independent of substrate alloy, plastic deformation as observed as evidenced by grain elongation and compression with no whiskers in the indentation region. Adjacent to the plastically deformed region whiskers are observed. Whiskers were typically found within 500um of the plastically deformed region. We interpret the results as being consistent with a theory that one of the authors has recently published "Creep and It's Affect on Sn Whisker Growth Whisker Formation".

3:50 PM Break

4:00 PM

Whisker Formation Induced by Component and Assembly Ionic Contamination: *Polina Snugovsky*[1]; Stephan Meschter[2]; Zohreh Bagheri[1]; Eva Kosiba[1]; Marianne Romanksy[1]; [1]Celestica; [2]BAE Systems

The paper will describe the results of an intensive whisker formation study on Pb free assemblies with different level of cleanliness. Thirteen types of as-received surface mount (SMT) and pin-through-hole (PTH) components were cleaned and intentionally contaminated with solutions containing chloride, sulfate, bromide, and nitrate. Then the parts were assembled on double sided boards that were also cleaned or intentionally contaminated with three fluxes having different halide contents – ROL0, ROL1, and ORH1. The assemblies were subjected to high temperature/high humidity testing (85°C/85%RH). Periodic examination found that contamination triggers whisker formation on both exposed tin and solder fillets. Whisker occurrence, morphology, and length parameters depending on the type and level of contamination will be discussed. Sequential cross-sections were used to assess the metallurgical aspects of whisker formation and the microstructual changes occurring during corrosion.

4:20 PM

Microstructure Formation and Whisker Growth in SAC105 Solder Joints with Rare Earth Elements: *Polina Snugovsky*[1]; Zohreh Bagheri[1]; Stephan Meschter[2]; Leonid Snugovsky[3]; John Rutter[3]; Doug Perovic[3]; [1]Celestica; [2]BAE Systems; [3]University of Toronto

Rare earth elements (REE) such as Y, Ce, and La being added to Sn-Ag-Cu alloys may improve mechanical properties of solder joints. It was shown in literature that the addition of 0.2% REE refines the microstructure positively effecting drop/shock behavior. Unfortunately, it has also been demonstrated that Sn-Ag-Cu solders with REE are prone to whisker formation. In the present work, microstructure of SAC105 solder with 0.1 – 0.4 % Y, Ce, or La reflowed on the Cu substrate, is examined. The results of high temperature/high humidity testing that may trigger whisker formation is discussed. The similarities and differences in microstructure and whisker growth of SAC105 solder joints with different REE is examined from a metallurgical perspective using sequential microsectioning. The influence of flux type and cleanness on propensity of whiskering is discussed.

4:40 PM

Tin Whisker Growth on the Surface of Sn-3.8Ag-0.7Cu-RE Solder Alloys: *Hu Hao*[1]; Fu Guo[1]; Yonglun Song[1]; Guangchen Xu[1]; Yaowu Shi[1]; [1]Beijing University of Technology

Alloying approach by adding trace amount of rare earth (RE) elements in Sn-3.8Ag-0.7Cu solder alloy has been widely reported to show its ability on the improvement of processing and reliability properties. However, bulk RE-Sn phases, when adding excessive RE elements, can be observed inside the solder matrix. This study systematically investigated the tin whiskers growth (including the incubation time, growth rate, quantity, morphology, size, etc.) on the surface of CeSn3, LaSn3, (La0.4Ce0.6)Sn3, ErSn3, and YSn3 by adding excessive Ce, La, La/Ce mixture, Er and Y, respectively, in the Sn-3.8Ag-0.7Cu solder. Most important, the finding of tin whiskers with a non-constant cross-section provided a better understanding to the spontaneous growth equation of tin whiskers. In addition, a hypothesis of "double stress zones" was proposed to demonstrate the underlying mechanism for tin whiskers growth on the bulk RE-Sn phases.

5:00 PM

Effect of Sn Layer on Whisker Growth Kinetics: Modifying the Surface, Microstructure, Composition and Heat Treatment: *Fei Pei*[1]; Gordon Barr[2]; Eric Chason[1]; Nitin Jadhav[1]; [1]Brown University; [2]EMC Corporation

Though it is well understood that compressive stress due to intermetallic (IMC) formation is the main driving force for whisker formation, the underlying mechanisms that control the stress distribution and cause whiskering are not clear. We present the results of studies done to understand how modification of the Sn layer (surface treatment, thickness, grain size, Pb-alloying, heat treatment) affects the growth and morphology of the IMC and the corresponding stress development in the Sn layers. We have quantified the whisker nucleation kinetics using an optical method so that we can relate the whisker growth to the underlying stress evolution. Changes in the Sn surface and bulk alter the mechanical properties and stress relaxation behavior and thus result in various whisker growth rates. The study also shows that a grain with a weaker oxide layer will not necessarily whisker, indicating that the underlying Sn microstrucutre plays a crucial role in whiskering.

Physical and Mechanical Metallurgy of Shape Memory Alloys for Actuator Applications: Characterization of Shape Memory Alloys: Structure-Property Relationships

Sponsored by: The Minerals, Metals and Materials Society
Program Organizers: S. Raj, NASA Glenn Research Center; Raj Vaidyanathan, University of Central Florida; Ibrahim Karaman, Texas A&M University; Ronald Noebe, NASA Glenn Research Center; Frederick Calkins, The Boeing Company; Shuichi Miyazaki, Institute of Materials Science, University of Tsukuba

Monday PM — Room: 11B
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Shuichi Miyazaki, University of Tsukuba; Huseyin Sehitoglu, University of Illinois

2:00 PM Invited
Structure and Thermomechanical Behavior of NiTiPt Shape Memory Alloy Wires: *Ken Gall*[1]; [1]Georgia Tech

The objective of this work is to understand the structure property relationships in polycrystalline NiTiPt (Ti 42.7 at.% Ni 7.5 at %Pt) with a composition showing pseudoelasticity at ambient temperatures. Structural characterization of the alloy includes grain size determination and textureanalysis while the thermomechanical properties are explored using tensile testing. Variation in heat treatment is used as a vehicle to modify microstructure. The results are compared to experiments on Ni-rich NiTi alloy wires (Ti¬51.0 at.% Ni), which are in commercial use in various biomedical applications. With regards to microstructure, both alloys exhibit a <111> fiber texture along the wire drawing axis; however, the NiTiPt alloy grain size is smaller than that of the Ni-rich NiTi wires, while the latter materials contain second-phase precipitates.

2:20 PM
Training and the Two-Way Shape Memory Effect in TiNiPd and TiNiPdX High Temperature Shape Memory Alloys: *Kadri Atli*[1]; Brian Franco[1]; Ibrahim Karaman[1]; Ronald Noebe[2]; [1]Texas A&M University; [2]NASA Glenn Research Center

The relationship between thermomechanical cycling (training) and the two way shape memory effect (TWSME) was investigated for a number of shape memory alloy (SMA) systems including equiatomic TiNi, a ternary Ti50.5Ni24.5Pd25 high temperature SMA (HTSMA), and its quaternaries TiNiPd25X (X = Sc, Ta replacing Ti). Training consisted of thermal cycling under various constant stress levels and stable and reproducible TWSME behavior was obtained for all materials after 100 thermal cycles. It was shown that training stress dictated the level of TWSME strain and thermal hysteresis behavior, which were in turn significantly affected by the strength of the material and the compatibility of the transforming phases. The work output capability of the TWSME was evaluated by constraining the "cold shape" under external stress-free conditions and heating above the transformation temperature. Improvements in TWSME strains and thermal hysteresis were found after microalloying TiNiPd25with Sc and Ta.

2:35 PM
Thermomechanical Testing of NiTiPdPt High Temperature Shape Memory Alloy Springs: *Douglas Nicholson*[1]; Santo Padula[2]; Ronald Noebe[2]; Raj Vaidyanathan[1]; [1]UCF; [2]NASA GRC

Stroke in high temperature shape memory alloys (HTSMAs) can be limited in cases where stability and repeatability at elevated temperatures are required. However, these alloys can still be used in actuator applications that require large strokes, by fabricating them in the form of springs. Thus there is value in evaluating the thermomechanical performance of HTSMA helical actuators. Towards that goal, a test setup was designed to acquire stroke, moment, and temperature data during thermomechanical cycling of NiTiPdPt springs under load. The role of grip constraints was also systematically investigated, i.e., by allowing the springs to freely rotate during actuation or fixing the ends of the springs. The experiments assessed the contributions of material behavior (i.e., microstructural evolution) and geometry (i.e., helical angle) to overall actuator performance. With appropriate assumptions, the mechanics of the spring behavior was approximately captured using spring theory and finite element analysis.

2:50 PM
Structure-Property Relationships in a Ni-Rich Ni-25Pd-48Ti (at.%) High Temperature Shape Memory Alloy: *Taisuki Sasaki*[1]; B. C. Hornbuckle[1]; Glen Bigelow[2]; Ronald Noebe[2]; Mark Weaver[1]; Gregory Thompson[1]; [1]University of Alabama; [2]NASA Glenn Research Center

Ni-rich NiTi alloys, when properly processed, display relatively stable shape memory and superelastic behavior. However, their potential applications are severely limited because of low transformation temperatures. Recent efforts have focused on using ternary additions to raise the transformation temperatures, while simultaneously improving stability through nanoscale precipitation. In this work, we have investigated the effect of aging on the structure and shape memory behavior of an extruded Ni-48Ti-25Pd (at.%) alloy. After aging at 400°C, nanoscale precipitates formed within the matrix, resulting in an increase in transformation temperatures. Electron diffraction studies and atom probe analysis showed that the structure and the chemistry of the precipitate are similar to the P-phase seen in NiTiPt alloys. The mechanical responses of this aged alloy were tested via superelastic and load biased thermomechanical testing, demonstrating the stabilizing effect of the fine precipitate phase.

3:05 PM
Structure-Property Relationships for Ni-Ti-Pt High Temperature Shape Memory Alloys: *Grant Hudish*[1]; Glen Bigelow[2]; Ronald Noebe[2]; Michael Kaufman[1]; [1]Colorado School of Mines; [2]NASA-GRC

Alloys of Ni and Ti in near equal amounts are commercially prevalent shape memory alloys (SMA's), but are limited to use near room temperature. Increasing the transformation temperature of traditional Ni-Ti SMAs, through ternary alloying of Pd, Pt, Au, Hf or Zr, would allow for their use in various higher temperature applications and industries including aerospace, automotive, and down hole energy exploration, to name a few. Pt is currently one of the most promising ternary additions for stable and predictable high temperature SMA's (HTSMA's), but little is understood about the effects of Pt on the microstructure and mechanical properties of Ni-Ti alloys. Using various techniques including scanning and transmission electron microscopy (SEM and TEM), differential thermal analysis (DTA), and load-biased thermally cycled compression tests, the current research explores the link between alloy microstructures and the shape memory properties of several Ni-Ti-Pt alloys.

3:20 PM Break

3:30 PM Invited
A New Precipitate Phase in High-Temperature TiNiPt and Its Effect on Shape Memory Properties: F. Yang[1]; L. Kovarik[1]; Y. Wang[1]; P. M. Anderson[1]; A. Garg[2]; R.D. Noebe[3]; *Michael Mills*[1]; [1]The Ohio State University; [2]University of Toledo - and - NASA Glenn Research Center; [3]NASA Glenn Research Center

The effects of aging on mechanical and shape memory properties of the high-temperature shape memory alloy $Ti_{50}Ni_{30}Pt_{20}$(at.%) were investigated in order to understand aging effects on transformation strain, work output and dimensional stability under cyclic load biased tests. Aging results in precipitation of a previously unidentified phase. The precipitate phase has been analyzed with electron diffraction and high-resolution STEM HAADF imaging. The precipitates are closely related to the high temperature cubic B2 phase, but have a unique structure due to their non-periodic character along one of the primary crystallographic directions. The importance of these precipitates in other high temperature shape memory alloys and their role in achieving optimal shape memory properties will be discussed.

3:50 PM

Grain Size Distribution Effects on Phase Transformation Behavior of NiTi Thin Films: *Xu Huang*[1]; David Wu[2]; Ainissa Ramirez[1]; [1]Yale University; [2]Institute of High Performance Computing

This presentation demonstrates the role of grain size and its distribution on phase transformations in sputter-deposited NiTi thin films. Two-step heat treatments—where growth and nucleation are both active in the first step, and nucleation is suspended in the second—narrow the grain size distribution, as determined by transmission electron microscopy. The associated transformation temperatures and actuation properties departed significantly from the Hall-Petch-like relationship of conventionally (one-step) annealed films. A change in texture was also apparent with two-step heat treatments. This presentation demonstrates a method for tailoring microstructures and illuminates the role of grain size distributions on properties.

4:05 PM

Shape Memory Characteristics of Time Gradient Annealing Treated Ti-50.9 at.% Ni Alloy: *Jae Il Kim*[1]; Su Ho Park[1]; Jun Hee Lee[1]; Yun-Jung Lee[2]; Tae-Hyun Nam[3]; [1]Dong-A University; [2]Gyungbook National University; [3]Gyungsang National University

In this study shape memory characteristics and superelasticity of a time gradient annealing treated Ti-50.9at%Ni alloy have been investigated by means of differential scanning calorimetry(DSC), thermal cycling tests under constant load and tensile tests. This method is an anneal within a time gradient after cold work, thus creating a structural gradient within the matrix of the alloy. By annealing 30% cold worked alloy under the time gradient from 3min to 20min at 773 K, 44K variation in R phase transformation interval (Rs - Rf) were obtained along the length of sample (80mm). Temperature dependence of R-phase transformation elongation (de/dT) of time gradient annealing treated sample at 773 K was 0.0068%/K. Also, it was found that the time gradient-anneal resulted in varying thermal transformation behavior along its length and a unique Lüders-type deformation behavior with a positive stress gradient. Such behavior provides improved controllability for actuation applications.

4:20 PM

Crystal Structures, Phase Transformations and Microstructures of Rapidly Solidified Heusler Type CoNiGa(Al) Ferromagnetic Shape Memory Alloys for Micro-Actuator Applications: *Shampa Aich*[1]; M. Vijaykumar[2]; I. Al-Omari[3]; M. Chakraborty[4]; D. Sellmayar[3]; [1]Indian Institute of Technology; [2]JSW Steel Ltd.; [3]University of Nebraska-Lincoln; [4]Indian Institute of Technology - Bhubaneswar

A series of arc-melted ingots of Heusler type $Co_{50}Ni_{25}Ga_{25-2x}Al_{2x}$ (x = 0, 1, 2, 3, and 4) alloys were rapidly solidified by melt-spinning onto a copper wheel with a linear velocity of 25 m/s. The melt-spun ribbons were heat-treated at 800 ºC-1200 ºC for 4 hours followed by water quenching. The occurrence of $L1_2$ ordered structure and disordered □-phase has been confirmed by XRD analysis in the as-spun and annealed alloys. The martensitic transformation temperature (T_m) increases with annealing temperature and Al addition in all alloys with deterioration of ductility due to replacement of Ga with Al. The microstructural observations performed by HRTEM and FESEM reveal the martensitic twin bands at higher concentration of Ga in the as-spun as well as in heat-treated ribbons. High T_m (~ 390 K-440 K) and high Curie temperature T_C (~ 400K) make those alloys good enough to be used as Ferromagnetic Shape Memory Alloys.

4:35 PM

Studies on Effect of Aging on Shape Memory Characteristics of Cu-Al-Fe Alloys: *Sampath Vedamanickam*[1]; Raju T.N.[1]; [1]Indian Institute of Technology Madras

The most popular SMAs based on Ni-Ti cannot be used in service at T>100°C. In addition, they are difficult to produce and process and are also expensive. There is therefore a need for producing SMAs for high temperature actuator applications. With this as the background and impetus, Cu-Al-Fe shape memory alloys in the range of 10-12 wt% of Al and 0-2 wt % of Fe, exhibiting β-phase at high temperatures and manifesting shape memory effect upon quenching to lower temperatures, were prepared through ingot metallurgy . The alloys were aged at 300°C and 500°C, respectively. The effect of ageing on the transformation temperatures and shape memory properties has been studied. The alloys aged at 300°C show an increase in the transformation temperatures. The results are presented and discussed in the paper.

4:50 PM

Stoichiometry and Aging Effects on the Microstructure and Properties of NiTiHf Shape Memory Alloys: *Daniel Coughlin*[1]; Ronald Noebe[2]; Glen Bigelow[2]; Anita Garg[2]; Michael Mills[1]; [1]Ohio State University; [2]NASA Glenn Research Center

Certain alloys belonging to the NiTiHf ternary system exhibit attractive high temperature shape memory properties, including relatively high transformation temperature, moderate transformation strain, and very small irrecoverable strain during load-biased, thermal-cycling tests at moderate to high stresses. Alloy compositions that span "stoichiometry", consisting of 20at.% Hf and varying Ti concentrations (from 29 to 30.5at.%) have been analyzed using SEM and TEM in the as-extruded condition. The phase constituents will be reported as a function of composition. The effect of aging on the Ni-29.7Ti-20Hf composition has also been explored using isothermal, constant strain rate testing above the austenite finish temperature. The results indicate that significant strengthening occurs upon aging due to the formation of a distribution of fine precipitates. The structure of these precipitates and their interaction with dislocations in the B2 phase will be discussed with relationship to the desirable shape memory properties.

5:05 PM

Magnetic Shape Memory Effect in NiMnCo-Based Meta-Magnetic Shape Memory Alloy Micropillars: *Nevin Ozdemir*[1]; Ibrahim Karaman[1]; Nathan Mara[2]; Douglas Pete[3]; [1]Texas A&M University; [2]Los Alamos National Laboratory; [3]Sandia National Laboratories

Magnetic Shape Memory Alloys (MSMAs) have recently emerged as an alternative to conventional Shape Memory Alloys (SMAs) due to their faster response, which is a consequence of the magnetic field-induced reversible shape change. NiMnCo-based SMAs show great promise for functional materials applications due to an order of magnitude higher actuation work output than other active materials and their potential for use in MEMS/NEMS devices. To the best of our knowledge, there is no work on the magnetic field-induced shape change via field-induced phase transformation on sub-micron structural length scales. Here, we studied the magnetic field-induced martensitic transformation on NiMnCoIn MSMAs at this size scale. The pillars were obtained from NiMnCoIn single- and polycrystals and deformed to various strain levels. Magnetic field was applied to recover the deformation via field-induced reverse martensitic transformation. The recovery strain levels and phase transformation characteristics were determined as a function of pillar size.

5:20 PM

In-Situ Phase Transformation Behavior of Ni-Rich NiTi Shape Memory Alloys: *Suresh Santharam*[1]; Dong-Ik Kim[2]; Subir Kumar Bhaumik[3]; Satyam Suwas[1]; [1]Indian Institute of Science; [2]Korea Institute of Science and Technology; [3]National Aerospace Laboratory (CSIR)

Ni-rich NiTi Shape memory alloy, characterized by room temperature austenite (B2) phase, was deformed in-situ in a Scanning Electron Microscope. The characteristic crystallographic features pertaining to B2 (austenite) to B19' (martensite) transformation were examined using Electron Back Scattered Diffraction. From the results, it has been shown that during stress induced transformation, different martensite variants appear within a single austenite grain. The Schmid factor calculations for different martensite variants indicate that in addition to the variants possessing the highest Schmid factor, certain variants with lower Schmid factor also get activated during stress induced martensitic transformation. The authors attribute the activation of different martensite variants with lower Schmid factor values as semi-coherent austenite-martensite interface. This proposition has been substantiated through transmission electron microscopic study.

5:35 PM End of Session

Polycrystal Modelling with Experimental Integration: A Symposium Honoring Carlos Tome: Neutron Diffraction and Prediction of Internal Stresses

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, ASM-MSCTS: Texture and Anisotropy Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Ricardo Lebensohn, Los Alamos National Laboratory; Sean Agnew, University of Virginia; Mark Daymond, Queens's University

Monday PM — Room: 6C
February 28, 2011 — Location: San Diego Conv. Ctr

Session Chairs: Mark Daymond, Queen's University; Bjoern Clausen, Los Alamos National Laboratory; Donald Brown, Los Alamos National Laboratory

2:00 PM Invited
Information Volumes in Diffraction Techniques for Strain Analysis: *I Noyan*[1]; [1]Columbia University

In this presentation we discuss how the modality (dynamical or kinematic) of scattering determines the volumes from which the information is obtained. In addition, this modality also impacts the uniqueness of the stress state measured. We use rigorous diffraction modeling to show that diffraction data obtained from weakly deformed crystals that scatter in the dynamical limit should not be used for validating continuum models. The "mean" strain obtained from polycrystalline continua and how this compares to the orientation-specific strain and mean-strain computed by mean-field models of deformation will also be discussed.

2:25 PM Invited
EPSC Modeling and Neutron Diffraction Measurements: *Bjørn Clausen*[1]; Donald Brown[1]; Levente Balogh[1]; Carlos Tomé[1]; [1]Los Alamos National Laboratory

Dr. Carlos Tomé has been one of the major contributors in developing and implementing the Elasto-Plastic Self-Consistent (EPSC) model throughout the years. Although the EPSC scheme was introduced in the 60's and 70's, the first publically released code implementing the EPSC model with full elastic and plastic anisotropy on both grain and polycrystal level originated in the 1994 Turner and Tomé paper. From the start, the predictions of the EPSC model were correlated with neutron diffraction measurements of internal strains and texture. In the following years there were many publications showing how internal strain measurements using diffraction can be used to verify the predictions of the EPSC model, and vice versa, how the EPSC model predictions can be used to interpret and deconvolute measured diffraction data. This symbiotic relationship between diffraction measurements and the EPSC model and how it has influenced the development of both techniques will be discussed.

2:50 PM Invited
Texture and Type-2 Strains in Rolled Zircaloy-2 and Their Relevance to In-Reactor Growth: *Thomas Holden*[1]; [1]Northern Stress Technologies

Zr-2 calandria tubes in CANDU reactors can grow by 0.1% in a fast neutron fluence thus creating an engineering problem. Neutron diffraction was used to measure the strain state of Zr-2 rolled plate, an analog for the tubes, in the late 1980's. The type-2 strains plus the texture determine the growth behaviour. The strong texture permits a practical estimate of the strain tensor of the material. With the aid of the elasto-plastic self-consistent model (EPSC), developed by C.N Tomé and his collaborators, one can compare experiment and theory for the strains in as-prepared or tensioned Zr-2 and incorporate this into the in-reactor growth model as set out by C. H. Woo. Carlos Tomé's contributions to the ever-improving EPSC, as well as the problem of in-reactor growth, and his enthusiastic encouragement of experimentalists to test the polycrystal models are the mark of an outstanding scientist. His friendship is highly valued.

3:15 PM
Internal Stresses and Microstructure by Neutron Diffraction Profile Analysis: Comparison with Other Techniques: *Vadim Davydov*[1]; Petr Lukáš[2]; Helena Van Swygenhoven[1]; Martin Petrenec[3]; Ondrej Man[4]; Pavel Strunz[2]; Radomír Kužel[5]; [1]Paul Scherrer Institut; [2]Nuclear Physics Institute; [3]Institute of Physics of Materials; [4]Brno University of Technology; [5]Charles University

The neutron and X-ray diffraction techniques combined with electron microscopy methods has been used to study the internal microstresses and micorstructure in low carbon steel. Since the real structure features affect the width and shape of the neutron or X-ray diffraction pattern, and, as these real structure aspects are very numerous and mutually superposed, diffraction effects in terms of real structure characteristics are not simple to interpret. Using the recently modified single-line profile analysis method, the dislocation content was addressed and compared in terms of dislocation density parameter with the results obtained by other microscopy methods. The single-line neutron diffraction profile analysis performed at NPI was extended using full diffraction patterns collected in time-of-flight mode on POLDI.

3:35 PM Break

3:50 PM Invited
On the Evolution of Crystal Stresses during the Elastic-Plastic Transition in FCC Polycrystals: *Paul Dawson*[1]; Su Leen Wong[1]; [1]Cornell University

Simulations based on crystal-scale elastoplasticity have proven to be a valuable tool for interpreting data measured by diffraction techniques. Diffraction measurements provide lattice strain data on subsets of the grains within the diffraction volume and offer insight into grain interactions. With finite element models the spatial arrangement of grains is included and interactions among the grains arise naturally in simulating an experiment. We discuss the roles of elastic and plastic anisotropies on grain interactions with attention to changes in the crystal stress distributions as the loading progresses through the elastic plastic transition. The simulations offer an explanation for the differences in lattice strain behaviors at the onset of yielding observed between metals of low and high elastic anisotropy under monotonic loading. We will show the implications of elastic and plastic anisotropies, as quantified by the directional strength-to-stiffness, on lattice strainsduring cyclic loading.

4:15 PM Invited
High Pressure Plastic Properties of Hcp Metals: Experiments and Elasto-Plastic Models: *Sebastien Merkel*[1]; [1]Universite Lille 1

Over the last few years, we developed new techniques for the study of plastic properties of materials under high pressure and temperature. Their physical properties are analyzed in-situ using x-ray diffraction to extract quantitative texture information and average elastic strains within the sample. Thanks to the contribution of Carlos Tomé, we now use EPSC models to relate the experimental observations and the plastic properties of the sample.In this presentation, I will show results on the hcp phase of Co deformed at 300 K between 0 and 42 GPa and results on the hcp phase of Fe deformed at pressures and temperatures reaching 19 GPa and 600 K. I will highlight how the combination of x-ray diffraction and EPSC modeling can be used to infer important information, such as the average stress within the sample, identify and constrain the plastic deformation activated, and evaluate stress heterogeneity with the sample.

4:40 PM Invited
A Decade Plus of Making Carlos Tomé Sweat: *Donald Brown*[1]; Bjorn Clausen[1]; Thomas Sisneros[1]; [1]Los Alamos National Lab

The Self-Consistent plasticity models developed by Carlos and his team are in the (un)enviable position of being readily, and robustly, validated by diffraction measurements, meaning the simulations can be easily tested. Since its inception, the SMARTS team has worked closely with Carlos to

"exercise" the models. Toward this end, we have moved to ever increasing complexity in the form of lower symmetry crystal structures (cubic to hexagonal to orthogonal) and more complicated thermo-mechanical histories (cycling, strain path changes, annealing, etc). Concomitantly, we have collected more diffraction data (e.g. during compression and tension) analyzed the data more aggressively, providing increased microstructural information to be used to constrain the model. This talk will use beryllium as an example material to discuss the evolution of both the modeling and experimental efforts toward improving the fidelity between the SC models and the actual physics occurring at the grain level during deformation.

5:05 PM
Finite Element Diffraction Model for Spatially Resolved Strain Measurements in the Bulk of (Poly-) Crystals: *Peter Reischig*[1]; Wolfgang Ludwig[2]; Andrew King[3]; Tilo Baumbach[4]; [1]TU Delft; [2]GEMPPM-MATEIS, INSA de Lyon; [3]GKSS-Forschungszentrum Geesthacht GmbH; [4]Institute for Synchrotron Radiation, Karlsruhe Institute of Technology

Measuring local strain state in the bulk of polycrystalline materials or single crystals is key to understanding their deformation processes and damage phenomena. We propose a linear model to spatially resolve the complete deformation tensor, i.e. 3 rotational and 6 elastic strain components, inside individual grains (diffraction contrast tomography) or a single crystal (rocking curve imaging) from full beam diffraction data. Practical implementation of the model comprises the concepts of finite elements, ray-tracing and kinematical diffraction. The linear approach of the crystal diffraction behavior under deformation enables fast and efficient computation and a rigorous analysis of the well-posedness of the inverse problem. The model allows for direct coupling with crystal plasticity simulation, hence for the verification of such models. Preliminary results on simulated and measured data will be presented.

5:25 PM
The Bauschinger Effect in Austenitic (317L) Stainless Steel: Validating a Slip-System Based Non-Linear Kinematic Hardening Model with In-Situ Neutron Diffraction: *James Wollmershauser*[1]; Bjørn Clausen[2]; Carlos Tomé[2]; Sean Agnew[1]; [1]University of Virginia; [2]Los Alamos National Laboratory

Accurate prediction of the Bauschinger effect is considered a litmus test for the validity of strengthening theories. The effect is known to arise from 'backstresses' having intergranular and intragranular sources. Self-consistent polycrystal models inherently capture intergranular effects but typically fail to account for intragranular (dislocation-based) sources. This inadequacy is illustrated by comparisons of model predictions with in-situ neutron diffraction measurements of the hystereses and internal stresses within a sample subjected to tension-compression fatigue. A single non-linear kinematic hardening rule, similar to the Armstrong-Frederick model, is implemented at the slip system level. With this simple adaptation, the polycrystal model accurately predicts the hysteresis loops and internal strains observed during the aforementioned in-situ low cycle fatigue tests, as well as monotonic stress strain curves and texture evolution after larger strain deformation. The physics-based polycrystal plasticity approach obviates the need for multiple kinematic hardening terms used in phenomenological models.

5:45 PM
Study of Lattice Strain Based on the Finite Strain Elastic-Viscoplastic Self-Consistent Model for Polycrystalline Materials: *Huamiao Wang*[1]; Peidong Wu[1]; Carlos Tome[2]; [1]McMaster University; [2]Los Alamos National Laboratory

The recently developed large strain elastic-viscoplastic self-consistent (EVPSC) model for polycrystalline materials is used to study the lattice strain measurements for conventionally extruded magnesium alloy AZ31 under uniaxial tension and compression. The EVPSC model is a completely general elastic-viscoplastic, fully anisotropic, self-consistent polycrystal model, applicable at large strain and to any crystal symmetry. At single crystal level, both the rate sensitive slip and twinning are included as the plastic deformation mechanisms, while elastic anisotropy is accounted in the elastic moduli. The transition from the single crystal plasticity to polycrystal plasticity is based on a completely self-consistent approach. In the present study, effects of texture evolution, twinning activity and stress relaxation on lattice strain are interpreted by the EVPSC model. It is found that the stress relaxation has a significant effect on the lattice strain measurements.

6:05 PM
High Pressure Deformation of Zirconium: James Wilkerson[1]; *Sven Vogel*[1]; Donald Brown[1]; [1]Los Alamos National Laboratory

Uni-axial deformation of pure Zirconium and Zircaloy-2 was performed with the D-DIA apparatus at the APS. The transformation from α-Zr to ω-Zr during hydrostatic compression was observed at ~4.5 GPa at room temperature with the applied pressure derived from the unit cell volume of α-Zr and the known equation of state. A combination of texture analysis of the diffraction data and energy minimization-based variant selection during texture modeling allows to establish the orientation relationship between the two phases for the first time from a large number of grains. We found that both published orientation relationships, derived from TEM data, are needed to describe the texture change during the phase transformation. From the uni-axial deformation at applied pressure of Zircaloy-2 we found that the twin activity at elevated pressure is increased compared to deformation at ambient pressure, confirming predictions by Lebensohn & Tomé.

Processing and Properties of Powder-Based Materials: Sintering Science and Technology

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Powder Materials Committee
Program Organizers: K. Morsi, San Diego State University; Ahmed El-Desouky, San Diego State University

Monday PM
February 28, 2011
Room: 33A
Location: San Diego Conv. Ctr

Session Chair: Randall German, San Diego State University

2:00 PM Introductory Comments

2:05 PM
Process Simulation of Cold Pressing and Sintering of Armstrong CP-Ti Powders: Sarma Gorti[1]; *Adrian Sabau*[1]; William Peter[1]; Stephen Nunn[1]; Yukinori Yamamoto[1]; Wei Chen[1]; [1]Oak Ridge National Laboratory

A computational methodology is presented for the process simulation of cold pressing and sintering of Armstrong CP-Ti powders. Since the powder consolidation is governed by specific pressure-dependent constitutive equations, solution algorithms were developed for the ABAQUS user material subroutine, UMAT, for computing the plastic strain increments based on an implicit integration of the nonlinear yield function, flow rule, and hardening equations. Sintering was simulated using a model based on diffusional creep using the user subroutine CREEP. The initial mesh, stress, and density for the simulation of sintering were obtained from the results of the cold pressing simulation, minimizing the errors from decoupling the cold pressing and sintering simulations. Numerical simulation results are presented for the cold compaction followed by a sintering step of the Ti powders. The numerical simulation results for the relative density were compared to those measured from experiments before and after sintering, showing that the relative density can be accurately predicted.

2:25 PM
Hot Isostatic Pressing of Beta Titanium Alloy Powders: *Xinjiang Hao*[1]; Nicholas Wain[1]; Xinhua Wu[1]; [1]University of Birmingham

Hot isostatic pressing ("HIPping") of Ti alloy powders is an effective near net-shape technique to produce complex parts which can have similar or even superior mechanical properties to wrought alloys. Ti alloy powders (such as Ti-6Al-4V) are normally hipped at temperature below beta-transus to avoid excessive grain coarsening. However, the beta-transus of metastable beta or

beta titanium alloys is lower than alpha-beta alloys and this makes hipping of beta alloys a challenge to produce a good bonding between powders and therefore good properties. In this work, powder bonding mechanisms in several beta titanium alloys (Ti-5553, Ti-6246 etc) were investigated and the hipping parameters were optimised.

2:45 PM

Effect of Particle Size Particle Surface Modification Pressing Pressure and Sintering Temperature on Microstructure and Mechanical Properties P/M Al-B4C Composites: *Fatih Toptan*[1]; Isil Kerti[1]; Sibel Daglilar[1]; Ahmet Sagin[1]; Taner Hacioglu[1]; [1]Yildiz Technical University

Al–Ti–B grain refiners are widely used as aluminium grain refiners despite the problems in application Al–Ti–C refiners have an increasing demand in recent years. In the present work, Al-Ti-C grain refiners with different Ti:C ratios were produced by in-situ method with the addition of elemental carbon into the Al-Ti master alloy and addition of K2TiF6 and elemental carbon powder mixture into the commercially pure aluminium. Microstructures were characterised by optic microscope and scanning electron microscope equipped with energy dispersive spectroscopy. The effects of production method and Ti:C ratio on the grain refinement process was investigated with Alcoa Coldfinger Test and optimum conditions were determined. Commercial Al-Ti-B grain refiners also tested with Alcoa Coldfinger Test in identical conditions and it has been stated that, insitu Al–Ti–C refiners are more effective than commercial Al-Ti-B grain refiners in grain refining.

3:05 PM

Microstructure and Mechanical Properties of TiNbSn-HA Biocomposite Fabricated by Mechanical Alloying and Sintering: Xiaopeng Wang[1]; *Yuyong Chen*[1]; Kee-Do Woo[2]; [1]Harbin Institute of Technology; [2]Chonbuk National University

Ti-based alloys have been used as biomaterials, due to their excellent properties. Recently nontoxic elements ß type Ti-based alloys cause researchers' interest because of their lower elastic modulus. But poor bioactivity of Ti-based alloys becomes a problem. Hydroxyapatite(HA) has tightly combination ability with bones tissue. However, poor mechanical properties limit their application. In present paper, ß type TiNbSn-HA composites were fabricated by mechanical alloying and sintering. These composites are expected to integrate advantages of TiNbSn alloy and HA. Elastic modulus, compression strength, wear resistance and microstructure of composites were investigated. XRD and DSC results showed that Ti was suggested to be fully transformed from a phase to ß phase after12h milling, transform temperature was 380.06□. TEM results indicated that nanostructure was obtained. With the increase of Sn content, composites' microstructure changed. Compression test and wear test results indicated that composites had lower elastic modulus and superior mechanical properties.

3:25 PM

Physical Principles and Technological Tools for the Control of Densification, Shape and Diffusion Bonding during HIP of Metal Powders And Ceramics: Charles Barre[1]; *Viktor Samarov*[1]; Dmitry Seliverstov[1]; Evgeny Khomyakov[1]; Roman Haykin[1]; [1]Synertech PM Inc.

The process to manufacture complex-shaped homogeneous or diffusion bonded products using Hot Isostatic Pressing (HIP) of power materials is utilizing a suit of the advanced technologies related to this consolidation process. The most essential of them is advanced computer modeling that enables to provide very complex shapes and a significant reduction of development and manufacturing costs. The suit also includes design of the HIP tooling to provide cost efficient manufacturing solutions and non-destructive inspection for complex shape parts with cavities. The paper analyses, the physical principles and technological tools for the control of densification, shape, mechanical properties and diffusion bonding of large and complex shape parts during HIP of various metal powders and ceramics.

3:45 PM Break

3:55 PM

Effect of Hcl Concentration on Zrb2 Separation from a Self-Propagating High-Temperature Synthesis (SHS) Product: *Burcu Akkas*[1]; Murat Alkan[1]; Bora Derin[1]; Onuralp Yucel[1]; [1]Istanbul Technical University

The self-propagating high-temperature synthesis (SHS) of zirconium diboride followed by hydrochloric acid leaching was carried out in this study. In the SHS stage, the initial mixture of B2O3, ZrO2 and Mg powders were ignited and then a mixture of the ZrB2, MgO, Mg3B2O6 and Mg2B2O5 were formed. In the leaching step, the SHS product was leached in the acid solution to remove impurities i.e. MgO, Mg3B2O6 and Mg2B2O5. The effects of temperature and acid concentration on the selective leaching were investigated. The obtained products were characterized by using X-ray diffraction and SEM techniques.

4:15 PM

Microwave Sintering of Different Aluminum Alloy Powders and Their Characterization: *Padmavathi Chandran*[1]; A Upadhyaya[1]; Dinesh Agrawal[2]; [1]Indian Institute of Technology, Kanpur, India; [2]The Pennslyvannia State University

Microwave processing has been emerged as a well recognized processing technique due to its advantages such as rapid heating rates, time savings, reduction in processing temperature, energy, and environmental friendly. The present work investigates the densification behaviour, microstructural and phase evolution during microwave sintering of various grades of aluminum alloy powders using multimode furnace operating at 2.45GHz. The microwave sintering resulted in approximately 55% reduction in processing time as compared with conventional sintering. The microwave sintered samples exhibited the higher densification response and absence of intermetallics phases as when compared with conventional counterparts. Keywords: P/M Al alloy, multimode-microwave sintering, densification behavior, microstructural aspects, phase transformation.

4:35 PM

Die-Press and Sinter Processing of Armstrong Process® CP-Ti and Ti-6Al-4V Powders for Near-Net-Shape Manufacturing: *Wei Chen*[1]; Yukinori Yamamoto[1]; Stephen Nunn[1]; Jim Kiggans[1]; Michael Clark[1]; Sarma Gorti[1]; Adrian Sabau[1]; William Peter[1]; Craig Blue[1]; Brian Fuller[2]; Kamal Akhtar[2]; [1]Oak Ridge National Laboratory; [2]Cristal US, Inc. / International Titanium Powder

This work investigates the die-pressing and sintering processes of new titanium and titanium pre-alloyed powders made by Armstrong Process® technology. The CP-Ti and Ti-6Al-4V powders were uniaxially die-pressed at designated pressures up to 100 ksi to form disk samples with varying aspect ratios (thickness to diameter). Samples with high aspect ratios tended to exhibit non-uniform green density along the pressing axis. This led to non-uniform shrinkage after sintering, forming disks of an hourglass shape. In addition, the sintering shrinkage of diameter tended to be higher than that of thickness for all samples. Effects of multiple different die-pressing techniques such as holding and repeated pressure strikes were performed to obtain desired sample shape after sintering. In-situ and ex-situ sintering were also performed to record the powder morphology change. This research was sponsored by the U.S. DOE, Office of EERE Industrial Technologies Program, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

Recent Developments in the Processing, Characterization, Properties and Performance of Metal Matrix Composites: Multimodal, Processing and Microstructure

Sponsored by: The Minerals, Metals and Materials Society
Program Organizers: Martin Pech-Canul, Centro de Investigacion y de Estudios Avanzados del Instituto Politecnico Nacional; Zariff Chaudhury, Arkansas State University; Golam Newaz, Wayne State University

Monday PM
February 28, 2011
Room: 6A
Location: San Diego Conv. Ctr

Session Chair: Zariff Chaudhury, Arkansas State University

2:00 PM
Effect of Al+B4C Agglomerate Size on Mechanical Properties of Trimodal Aluminum Metal Matrix Composites: *Bo Yao*[1]; Travis Patterson[1]; Yongho Sohn[1]; Matthew Shaeffer[2]; Cory Smith[3]; Mark van den Bergh[3]; Kyu Cho[4]; [1]University of Central Florida; [2]The Johns Hopkins University; [3]DWA Aluminum Composites; [4]U. S. Army Research Laboratory

Trimodal aluminum (Al) metal matrix composites (MMCs) consisting of a nanocrystalline Al (ncAl) phase, B4C reinforcement particles, and a coarse grain Al (cgAl) phase contain multiscale microstructure and interfacial features that contribute to exceptional mechanical properties. One of the hierarchal microstructural features within the composite, namely the ncAl+B4C agglomerates formed during cryomilling, is an important feature whose size can range from tens of micro-meters to millimeters. This paper reports a study on the influence of the ncAl+B4C agglomerate size on the dynamic compressive strength of the trimodal Al MMCs. A desired hierarchal microstructure for high-strength of Al MMCs is identified to contain uniformly distributed relatively small ncAl+B4C agglomerates. Commercial-scale fabrication process to achieve this microstructure includes size classification by sieving the cryomilled ncAl+B4C agglomerates.

2:20 PM
Alternate Processing of Trimodal Aluminum Composites: *Joseph Paras*[1]; Deepak Kapoor[1]; Tony Zahrah[2]; Rod Rowland[2]; [1]U. S. Army ARDEC; [2]Matsys Inc.

The Trimodal Aluminum 5083-Boron Carbide (Al5083-B4C) system has generated much military interest due to the composite's light weight and high strength. The system has traditionally been processed through powder metallurgy means via cryomilling, resulting in long processing times. In an effort to make the process more efficient, we have explored dry high energy milling as an alternate process. Preliminary results indicate that there is very little difference between cryomilled Bimodal Al5083-B4C and dry milled Bimodal Al5083-B4C. It was also observed that poor bonding can exist between the bimodal composition and the coarse-grained Al5083 when consolidated. This was remedied by light milling of the coarse-grained Al5083 to break the nascent oxide layer and provide a clean metallurgical interface. Evidence of the increased bonding is seen in the fracture surfaces, and mechanical testing has shown a 10% increase in compressive strength and a 30-50% increase in ultimate failure strain.

2:40 PM
Effect of Layered Interface on Mechanical Behaviors in TiO2 Nano-Particle Reinforced Aluminum Matrix Composites: *Jaehyuck Shin*[1]; Hyunjoo Choi[1]; Donghyun Bae[1]; [1]Yonsei University

Interface characteristics, with their mechanical behaviors, have been investigated for aluminum based composites reinforced with titanium dioxide nano-particles (TiO2, 20 nm). The composites are produced by hot rolling the ball-milled mixture of Al powders and TiO2 nano-particles. During a milling process, TiO2 nano-particles are gradually dispersed and embedded within the Al powders. After the subsequent annealing process, an interface layer composed of TiO and □-Al2O3 has newly formed between the TiO2 nano-particles and the aluminum matrix. The composites show significant increase in yield strength with increasing volume fraction of TiO2 nano-particles (514 MPa with 5vol.% TiO2 nano-particle), and the semi-coherent interface layer between the Al matrix and the TiO2 nano-particle enables the nucleation of dislocation at the particle/matrix interface during the plastic deformation. Mechanical properties with the variation of volume fraction of TiO2 nano-particles and their deformation behaviors will be presented.

3:00 PM
Fabrication of Extruded Aluminum Composites Reinforced by Vapor Grown Carbon Nanofibers and Characterization, Modeling of Their Properties: *Fumio Ogawa*[1]; Toshiyuki Nishimura[2]; Chitoshi Masuda[1]; [1]Waseda University; [2]National Institute for Materials Science

Aluminum composites reinforced by vapor grown carbon nanofibers (VGCNF) are expected to be used as thermal management devices and structural parts due to high strength and thermal conductivity of VGCNF e.g. about 2000W/mK along fiber direction. However, because of transverse anisotropy of VGCNF e.g. about 30W/mK transverses to fiber direction, it is expected their properties strongly depend on fiber orientation. In this study, composites were fabricated by powder metallurgy and extruded to control fiber orientation. Microstructure such as orientation, length of VGCNF, porosity in matrix was investigated. And relation of them with extrusion condition was discussed. Strength, thermal conductivity along and transverse to extrusion direction were measured and effect of orientation, anisotropy of VGCNF on them was studied. Modeling of their properties as function of fiber anisotropy, fiber aspect ratio, matrix and interface was conducted and mechanism of reinforcing and heat transfer in composites was discussed.

3:20 PM
Multi-Scale Modeling on the Mechanical Behavior of Aluminum-Based Metal-Matrix Nano Composites (MMNCs): *Changsoo Kim*[1]; Pradeep Rohatgi[1]; Amirreza Sanaty-Zadeh[1]; [1]University of Wisconsin Milwaukee

A systemic, multi-scale computational model has been developed to describe mechanical behavior of metal-matrix nano composites (MMNCs). The modeling begins by using nano-scale (molecular dynamics, MD) simulations for the quantitative evaluation of material properties and dislocation movements. The results from MD simulations are then incorporated as a basis for the meso-scale (finite element analysis, FEA) simulations to predict mechanical performance and failure of MMNCs. Pseudo-plastic failure has been approximated for the fracture of MMNCs. Using this model, the influence of microstructural features such as fine, coarse grain sizes, fine-to-coarse heterogeneous multi-modal phase ratio, and the type, shape, contents of reinforcement particles on the macroscopic mechanical performance can be quantitatively characterized. The developed algorithm has been applied to Aluminum-based (Al) fine/coarse multi-modal MMNCs that are reinforced with SiC and/or B4C particles. The presentation will elucidate the potential failure mechanisms and propose optimized microstructures of these Al MMNC systems.

3:40 PM Break

4:00 PM
Strain Induced Grain Growth of Cryomilled Nanocrystalline Al in Tri-Modal Al Composites during Forging: *Bo Yao*[1]; Cory Smith[2]; Mark van den Bergh[2]; Bhaskar Majumdar[3]; Yongho Sohn[1]; [1]University of Central Florida; [2]DWA Aluminum Composites; [3]New Mexico Institute of Mining and Technology

This report presents our study on the grain growth of nanocrystalline aluminum (NC-Al) in tri-modal Al metal-matrix-composites (MMCs) under forging process. The NC-Al phase formed through cryomilling in liquid nitrogen atmosphere exhibits an excellent thermal stability when annealed without applied stress. However, grain growth was observed when the Al MMCs were forged at temperatures where the NC-Al grain growth might have remained stable without applied stress. The influence of strain and temperature on the NC-Al grain growth during forging is presented. The grain growth activation energy with forging is lower than that without any

applied stress. The role of pinning effect by dispersoids and impurities at grain boundaries is presented and discussed.

4:20 PM

Origin of High Strength in a Micro-Alloyed Ferritic Steel: *Hesamaldin Askari*[1]; Yong Shen[2]; C. Wang[2]; Xin Sun[3]; Hossein Zbib[1]; [1]Washington State University; [2]Northeastern University; [3]Pacific Northwest National Laboratory

The strengthening role of carbide precipitates in a high strength ferritic steel with finely dispersive precipitates was investigated using a combined experimental and modeling approach. Using EDXS and TEM, fine carbides with an average diameter of 10 nm were observed in the ferrite matrix of the 0.08%Ti steel, as well as some cubic M23C6 precipitates at the grain boundaries. The dual precipitate structure provides combined matrix and grain boundary strengthening. The increase in strength due to precipitation is 2-3 times higher than the conventional high strength steels depending on the average size and distance of the nanoscale precipitates. The effects of the particle size and density on the strength have been investigated through Dislocation Dynamics simulations. The DD simulations and experimental analysis showed that the finely dispersive precipitates can strengthen the steel by pinning the dislocations up to a critical shear stress and retarding the recovery and annihilation of dislocations.

4:40 PM

Synthesis of MWCNT Reinforced Al Based Nanocomposite Via Spark Plasma Sintering: *Tapas Laha*[1]; Lakshmikanth Reddy[1]; Anup Keshri[2]; Devrupa Lahiri[2]; Anway Maiti[1]; [1]Indian Institute of Technology; [2]Florida International University

A trend has been perceived in the field of composite materials to employ carbon nanotubes as reinforcement in synthesizing composites of unique properties. In this endeavor, Al-based nanostructured composite with carbon nanotubes, as second phase particles, has been synthesized by combination of two novel processing techniques, viz. Cryomilling and Spark Plasma Sintering. Scanning electron microscopy and quantitative image analysis has been performed to understand the microstructural evolution and to verify the retention of carbon nanotubes. Nanoindentation and microhardness test have been carried out to obtain the effective elastic modulus and hardness of the consolidated samples. Tribological study on the nanocomposites has been done in order to understand the effect of CNT reinforcement on the mechanical properties. The characterization affirms the retention of carbon nanotubes in the nanocomposites. Raman spectroscopy revealed that the MWCNTs were partially damaged during cryomilling.

5:00 PM

Effects of SPS Parameters on the Mechanical Properties and Microstructures of Titanium Reinforced with Multi-Wall Carbon Nanotubes Produced by Hot Extrusion: *Thotsaphon Threrujirapapong*[1]; Katsuyoshi Kondoh[1]; Junko Umeda[1]; Bunshi Fugetsu[2]; [1]Osaka University; [2]Hokkaido University

The effects of spark plasma sintering (SPS) temperature and time on the mechanical properties and microstructures of titanium (Ti) reinforced with multi-wall carbon nanotubes (MWCNTs) were investigated. The Ti powders were coated with MWCNTs via a wet process using a zwitterionic surfactant solution containing 2.0 wt.% of MWCNTs. The coated Ti powders were consolidated by SPS process at various temperatures and times. All sintered billets were subsequently extruded at 1000oC. The mechanical properties responses of these extruded Ti composites showed the decrease of yield and tensile strength when higher consolidation temperatures were used. The microstructures of those Ti composites were observed using optical microscopy and scanning electron microscopy. All of the results were used to select the suitable condition for fabrication of Ti/MWCNTs composite materials.

5:20 PM

On the Methods for Grain Size Analysis and Grain Growth Kinetic Studies for a Thermally Stable Al 5083 Nanocomposite: *Leyla Hashemi-Sadraei*[1]; S. E. Mousavi[1]; Rustin Vogt[1]; Ying Li[1]; Zhihui Zhang[1]; Enrique Lavernia[1]; Julie Schoenung[1]; [1]University of California Davis

A nanocomposite of Al 5083 and B_4C was fabricated by cryomilling the powders for 24 hours. Powders annealed at temperatures as high as approximately 0.9 T_m were investigated for grain growth and were found to exhibit superior thermal stability compared to Al 5083 powders without B_4C that were cryomilled for shorter durations. The high thermal stability was attributed to the pinning effect of ultrafine secondary dispersoids residing on grain boundaries. Several approaches were evaluated, on the basis of X-ray diffraction and transmission electron microscopy, in order to determine average grain sizes. Grain size results from alternative X-ray-based methods were utilized in order to study grain growth mechanisms by fitting grain growth curves to established equations. Two distinct temperature-dependent grain growth regimes were observed. By comparing the kinetic results achieved when using different grain size calculation methods, the relevance and applicability of each method is described.

Size Effects in Mechanical Behavior: Size Effects in Multilayer Structures

Sponsored by: The Minerals, Metals and Materials Society, Not Applicable, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Erica Lilleodden, GKSS Research Center; Amit Misra, Los Alamos National Laboratory; Thomas Buchheit, Sandia National Laboratories; Andrew Minor, UC Berkeley & LBL

Monday PM
February 28, 2011
Room: 2
Location: San Diego Conv. Ctr

Session Chairs: Amit Misra, LANL; Jian Wang, Los Alamos National Laboratory

2:00 PM

A Quantized Crystal Plasticity Finite Element Model for Nanoindentation: Size Effect from Discrete Plasticity: *Lin Li*[1]; Myoung-Gyu Lee[2]; Peter Anderson[1]; [1]The Ohio State University; [2]Pohang University of Science and Technology

Quantized crystal plasticity (QCP) simulations are employed to study a distinctive observation from an in-situ nanoindentation test on nanocrystalline Al. This displacement controlled indentation test shows that the sample surface recedes even faster than the rate of indenter as a consequence of incipient dislocation events. This direct experimental observation emphasizes the discrete nature of dislocation slip in a confined volume. The QCP model incorporates such a feature showing that the plastic shear strain can jump by discrete amounts that scale as 1/grain size. This unique feature is motivated by recent molecular dynamics results, showing that the grain averaged shear strain jumps ~1% as a dislocation unstably traverses a grain. This quantized aspect enables the QCP model to simulate the load-displacement trace and the underlying slip pattern during indentation. Also, the grain size effect on indentation response can be assessed through a comparison of the conventional and quantized simulations.

2:20 PM

Size Effects in Fatigue of Cu/Ta Multilayers: *Guangping Zhang*[1]; Xiaofei Zhu[1]; Jiawei Yan[1]; Jin Xu[1]; [1]Chinese Academy of Sciences

Although strengthening and deformation mechanisms of metallic multilayers with different length scales ranging from microns to nanometers are investigated extensively, fatigue behavior of the metallic multilayers is known little. Here we present a systematic investigation of fatigue properties of Cu/Ta multilayers deposited on a flexible polyimide substrate. We will show that fatigue resistance of the multilayers gradually increases with decreasing individual layer thickness from 500 nm to 20 nm. Microscopic characterization reveals an evident transition of fatigue damage from typical

bulk-like extrusions/intrusions to boundary-localized behavior. Such size effects in fatigue are evaluated and compared with recent findings of fatigue of thin metal films constrained by a substrate as well as unconfined small-volume metals.

2:40 PM
Microstructure and Strengthening Mechanisms of Highly Textured Cu/Ni Multilayers: *Yue Liu*[1]; Dan Bufford[1]; Haiyan Wang[1]; Cheng Sun[1]; Xinghang Zhang[1]; [1]Texas A&M University

We report on the synthesis of sputtered, highly (111) and (100) textured Cu/Ni multilayers with individual layer thickness, h, varying from 1 to 200 nm. At greater h, XRD patterns of Cu and Ni (100) (or (111)) peaks are clearly separated indicating that the existence of semi-coherent interface. When h decreases to 10 nm or less, XRD spectra show significant peak distortions due to coherency stress. High resolution TEM studies confirm the coexistence of twin and coherent layer interfaces in highly (111) textured Cu/Ni mutilayers. Multilayer hardnesses increase with decreasing h, approach a maxima at h of 2.5-5 nm, and show softening thereafter. A detail comparison between (111) and (100) textured Cu/Ni is made in both microstructure and strengthening mechanisms. The influences of both coherent layer interfaces and twin interfaces on strengthening mechanisms are discussed.

3:00 PM
Hardening in He Ion Implanted V/Ag Multilayers: *Qiangmin Wei*[1]; Nathan Mara[1]; Michael Nastasi[1]; Amit Misra[1]; [1]LANL

Hardness before and after irradiation as a function of layer thickness on sputter-deposited V/Ag multilayers, as well as pure Ag and V, was measured by nano-indentation. Radiation-induced hardening was found to decrease with decreasing individual layer thickness. No significant radiation hardening was observed at layer thickness less than 6 nm within the error limit of equipment. A model describing radiation effects on the hardness of multilayers with length scale from micrometer to less than one nanometer was developed on the basis of Freidel model and confined layer slip (CLS) model. At layer thickness larger than 50 nm, hardening from He bubbles dominates. With decreasing layer thickness, hardening from the interfaces becomes dominant with less contribution from He bubbles.

3:20 PM Invited
Deformation Mechanisms of Nanoscale Multilayers: *Jian Wang*[1]; Amit Misra[1]; [1]LANL

Nanoscale metallic multilayer composites exhibit relatively high flow strengths in comparison with bulk polycrystalline materials due to the presence of the high density of interfaces in composites. The flow strength of composite materials produced in this way increases as decreasing the thickness of individual layers. The systematic study regarding deformation mechanisms of layered composites from experimental measurements to theoretical and numerical studies, suggests that crossing of dislocations across interfaces becomes an important unit process at layer thicknesses in the range of a few nanometers, in turn, implying that interface structures and the resultant properties of interfaces play a vital role in determining material strength. The difficulty of crossing of dislocations across interfaces is ascribed to different mechanisms with respect to interface types. Using atomistic simulations, deformation mechanisms in nanolayered materialls, such as Cu/Nb layered composites are discussed. Weak interface strengthening mechanisms are developed and examined.

3:50 PM Break

4:20 PM
Fundamental Investigation of Deformation Using Spherical Indentations and 3D X-Ray Microscopy: *Bennett Larson*[1]; Jon Tischler[1]; Yanfei Gao[2]; [1]Oak Ridge National Laboratory; [2]Oak Ridge National Laboratory, University of Tennessee Knoxville

3D x-ray microscopy measurements of the spatially confined lattice distortions under spherical indentations provide an opportunity for fundamental investigations of deformation on mesoscopic length scales and detailed testing of finite element crystal plasticity simulations of deformation in crystalline materials. Direct comparisons of 3D x-ray microscopy measurements of deformation below 100 mN, 100 micron radius spherical indents in (100), (110), and (111) oriented Cu with crystal plasticity simulations have shown remarkable overall agreement. However, both qualitative and quantitative differences are found to exist between measured and simulated local rotational distortion components and geometrically necessary dislocation density patterns. Strong, depth dependent dislocation patterning under indents provides a further fundamental challenge to deformation simulations. Research sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy.

4:40 PM
Structure and Hardness of Aged Ti-W Multilayer Films: *Ross Economy*[1]; Marian Kennedy[1]; [1]Clemson University

Metallic multilayer film systems are being studied for potential use in microelectromechanical systems and freestanding high-strength structures. Control of layer thickness has been shown to influence the mechanical response by controlling dislocation slip. Only a few groups, however, have looked at the influence of aging and porosity on the mechanical properties of these multilayer systems. As a result, this study will look at the influence of film porosity and thermal aging on the properties of Ti-W multilayer systems. 500nm Ti, W, and nano-porous Ti thin films, as well as multilayer Ti-W systems (20nm layers), were deposited onto Si(100) via magnetron sputtering. SEM and AFM were used to characterize film morphology and microstructure. Hardness was characterized using nanoindentation. Initial data shows that multilayer film systems exhibit an increase in hardness of ˜200% when compared with bulk W and a slight decrease when compared to the W films.

5:00 PM
Flow Stress Partitioning in Two-Phase Metallic Composites at Decreasing Phase Length Scales: *Rainer Hebert*[1]; Girija Marathe[1]; [1]University of Connecticut

Repeated cold-rolling and folding of metallic multilayers generates two-phase composite materials with continuously decreasing length-scales in the layer thickness direction. Within a few rolling and folding passes, the layer thickness decreases for systems such as Cu-Ni from the micrometer to the nanometer scale. Instrumented indentation measurements are used to probe the hardness and flow stresses of the individual layers at increasing strain levels. During the initial stages, i.e. at the micrometer scale for the layer thickness, the indentation measurements allow for studies of the impact of the strain compatibility between the layers on the flow stress evolution. The strain compatibility between the layers modifies the work-hardening behavior and the observed changes in the flow stress evolution are discussed in light of crystal plasticity models based on geometrically necessary dislocations. The results reveal a mutual impact between the mechanical properties of the individual layers and the geometry of the layer arrangement.

5:20 PM
Film Thickness Effects on Interfacial Failure in Polymer Metal Thin Film Structures: *Neville Moody*[1]; Markus Ong[2]; E. David Reedy Jr.[1]; Edmundo Corona[1]; David Adams[1]; John Yeager[3]; David Bahr[3]; [1]Sandia National Laboratories; [2]Whitworth University; [3]Washington State University

Interfaces are the critical feature governing performance of polymer-metal thin film structures where differing properties between adjacent films can induce strong interlaminar stresses and catastrophic failure. We are studying these effects in a model system created by spin coating 10 to 650nm thick PMMA films onto copper coated silicon substrates followed with a sputter deposited overlayer of highly stressed tungsten. The high film stresses triggered spontaneous delamination and buckling along the PMMA-tungsten interface accompanied by intense deformation in the PMMA layers that varied markedly between each system studied and from model elastic behavior. In this presentation we will use crack growth simulations and comparison to metal film structures to show that film compliance provides a lower bound to behavior for all samples while constrained yielding accounts for the pronounced differences in behavior between samples. This work was

supported by Sandia National Laboratories through USDOE NNSA Contract DE-AC04 94AL85000.

5:40 PM
Size Effect in Cleavage Cracking in Polycrystalline Thin Films: *Yu Qiao*[1]; Weiyi Lu[1]; [1]University of California San Diego

As a cleavage crack front propagates across a wide high-angle grain boundary, it would first penetrate across a number of break-through points, and the persistent grain boundary areas would then be separated through shear fracture or ligament bending. Therefore, as the film thickness is smaller than the characteristic distance between the break-through points, which is often in the range of 1-5 microns, the crack front transmission can be significantly confined by the film surfaces leading to an either beneficial or detrimental size effect. That is, the fracture toughness of the polycrystalline thin film is not a material constant; rather, it highly depends on the film thickness. This concept was recently validated in our bicyrstal thin film testing.

Surfaces and Heterostructures at Nano- or Micro-Scale and Their Characterization, Properties, and Applications: Growth, Characterization, and Devices II - and - Coatings, Surfaces, and Interfaces I

Sponsored by: TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Nanomaterials Committee, TMS: Surface Engineering Committee
Program Organizers: Nitin Chopra, The University of Alabama; Ramana Reddy, The University of Alabama; Jiyoung Kim, Univ of Texas; Arvind Agarwal, Florida International Univ; Sandip Harimkar, Oklahoma State University

Monday PM
February 28, 2011

Room: 31B
Location: San Diego Conv. Ctr

Session Chairs: Nitin Chopra, The University of Alabama; Jiyoung Kim, University of Texas at Dallas; Sandip Harimkar, Oklahoma State University; Arvind Agarwal, Florida International University

2:00 PM Invited
On the Role of Interface Engineering in Future Technology Applications: *Aarthi Venkateshan*[1]; Rajendra Singh[2]; [1]Canon Anelva Corporation; [2]Clemson University

The global need for high performance, low power computing continues as a major driver of the semiconductor industry. A decade ago, Moore's Law scaling meant oxide thickness, transistor length and width were scaled by factor (1/k) to provide delay improvement 1/k at constant power density. In subsequent generations, performance enhancers were added to drive the transistor roadmap forward. The continued decrease in feature sizes means management of interfaces will play an important role in future scaling. High quality ZrO2 high-k films were grown, EOT 1.4 nm 1V leakage current density 1.83 10-11 A/cm2 was observed. For Al2O3 of EOT 1.04 nm at 1 V leakage current density 5.71 10-11 A/cm2 was achieved. We report results of EOT 0.39 nm HfO2 with leakage current density 1.0 10-12 A/cm2 for gate voltage -3 to +3 V. Based on DOE and process variation data, process is robust and will transfer from laboratory to manufacturing environment.

2:30 PM Invited
Role of Rapid Photothermal Processing in Providing Homogenous Nanostructure of Silica Clad Silicon Fibers: *Rajendra Singh*[1]; Nishant Gupta[1]; T. Wade Hawkins[1]; Paul Foy[1]; Colin McMillen[1]; Stephanie Morris[1]; Robert Rice[2]; Kelvin Poole[1]; John M. Ballato[1]; [1]Clemson University; [2]Northrop Grumman Space Technology

The recent realization of silicon core optical fibers has the potential for rack-to-rack optical interconnects in integrated optoelectronic applications. Incoherent light source based rapid photothermal processing was used to carry out ex-situ annealing on the as-drawn silicon core optical fibers. The dark current-voltage characteristics of annealed silicon fibers diodes showed lower leakage current than the diodes based on as drawn fibers. X-ray diffraction examination of the crystal structure of as-drawn fiber of 5 mm length showed 3 mm stretch of single crystal Si and 2 mm of polycrystalline material. On the other hand, the entire 10 mm length of the annealed sample was crystalline. The role of rapid photothermal processing in obtaining homogenous nanostructure of the silica clad silicon fiber will be presented. Structural, electrical, and optical characterization of annealed and as-drawn fibers will be presented. This work is supported by Northrop Grumman Corporation.

3:00 PM
High Surface Metal/Metal Nano-Microstructured Porous Monoliths: *Martin Bakker*[1]; Franchessa Maddox Sayler[1]; Amy Grano[1]; Keana Graves[1]; [1]The University of Alabama

Porous metal electrodes are of interest in a wide range of electrochemical processes. We report here on a versatile process for making a range of such electrodes using a porous silica template. The porous silica monoliths are produced by a sol-gel process with PEO polymer and cationic surfactant added to give porosity at nanometer and micrometer length scales. Metal nitrates are infiltrated into the silica monolith either as a saturated solution or as a melt. Decomposition under hydrogen results in formation of metal replicas. By controlling the number of infiltration cycles, the extent of replication can be controlled. By changing the metal salt used between infiltration cycles, heterostructures consisting of nanostructures of one metal on a continuous microstructure of a second metal can be produced. Typical surface areas are 40-90 m2/g. Significant variations in pore size distributions are found for different metals and for different methods of producing the metals.

3:15 PM Break

3:25 PM Introductory Comments for Coatings, Surfaces, and Interfaces I

3:30 PM Invited
Microstructural Evolution in NiAlCrHf and NiAlCrZr Coated Superalloys: *Mark Weaver*[1]; Joel Alfano[1]; [1]The University of Alabama

Coated Ni based superalloys are used for power generation due to their ability to function in oxidative and corrosive environments. However, it is well known that the microstructures of these coated systems change in service due to interactions with the environment and interdiffusion with the underlying substrate. In this study, NiAlCr-(Hf,Zr) overlay coatings were produced via dc magnetron sputtering. Post-deposition annealing in a mixture of Ar+5%H_2 and oxidation in laboratory air produced microstructural and chemical changes within the coating/substrate systems which significantly influenced their performance. The purpose this presentation is to describe the results of a study to quantify the microstructural changes occurring in NiAlCrHf and NiAlCrZr coated superalloys in comparison with (Ni,Pt)Al coated superalloys tested under the same conditions.

4:00 PM Invited
Nature of the B2 Phase in Sputter-Deposited 304 Stainless Steel + 10 wt.% Al Coatings: U.M.R. Seelam[1]; *C. Suryanarayana*[1]; T. Ohkubo[2]; Kazuhiro Hono[2]; N. S. Cheruvu[3]; [1]University of Central Florida; [2]National Institute for Materials Science; [3]Southwest Research Institute

A Fe-18Cr-8Ni-10Al (wt.%) coating was deposited on a 304-type austenitic stainless steel substrate by magnetron sputtering technique using SS304 and Al targets. The as-deposited coatings were characterized by XRD, SEM, TEM, and 3DAP techniques. The coating consists of columnar grains with alpha-ferrite and B2 phase particles uniformly distributed. It also has a deposition-induced layered structure with two alternate layers (of 3.2 nm wavelength), one rich in Fe and Cr, and the other rich in Al and Ni. Conventional TEM techniques were able to provide the general microstructural features of the coating, but it was not able to determine the exact nature and chemistry of the B2 phase; 3DAP studies helped in unambiguously identifying that the B2 phase is NiAl. Based on the above

results, it was concluded that NiAl is the preferred phase over FeAl, further correlated with the heats of mixing among Fe, Ni, and Al elements.

4:30 PM Invited
The Solution Precursor Plasma Spray Process for Depositing Multi-Functional, Nanoscale Coatings: *Maurice Gell*[1]; Eric Jordan[1]; [1]University of Connecticut

The Solution Precursor Plasma Spray Process (SPPS) is a highly flexible process that can deposit single- or multi-phase oxide coatings for functional applications. The coating microstructure is on the nanoscale and is compositionally homogeneous. In this presentation, the SPPS process and its utility to fabricate multi-functional materials will be described. Comparisons will be made to alternate plasma spray processing methods, including those using powder and suspensions. The applications include: (a) thermal barrier coatings with a unique strain-tolerant microstructure, (b) coatings with in-situ sensors on the atomic scale, and (c) two-phase transparent ceramics that meet stringent optical and elevated temperature mechanical properties.

5:00 PM
Tribological Behavior of Spark Plasma Sintered Iron-Based Bulk Amorphous Alloys: *Ashish Singh*[1]; Sandip Harimkar[1]; [1]Oklahoma State University

Amorphous alloys (BMG's) exhibit high-micro hardness, fracture toughness, and yield strength at ambient temperature. These properties result in good tribological behavior of BMG's and make it a potential material for high performance wear resistance applications. Spark plasma sintering technique is used to fabricate fully amorphous and in-situ bulk amorphous matrix composites near crystallization temperature of the alloy. Sintered samples exhibited heterogeneous microstructure which involves nano crystallized phases embedded in-between amorphous matrix. Frictional and wear behavior of spark plasma sintered iron based BMG's are investigated under dry sliding conditions. Wear behavior of sintered specimens are analyzed at different normal loads and rotational speeds. The surface roughness, coefficient of friction, and wear loss of the wear tested samples are studied.

The Second Symposium on the Recycling of Electronic Wastes: Management and Technology Overview of Electronic Wastes

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Electronic Packaging and Interconnection Materials Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Lifeng Zhang, Missouri University of Science and Technology; Gregory Krumdick, Argonne National Laboratory; Jaan Kers, Tallinn University of Technology; Thomas P. Schuman, Missouri University of Science and Technology (Missouri S&T); Markus Reuter, Ausmelt Limited

Monday PM
February 28, 2011

Room: 15B
Location: San Diego Conv. Ctr

Session Chairs: Jaan Kers, Tallinn University of Technology; Thomas Schuman, Missouri S&T; Markus Reuter, Ausmelt Limited

2:00 PM
Willingness to Recycle Electronic Waste: Results from a National Survey of U.S. Households: *Jean-Daniel Saphores*[1]; Oladele Ogunseitan[1]; Andrew Shapiro[2]; [1]University of California Irvine; [2]Jet Propulsion Laboratory, California Institute of Technology

The fate of used electronic products (e-waste) is of increasing concern because of the toxicity of e-waste, its growing volume, and the environmental and public health consequences of its inadequate handling. The U.S. EPA estimates that e-waste has become the main contributor of lead (Pb) to landfills. Households also store large volumes of e-waste, yet little is known about their willingness to recycle e-waste at the national level. This paper starts filling this gap based on a 2006 survey of U.S. households. Using multivariate models, we find that gender, education, convenience, moral considerations, environmental beliefs, and knowledge of e-waste toxicity, but neither income nor political affiliation, are key factors explaining people's willingness to drop off e-waste at recycling centers. Our results suggest targeting e-waste public education programs at younger adults and making recycling more convenient for older adults.

2:30 PM
Development of Technology for Recycling Valuable Metals from Electronic Wastes in Korea: *Jae-chun Lee*[1]; Byung-Su Kim[1]; Min-seuk Kim[1]; Jinki Jeong[1]; Banshi Pandey[2]; [1]Korea Institute of Geoscience and Mineral Resources (KIGAM); [2]National Metallurgical Laboratory, CSIR

The generation of electronic wastes in Korea has been increasing significantly over the years. These wastes viewed previously as the source of contaminants for environment due to the hazardous materials are now recognized as the new resources termed as "urbane ore" because of presence of the valuable metals. The development of a sustainable recycling technology for electronic wastes is urgently required with the aim of environmental protection and secured supply of resources simultaneously. The commercial recycling technology of electronic wastes uses pyrometallurgical process to recover copper, gold and silver partially in the existing smelters. This paper elaborates the development of a combined process involving mechanical pretreatment and hydrometallurgical reclamation which has the advantage to recover not only copper but most rare metals as well. The research focus is on the development of the liberation technology using various pulverization methods along with the eco-friendly and energy-saving metal extraction – separation technology.

3:00 PM
State of the Art in the Recycling of Waste Printed Wiring Boards: *Lifeng Zhang*[1]; [1]Missouri University of Science and Technology

Printed Wiring Board (PWB) is a platform upon which microelectronic components such as semiconductor chips and capacitors are mounted. It is also called printed circuit board (PCB). In the PWBs, the organic substances are approximately 72%. The main composition of organic substances in PWBs is ethoxyline resin bromide or ethoxyline resin chlorinate. Depending on the different application and design of the PWB, various metals may be used in the manufacturing process, including precious metals such as copper, lead, silver, gold, platinum, and toxic metals such as mercury, cadmium, barium, gallium, cadmium, lead, bismuth. The current paper reviewed the recycling process for waste PWB materials, including mechanical recycling, combustion for energy recovery and different pyrolysis processes, such as vacuum pyrolysis, atmospheric inert pyrolysi and flux pyrolysis process, and hydrometallurgical recycling process.

3:30 PM
Overview of Electronics Waste Management in India: *Sandip Chatterjee*[1]; Krishna Kumar[1]; [1]Department of Information Technology

Growth of electronics waste (e-waste) and lack of appropriate disposal mechanism are the great concern for developing countries like India. The domestic generation of e-waste in India is rapidly growing with the development. The imported substance is also substantially contributing to the e-waste inventory. Recycling of e-waste is mainly carried out in unorganized units in India. The primitive recycling technique used by the unorganized sector is a great threat to the environment and the health of the operators involved in these profession. Therefore, improvement of technology used by these units towards a cost effective, environmental friendly alternative could be the solution to the present crisis. Few initiatives of recycling have, however, come up in recent years in organized units. The precious metals are still recycled in the developed countries. This report discusses the inventory, technology status, legal legislative and existing practices of e-waste management in India.

4:00 PM Break

4:10 PM

Prospective Scenario of E-Waste Recycling in India: Manis Kumar Jha[1]; *Abhishek Kumar*[1]; Vinay Kumar[1]; Jae-chun Lee[2]; [1]National Metallurgical Laboratory; [2]Korea Institute of Geoscience and Mineral Resources (KIGAM)

However, India is rich in ores and minerals, but e-waste recycling is necessary due to the report of national and international studies, which cautioned on the generation, treatment and accumulation of e-waste in India. Current data indicate that the total domestic e-waste generation, including imports, is around 382979 MT. However, waste available for recycling and actually recycled are 144143 MT and 19000 MT, respectively, in which recycling by non-formal and formal sector are 95% and 5%, respectively. On the other hand, India has developed an expertise in handling varieties of metallic wastes in an organized and safe manner. The development of individual processes or combined processes for handling the e-waste is underway. Eco- friendly and energy-saving processes are necessary to comply with stringent environmental regulations. The paper includes the recent trend of e-waste generation, recycling process and its future prospects particularly in India.

4:40 PM

From a Technical Marvel to a Hazardous Box: An Estimate of the Volumes of Potentially Toxic Materials in Obsolete TVs Stored by US Households Based on National Survey: *Natalia Milovantseva*[1]; Jean-Daniel Saphores[1]; [1]University of California, Irvine

The increasing popularity of consumer electronics continues to present tough challenges for policy makers concerned with the risks for human health and the environment of improperly disposing of electronic waste (e-waste). In 2007, the EPA estimates that approximately 26.9 million TVs were discarded in the US alone, yet only 18 percent by weight was recycled; unfortunately, these numbers are very uncertain. Discarded cathode ray tube (CRT) TVs contain large amounts of lead but flat panel TVs contain some mercury and present new recycling challenges. It is important for public policy to get better estimates of the number of TVs currently in storage and of the quantity of potentially toxic materials they contain. This paper relies on count models to analyze the data from a national survey (n=3165) of US households. Our models estimate the number and the size of obsolete TVs stored by US households using a set of behavioral, socio-economic and demographic variables, and we then approximate the volumes of potentially toxic materials they contain. Our findings provide information to support future state and national policy decisions on the management of hazardous materials.

5:10 PM

Methodology for Recovery Precious Metals: Gold, Silver and Platinum Group from Electronic Waste: *Oscar Restrepo*[1]; Honorio Oliveros[1]; [1]National University of Colombia

Colombia produces nine tons of electronic waste per year and there are approximately 60 tons accumulation generated in the last nine years. This work aims to establish a general methodology in the laboratory to recover gold, silver and platinum groups present in electronic waste and also to explore other forms of recovery of metals contained in electronic waste. We selected seven types of electronic cards present in the computers, which have their parent desoldering electronic components. Initially, we characterized the components of the cards through x-ray diffraction and SEM to identify the metal species and composition. Tests were conducted in laboratory hydrometallurgical recovery of metals. This paper proposes a methodology for laboratory-scale work.

5:40 PM

WEEE: Obsolete Mobile Phones Characterization Aiming at Recycling: Viviane Moraes[1]; Denise Espinosa[1]; *Jorge Tenório*[1]; [1]Escola Politecnica da Universidade de São Paulo

The development of mobile phone technology has increased the replacement of outdated equipment for more modern models, transforming the disposal of such devices into a serious environmental problem. Recycling appears as an attractive alternative to minimize the environmental impact of disposing such large quantities. Recycling can be accomplished by treating the printed circuit boards of used mobile phones with the same techniques used in ore treatments with hammer mill: magnetic separation, electrostatic separation and grain size range. Before performing the recycling process, a PCB characterization was perfomed to identify the fraction with the higher metals concentration/content. This study demonstrated that the electrostatic separation was not the best metals concentration of PCB aiming the recycling, while the non-magnetic residue had presented a more significant metal concentration. Furthermore, after the magnetic separation, it was possible to separate the metals iron and nickel.

Thermally Activated Processes in Plastic Deformation: Grain Boundary Evolution and Dislocation Core Effects

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizer: Christopher Woodward, Air Force Research Laboratory

Monday PM
February 28, 2011

Room: 1B
Location: San Diego Conv. Ctr

Session Chairs: David Rodney, INP Grenoble; Gregory Rohrer, Carnegie Mellon University

2:00 PM Invited

Grain Boundary Diffusion: From Nearly Perfect Bicrystals to Severely Deformed Polycrystalline Materials: *Sergiy Divinski*[1]; [1]University of Münster

An overview of recent advances in grain boundary (GB) diffusion and segregation in bi-crystalline, tri-crystalline, and poly-crystalline metals is presented with an emphasize on the relationship between the structure and kinetic properties. GB diffusion is strongly affected by the structural state of interfaces and segregation of residual impurities. The so-called 'non-equilibrium' GBs are introduced by severe plastic deformation of metals using deferent routes. These GBs are characterized by an increased free volume, larger excess free energy and, as a result, enhanced diffusivities. The kinetic and structure properties of the 'non-equilibrium' GBs are investigated for several pure metals (Cu, Ni, Ti) and Cu-based alloys severely deformed by equal channel angular pressing. The relaxation behavior of interfaces in ultra-fine grained metals is investigated by radiotracer diffusion measurements and differential scanning calorimetry.

2:30 PM Invited

Experimental Measurements of the Shear-Coupled Stress Driven Grain Boundary Migration in Al Bicrystals: *Dmitri Molodov*[1]; Tatiana Gorkaya[1]; Günter Gottstein[1]; [1]RWTH Aachen University

Recent results of experimental research into stress induced grain boundary migration in aluminum bicrystals will be briefly reviewed. Boundary migration under a shear stress was observed to be coupled to a lateral translation of the grains for planar symmetrical <100> tilt boundaries. This coupling proved to be the typical migration mode of any <100> tilt boundary, no matter whether low- or high angle, low Σ CSL coincidence or non-coincidence boundary. The measured ratios of the normal boundary motion to the tangential displacement of the grains were in an excellent agreement with theoretical predictions. Measurements of the temperature dependence of coupled boundary migration revealed that there is a specific misorientation dependence of migration activation parameters. The experimental observations provide evidence that the investigated boundaries can act during their migration under stress as sources of lattice dislocations that results in the formation and growth of new grains in the boundary region.

3:00 PM Invited
Evolution of the Grain Boundary Character Distribution during Grain Growth: *Gregory Rohrer*[1]; Herbert Miller[1]; [1]Carnegie Mellon University

The grain boundary character distribution is the relative area of grain boundaries as a function of the lattice misorientation and grain boundary plane orientation. This paper will focus on experiments undertaken to assess the evolution of the grain boundary character distribution as a function of thermal annealing that leads to grain growth. The analysis is based upon electron backscatter diffraction (EBSD) data, which allows the crystallography and relative areas of all of the grain boundaries to be measured. Data from SrTiO3 taken a range of grain sizes indicates that the grain boundary character distribution changes as the grains grow. Measurement of the relative grain boundary energies indicates that as grain growth proceeds, higher energy boundaries are preferentially eliminated from the distribution.

3:30 PM Break

3:45 PM Invited
Grain Growth in 4D: A Comparison between Simulation and Experiment: E. Lauridsen[1]; I. McKenna[2]; S. Poulsen[1]; W. Ludwig[3]; *Peter Voorhees*[2]; [1]Riso Laboratory for Sustainable Energy; [2]Northwestern University; [3]European Synchrotron Radiation Facility

The evolution of polycrystalline materials is examined using 3D imaging tools based on phase- and diffraction- contrast X-ray tomography and phase field simulations. Since the techniques are nondestructive, it is possible to record the morphological and topological evolution of an individual grain and compare this to the predictions of simulations using the experimentally measured structure as an initial condition. Through this comparison, we find that the phase field model can reproduce quite accurately grain morphology and topology in regions of the Ti-21S sample with isotropic grain boundary properties. However, in other regions, we find grain boundary mobilities that are orders of magnitude larger than the average, mobilities that depend strongly on the grain boundary normal, and clear influence of grain boundary energy anisotropy on the morphological evolution of grains.

4:15 PM
A Comparison between Creep in Molybdenum and Iron toward Understanding Dynamic Abnormal Grain Growth: *Phi Thanh*[1]; Daniel Worthington[2]; Cory Guebels[1]; J. P. Delplanque[1]; Joanna Groza[1]; [1]University of California Davis; [2]University of Texas

Dynamic abnormal grain growth (DAGG) is a phenomenon which produces abnormal grains during straining above a critical temperature. DAGG has been observed in molybdenum but not in iron at similar homologous temperatures (T = 0.4 TM) and strain rates (strain-rate □ 10-3 to 10-5 s-1). We compare high-temperature plastic deformation data of molybdenum and iron via creep and activation area analyses. Experiments for both materials were conducted under displacement-controlled tensile deformation. We utilize the present analyses to discuss the occurrence of DAGG in body-centered cubic metals.

4:35 PM Invited
Quantum Dynamical Effects in Thermally-Activated Dislocation Glide: Origin of the Discrepancy between Experimental and Simulated Peierls Stresses: *David Rodney*[1]; Laurent Proville[2]; [1]INP Grenoble; [2]CEA Saclay

The Peierls stress is one of the most fundamental properties of a dislocation. In BCC crystals for example, it is appreciably high, causing a strongly temperature-dependent plasticity at low temperatures. Atomic-scale simulations are known to overestimate the Peierls stress. Indeed, calculations based on electronic structure theories and interatomic potentials predict Peierls stresses two to three times larger than experimental values. We show here that this discrepancy arises from quantum effects in atomic motion (zero-point motion and tunneling) so far neglected in atomistic calculations although the experiments were performed well below the Debye temperature. We used a Quantum Transition State Theory based on Path Integrals to evaluate how quantum dynamics alter the Peierls potential of several high-Peierls stress dislocations and find that the free energy barrier against motion disappears at low temperatures for applied stresses below half the "classical" Peierls stress, thus in the experimental range.

5:05 PM
Core Properties of Screw Dislocations in Fe and W Based Materials: *Lisa Ventelon*[1]; François Willaime[1]; Emmanuel Clouet[1]; [1]CEA

A quantitative description of straight and kinked screw dislocations in iron and tungsten based materials from first principles is presented. We show that standard convergence criteria for relaxation in DFT calculations are not sufficient for a quantitative determination of the onset of glide and hence of the Peierls stress. We propose an alternative method, which consists in calculating the Peierls barrier for various strains, using a constrained minimization method. The calculation has been repeated for several crystal orientations to determine the deviation from Schmid law. We have also performed DFT simulations on the effect of rhenium on screw dislocations in tungsten using the VCA approximation, which confirm a recent prediction of the unexpected transition from symmetric to asymmetric core upon alloying. More insight into the stability of the core structure in tungsten alloys for different rhenium concentrations has been gained by looking at the response of the core polarization.

5:25 PM
Thermal Activation in BCC Metals: Linking Local Atomistic Information to the Mesoscale: *Kinshuk Srivastava*[1]; Daniel Weygand[1]; Peter Gumbsch[1]; [1]Karlsruhe Institute of Technology

High yield and flow stress in bcc metals is attributed to the large intrinsic resistance called Peierls stress experienced by the <111> screw dislocations. This is because of the non-planar nature of the dislocation core. However, at finite temperatures, the screw dislocations overcome the intrinsic energy barriers at less than the theoretical stresses due to stress-assisted thermal activation of kink-pairs on screw dislocations and their subsequent migration along them. In this work, the activation enthalpies of kink-pair nucleation are obtained directly from atomistic data incorporating the effect of local non-glide stresses and orientation dependance of the peierls barrier of the 1/2<111> screw dislocation. The effects of non-glide stresses and their importance on dislocation mobility in a three-dimensional discrete dislocation dynamics framework is presented. Simulations on micro-meter sized Tungsten pillars under bending show the effects of tension-compression asymmetry on dislocation micro-structure.

5:45 PM
Thermally Activated Motion of ½<111> Screw Dislocation in BCC Iron: *Zhiming Chen*[1]; Matous Mrovec[2]; Peter Gumbsch[2]; [1]Karlsruher Institut für Technology (KIT); [2]Fraunhofer-Institute Für Werkstoffmechanik IWM

The plastic deformation of body-centered cubic metals is governed by ½<111> screw dislocations. We perform atomistic computer simulations of the response of the ½<111> screw dislocation to various applied loadings in iron. The calculations have been made using tight-binding based bond-order potential, which also correctly accounts for magnetic effects. We study the orientation dependence of the critical resolved shear stress for pure shear stresses as well as the effect of non-glide stresses, which have a profound influence on the dislocation core structure. Our simulations show that iron possesses a significant twinning-antitwinning and tension-compression asymmetries like other bcc metals and does not follow the Schmid law. The atomistic results are transferred to macroscopic level by formulating single crystal yield criteria which can be applied in discrete dislocation dynamics or continuum approaches.