WEDNESDAY AM

Daily Personal Schedule - Wednesday - March 15

Time	Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
8:30 am				
9:00 am				
9:30 am				
10:00 am				
10:30 am				
11:00 am				
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Institute of Metals Lecture & Robert F. Mehl Medalist
"Some Generalities in the Analyses of Equilibria in Ionic Solutions"
Robert Rapp
12:00noon
Convention Center, Presidential Ballroom - Jefferson A

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LMD Luncheon
"The Aluminum Industry of the Future Partnership"
Denise Swink
12:00noon
Convention Center, Tennessee Ballroom - Nashville

12th International Symposium on Experimental Methods for Microgravity Materials Science: Session 3

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Thermodynamics & Phase Equilibria Committee

Program Organizers: Robert Schiffman, R.S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica, Bologna I-40126 Italy

Wednesday AM — Room: Memphis A
March 15, 2000 — Location: Opryland Convention Center

Session Chair: Reginald W. Smith, Queen's University, Dept. of Matls. and Metallu. Eng., Kingston K7L3N6 Canada

8:30 AM
Double-Layered Liquid Mass Under Microgravity: *Masato Takahashi*[1]; *Takamitsu Kurokawa*[1]; Kazuyuki Shimamura[1]; Itaru Jimbo[1]; [1]Tokai University, Dept. of Metallu. Eng., Hiratsuka, Kanagawa 259-1292 Japan

The study of Double-layered Liquid Mass under microgravity is undertaken in Tokai University, Japan. The DLM, in which the first spherical liquid mass is covered with the second liquid layer or shell, may be one of the promising material refining and processing procedures, where the reaction occurs at all over the interface between the two liquid phases. This can be well applied in the container-free processes under microgravity. The DLM consisting of silicone oil and water was successfully produced in a plateau tank facility and the transformation process of the DLM was carefully observed. Fundamental factors to affect the stability and the transformation of DLM will be discussed. The effect of the application of ultrasonic wave on the breakup and the reunification of DLM will also be discussed with the variation in interfacial energy of the system.

8:50 AM
Solidification Studies from the Electrostatic Levitation System at the Marshall Space Flight Center: *Jan R. Rogers*[1]; Robert W. Hyers[1]; Michael B. Robinson[1]; [1]NASA/MSFC, Mail Code SD47, Huntsville, AL 35812 USA

A containerless environment offers several advantages for studying the nucleation, solidification, and thermophysical properties of molten materials. In particular, containerless processing offers and ideal environment for studying materials in the undercooled state and highly reactive materials without contamination form crucible walls. One relatively new technology for containerless processing of materials is an electrostatic levitator (ESL). The current status of the MSFC ESL Facility is discussed along with recent measurements for time, temperature, transformation (TTT) diagrams for metallic glass-forming alloys, solidification velocity, and discusses related microstructures.

9:10 AM
The Source of Voids in Al-In Samples Processed during the LMS Mission: *J. B. Andrews*[1]; L. J. Hayes[1]; D. Downs[1]; [1]University of Alabama, Dept. of Matls. Mech. Eng., 1150 10th Ave. S., BEC 254, Birmingham, AL 35294 USA

Three aluminum-indium immiscible alloys were directionally solidified during the STS-78 Life and Microgravity Spacelab shuttle mission. These samples were part of an ongoing experiment entitled Coupled Growth in Hypermonotectics that is designed to study fundamental aspects of solidification processes in immiscible alloy systems. Postflight analysis revealed that two of the three flight samples contained small voids in some areas that were sufficient in size to locally perturb the solidification process. Great pains had been taken to minimize the likelihood of void/gas bubble formation in these samples. Steps taken included: 1) vacuum induction melting the alloys, 2) vacuum bake-out of all portions of the ampoule assembly, 3) the use of a piston and spring arrangement to compensate for thermal contraction and solidification shrinkage, and 4) loading and sealing the ampoule under vacuum. The presentation will address the results of tests carried out in an attempt to identify the source of these voids and modifications underway to help control this difficulty in the future.

9:30 AM
Growth Velocity-Interface Figuration Relationships in Undercooled Semiconductors: *T. Aoyama*[1]; K. Kuribayashi[1]; [1]The Institute of Space and Astronautical Science, 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan

A crystal growth behavior of a semiconducting material from deeply undercooled melt is expected to be different from that of a metal. Several investigators have reported the transition from lateral to continuous growth in solidification of undercooled pure Ge and Ge-based alloys. However, there are many discrepancy between their results with respects to the critical undercooling for the transition. In the present experiment, pure Si, Ge and their alloys were undercooled by an electromagnetic levitator combined with a laser heating facility. The crystal growth velocities were measured as a function of undercooling by means of two photodiodes and a high-speed video camera. The transition of the dendrite growth behavior was observed from the change of the solid-liquid interface figuration.

9:50 AM Break

10:10 AM
Effect of Buffer and Salt on the Rate of Nucleation of Protein Crystals: *James K. Baird*[1]; [1]University of Alabama, Dept. of Chem., Huntsville, AL 35899 USA

Protein crystals nucleate from pH buffered aqueous solutions of strong electrolytes. In aqueous solution, protein molecules exist as highly charged macro-ions. The first step in the nucleation mechanism is the formation of a dimer from two of these macro-ions. To prevent the dimer and its successor nuclei from building an excessive charge, we propose a nucleation mechanism which includes donation of H^+ to the buffer. We suggest that the ions contributed by the dissolved salt produce Debye-Huckel plasma screening of these charged species, which serves to accelerate their rate of agglomeration. We modify standard nucleation theory to take these effects into account. This research was sponsored by the National Institute of General Medical Sciences of the National Institutes of Health through grant 1R15 GM51018 and in part by the Naval Research Laboratory in Washington, DC under grant N00014-94-1-GO16 from the Office of Naval Research.

10:30 AM
The Directional Solidification of Hypermonotectics to Determine the Effect of Convective Flow on Interface Stability: *J. D.*

Barnes[1]; J. B. Andrews[1]; [1]University of Alabama, Dept. of Matls. and Mech. Eng., 1150 10th Ave., SBEC 254, Birmingham, AL 35294 USA

The transparent-metal-analog system succinonitrile-glycerol was used to directly observe and determine the effect of convective flow on interface stability. Hypermonotectic alloys were directionally solidified using a temperature-gradient-stage microscope. Samples were first processed horizontally in order to minimize flow. No flow stability limits were determined by systematically increasing the growth rates to promote instability. Vertical processing orientations were then used in order to determine the flow-modified stability limits. The sample thickness was systematically increased in order to decrease the amount of damping on the fluid flow. Different hypermonotectic compositions were studied so that a comparison could be made on the influence of both composition and convective flow on interface stability.

10:50 AM

Effects of End-Wall Vibration on Oscillatory Thermocapillary Flow: *J. Bhowmick*[1]; Q. Kou[1]; A. Anilkumar[1]; R. N. Grugel[2]; [1]Vanderbilt University, Ctr. for Microgravity Rsch. and Appls., Nashville, TN 37235 USA; [2]University Space Research Association, Huntsville, AL 35812 USA

Our previous flat zone experiments with NaNO3 revealed that steady thermocapillary flow (TC flow) can be balanced/offset by the controlled surface streaming flow (CSS flow), induced by end-wall vibration. In the current experiments, we are examining the effects of surface streaming flow on steadying/stabilizing oscillatory thermocapillary flow. To this effect, we have set up a controlled NaNO3 half-zone experiment, where the processing parameters like zone dimensions and temperature gradients can be easily varied to achieve oscillatory TC flow. In the present paper, we discuss the thermal signature of the TC flow, and how it is affected by imposition of CSS flow. The results will also include a comparison of the microstructure of a NaNO3-Ba(NO3)2 eutectic, processed under oscillatory TC conditions, with and without imposed CSS flow.

11:10 AM

Effect of Gravity on Directional Equiaxed Solidification of a Refined AL-3.5WT%NI Alloy: *M. D. Dupouy*[1]; D. Camel[1]; [1]DEM/SPCM, CEA-Grenoble, 17 Rue Des Martyrs, Grenoble, Cedex 38054 France

The formation of equiaxed microstructures in castings is well known to be strongly influenced by convection and sedimentation. Following our previous experiments performed during EUROMIR95 and LMS96 missions on the Columnar to Equiaxed Transition in refined Al-4wt%Cu alloys [1-3], a new series of comparative ground and space experiments has been performed in order to analyse multigrain equiaxed solidification. A refined Al-3.5wt%Ni alloy, with a smalll solidification interval and a high eutectic fraction, has been chosen in order to directly reveal the morphology of the growing solid frozen at an early stage by the eutectic transformation of the remaining liquid. Samples of this alloy were directionally solidified with several velocity steps, respectively in the AGHF furnace during STS-95 mission (Nov. 98, AGHF6 experiment), and in TITUS during MIR-PERSEUS (May-June 99, collaboration with ACCESS). Comparative ground experiments were performed vertically upwards. We present here the preliminary results of the experiment AGHF6: Space samples show a homogeneous dendritic equiaxed structure with increasing grain sizes for decreasing solidification rates. By contrast, a transition to mixed columnar-equiaxed and then purely columnar structures is observed in ground samples. Morphological parameters of the microstructures are determined by Image Analysis, and the influence of gravity driven convection on these morphologies is discussed in relation with the radial macrosegregation observed on the ground. [1] M.D. Dupouy, D. Camel, F. Botalla, J. Abadie and J.J. Favier, Proc. 8th Int. Conf. on Modeling of Casting, Welding and Advanced Solidification Processes, San Diego 1998, p.415. [2] M.D. Dupouy, D. Camel, F. Botalla, J. Abadie, J.J. Favier, Microgr. Sci. Techn. XI/1 (1998), p.1. [3] M.D. Dupouy, D. Camel, J.E. Mazille and I. Hugon, Proc. 3rd Int. Conf. Solidification And Gravity, Miskolc, Hungary, April 26-29, 1999.

Alumina and Bauxite: Bayer Process Development

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Vito Cedro, Alcoa World Alumina, Pittsburgh, PA 15219 USA; Joe Anjier, Queensland Alumina Limited, Gladstone, Queensland 4680 Australia

Wednesday AM Room: Jefferson B
March 15, 2000 Location: Opryland Convention Center

Session Chair: Ashwadama Pasupulatey, Alcoa World Alumina, Point Comfort, TX 77971 USA

8:30 AM Invited

Organics Removal from Bayer Liquor: *Suresh K. Bhargava*[1]; [1]RMIT University, Dept. of Appl. Chem., GPO Box 2476V, Melbourne, Victoria 3001 Australia

The Bayer process for the production of alumina from bauxite is used to produce ~95% of the world's smelting grade alumina. The initial reaction in this process is the dissolution of aluminum hydroxide Al(OH)3 and AlO(OH) in hot sodium hydroxide to produce sodium aluminate Na+Al(OH)-4. After removal of insoluble impurities (mostly quartz and fine iron oxides) the aluminum hydroxide {gibbsite-Al(OH)3}. This is subsequently calcined (10000°C) to produce smelting grade alumina Al2O3). The Bayer process is cyclic and has only a very small bleed of solution with the insoluble residues. Consequently, any organic substance, which is soluble in hot alkali, will accumulate in the Bayer process liquor to reach a very high steady-state concentration. For example, starting with a pure Bayer liquor (sodium hydroxide/aluminate), each passage through the bauxite digestion step will result in solution containing ~0.4g/L of the total organic carbon (TOC). However, the steady-state TOC concentration is ~30g/L. This especially trues for Western Australian alumina refineries, which process very low-grade bauxite. Such impurities may be humic and fulvic acids and salts, tannins and other polyphenolic salts, low rank coal precursors, and their degradation products. These have been classified according to their molecular weight distribution. The presence of these dissolved organics pollutants (or TOC) causes major processing problems and production inefficiencies including: Reduced aluminum hydroxide yield, impure aluminum hydroxide, colored aluminum hydrate, interference with flocculation of iron oxide residues, foaming of process liquor and increased liquor viscosity. Many processes have been devised to reduce the concentration of organics from the Bayer liquor. However, very few of these suggested processes have been put into commercial use since they are generally capital expansive and risky. One successful process that is in use at certain alumina refineries is "liquor burning". This is based upon technology licensed from a Japanese company-Showa. However, this is also capital intensive, expansive and associated with other air pollution problems. This lecture will discuss various issues related to limitation caused by organics on the Bayer process production and probable solutions to reduce or remove these organic pollutants from the aluminate liquor of Bayer process.

9:15 AM

A Year of Operation of the SLC Process: *Benny Erik Raahauge*[1]; Jens Fenger[1]; Hélène Boily[2]; José Pulpeiro[3]; Martín Gayol[3]; [1]FFE Minerals DK A/S, MIA, 77 Vigerslev Allé, Copenhagen DK 2500 Denmark; [2]Alcan International Limited, Banbury Lab., Southam Rd., Banbury, Oxfordshire OX16 7SP UK; [3]Alcoa Europe, Apartado de Correos 71, San Ciprián (Lugo) ES 27890

The first Solid-Liquid Calcination plant for destruction of organics in Bayer plant liquor was commissioned in 1999 at Alcoa Europe's San Ciprián plant in Spain. The paper compares the first year of operational experience with the design criteria and reports on the testing of alumina dust from a calciner as a feed component.

9:40 AM

Some Capabilities of Removal of Organic Substances from Australian Bayer Process Liquors: Yury A. Zaytsev[2]; Valery P. Lankin[1]; Vadim A. Lipin[1]; *Michael B. Stoljar*[3]; [1]Russian National Aluminum-Magnesium Institute, 86 Sredny Pr., St. Petersburg 199026 Russia; [2]St. Petersburg Mining Institute, 2, 21 Line, St. Petersburg 199026 Russia; [3]Nikolaev Alumina Plant, Nikolaev 327054 Ukraine

The organic substances have an impact on different operations in Bayer process substantially. It arriving at the process from bauxites is interacted with alkaline liquors, and still they repeated circulating in Bayer process during long time is stockpiled in spent liquor. In consequence of repeated circulation the organic substances are subjected to great transformations. The formed new organic substances differ from initial bauxite organic in chemical constitution. The elevated content of organic substances is the Darling Range bauxite distinctive feature. The extraction from one ton of bauxite of organic substances at green liquor averages between 1.5 kg and 2 kg. The fraction distribution of organic substances in evaporated spent liquor by gravimetric method was determined. An alkaline earth compounds are attractive as sorbent of organic substances and re-usable. The possibility of application number reasonable magnesium compounds for purification of Bayer process evaporated liquors was considered. The influence of temperature, duration of the treatment, concentration of the putting magnesium compounds on results of the sorption organic substances and losses of alumina were studied. It was established that amounts of putting sorbent have great impact on the sorption, and at "LGI Process" the losses of alumina are minimum. Some feasible variations of regeneration and repeated application are pursued further.

10:05 AM Break

10:25 AM

The Adsorption of Sodium Oxalate Stabilizers to the Surface of Gibbsite (a Bayer Process Solid) under High Ionic, High pH Strength Conditions: *Andrew Robert Hind*[1]; Suresh Bhargava[1]; [1]R.M.I.T., Dept. of Appl. Chem., GPO Box 2476V, Melbourne, Victoria 3001 Australia

Using a recently developed FTIR/ATR method for the in situ investigation of solid surfaces in highly alkaline, high ionic strength, aqueous media, the adsorption of a series of surface active quaternary ammonium (QA) compounds to the surface of gibbsite has been investigated. The technique involves the use of a finely ground gibbsite, combined with an appropriate adsorption matrix, and permits the in situ investigation of interfacial phenomena in high ionic strength, highly alkaline (pH 12-13), aqueous media. Spectroscopic results show the formation of surfactant aggregate (or hemimicellar) clusters on the surface of gibbsite (under the high ionic strength, high pH conditions used), and suggest adsorption may occur in the order C16<C12<C14. It is anticipated that this method will allow the acquisition of "dose response" curves for the aforementioned QAs on gibbsite, whilst also leading to the in situ investigation of the surface of gibbsite (and other Bayer process solids) in synthetic and process Bayer liquors (high ionic strength, extremely alkaline media).

10:50 AM

A New Technology of Slurry Preparation for Bayer Process: *Zhang Chengzhong*[1]; *Hao Xiangdong*[1]; Li Ming[1]; *Peng Zhihong*[2]; Liu Guihua[1]; Li Xiaobin[2]; [1]Shanxi Aluminum Plant, Directors Office, Hejin, Shanxi 043300 China; [2]Central South University of Technology, Dept. of Metallu., Changsha, Hunan 410083 China

Slurry in Bayer Process, prepared by the traditional one stage closed-circuit grinding with a ball mill and a spiral classifier, cannot easily meet the demands of particle size composition in tube digestion. A new technology, with a hydraulic cyclone instead of the previous spiral classifier, can prepare qualified slurry. This new technology is different from other grinding flow sheets in application at present. It can increase the output of ball mill, decrease the energy consumption and raise the target of technology and economy.

11:15 AM

Tube Digestion Technology for Treating Diaspore Bauxite in China: *Wang Xing Li*[1]; Pei Kai Song[1]; Zi Jian Lu[1]; [1]China Great Wall Aluminum Corporation, Shangjie, Zhenzhou, Henan 450041 China

Based on the property of bauxite in Henan Province of China, the effect of alumina digestion efficiency and lime addition after digestion on scaling of bauxite containing silicon and the abrasion to equipment and tubes were studied and industrially tested. The first set of industrial tube digestion equipment of China, which can treat 300 m3/h original diaspore bauxite pulp, has been set up in the China Great Wall Aluminum Corporation. The main part of the equipment was imported from Germany. The half a year of industrial production with it shows the objectives for energy-saving and consumption-reducing have been attained.

11:40 AM

Application of Thoroughly Carbonizing Pregnant Liquor to Soda-Sintering Process: *Shangguan Zheng*[1]; Yang Zhongyu[1]; Hu Shenxing[2]; Luo An[2]; [1]Central South University of Technology, Changsha, Hunan 410083 PRC; [2]Shandong Aluminum Corporation, Zibo, Shandong 255052 PRC

Thoroughly carbonizing pregnant liquor from leaching sinter obtained by sintering mixture of soda and bauxite with high A/S, namely soda-sintering process, can get raw Al(OH)3 with high silica content. Then the raw Al(OH)3 is processed by low-temperature Bayer process producing sandy alumina[1]. This new technology makes soda-sintering process operated at high concentrations and can process bauxite with low silica content, decreasing production cost greatly.

Aluminum Reduction Technology: Environmental

Sponsored by: Light Metals Division, Aluminum Committee

Program Organizers: John Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland, New Zealand; Georges J. Kipouros, Dalhousie University, Department of Mining and Metallurgical Engineering, Halifax, NS B3J2X4 Canada

Wednesday AM Room: Sewanee
March 15, 2000 Location: Opryland Convention Center

Session Chair: Halvor Kvande, Hydro Aluminum Metal Products, Stabekk N-1321 Norway

8:30 AM Invited

Changing Knowledge and Practices Towards Minimising Fluoride and Sulphur Emissions from Aluminium Reduction Cells: *Margaret M. Hyland*[1]; Barry J. Welch[1]; James B. Metson[2]; [1]University of Auckland, Dept. of Chem. and Matls. Eng., Private Bag, Auckland 92019 New Zealand; [2]University of Auckland, Dept. of Chem., Private Bag, Auckland 92019 New Zealand

Despite continuing and substantial improvements in the capture and treatment of fluoride emissions from smelting cells, the industry continues to be faced with new environmental challenges. International acceptance of protocols to reduce the release of greenhouse gases has again raised the need for the aluminium industry to closely examine the sources, nature and treatment of all smelting emissions Changing practice, such as use of more acidic electrolyte coupled with point fed cell design has also focused attention on the design and operation of dry scrubbers. The short term solutions have been to increase the recycle in the scrubber and to use aluminas with higher fluoride capture capacities (higher BET surface area and/or alpha content). Recent advancements in the understanding of the reactions occurring in the scrubber raises questions over the efficacy of these approaches and suggests a different balance between optimal reactor design and alumina properties. Furthermore, an intimate knowledge of the reactions which generate HF (e.g. the role of residual hydrogen in anodes) may result in innovative approaches in the minimisation of emissions. With the substantial advances in the capture of fluoride emissions, attention has turned to other cell gases. Much of the anode

sulfur is released as COS, which is not entirely oxidised to SO2 before its release to the environment. COS is a critical gas in the atmospheric sulfur cycle, owing to its transportability into the stratosphere. The generation of this gas is not well understood, nor is its fate in the dry scrubber. Ensuring the complete oxidation of the sulfur gases released from the anode is one strategy to ensure COS is not emitted. The industry has seen a dramatic reduction in anode effect frequency in the last two decades but we need to pause and examine whether modern knowledge can be applied towards reducing them further.

9:00 AM

Environmental Improvements in a Soderberg Potline: *Nancy C. Holt*[1]; Marit S. Aalbu[2]; Kirsten L. Bolstad[1]; T. Foosnaes[1]; Morten Karlsen[1]; Victoria Kielland[1]; H. Kvande[1]; [1]Hydro Aluminium Metal Products, Tech. Centre Årdal, Øvre Årdal N-6882 Norway; [2]Hydro Aluminium, Ardal Smelter, Norway

A traditional Soderberg potline usually has higher roof emissions than a prebake line. Much work has been made over the last two decades and considerable reduction of roof emissions and improvements of the internal working atmosphere have been obtained. During 1998 the Soderberg potlines in Årdal were converted to point feeder technology, and a new study was initiated to analyze and identify factors with further improvement potential. This study consists of analysis of HF and dust emissions data, potroom operating routines, ventilation conditions, alumina quality and technical elements such as feeding. It is found that technological elements and changes of raw materials are the more important reasons for periods of high roof emissions of the potline. Average HF and dust emissions from the pots with point feeding are not higher compared with the ones with side breakers. It is observed that filling and fluidizing of alumina silos on the pots may cause dusting, and that a combination of alumina characteristics and certain climatic conditions can be a source for seasonal variations in roof emissions. Potroom ventilation does not contribute to unusually high emissions, however, conditions at the center of the potroom may not be optimal. CFD simulations resulted in suggestions for improved working atmosphere.

9:25 AM

The Surface Chemistry of Secondary Alumina from the Dry Scrubbing Process: *Alistair Ross Gillespie*[1]; Margaret M. Hyland[2]; James B. Metson[3]; [1]Comalco Research and Technical Support, Alumina Tech., 15 Edgars Rd., Thomastown, Vic 3074 Australia; [2]University of Auckland, Dept. of Chem. and Matls. Eng., Private Bag, Auckland 92019 New Zealand; [3]University of Auckland, Dept. Of Chem., Private Bag, Auckland 92019 New Zealand

Dry scrubbers at modern aluminium smelters prevent discharge of particulate material and gaseous hydrogen fluoride by returning virtually all of the collected material to the electrolytic cell. As such, they have an important impact on the smelter's materials balance. Return of fluoride is beneficial as it reduces the smelter's requirement to add aluminium fluoride to maintain constant bath ratio. On the other hand, return of hydrogen in the form of water, HF, or chemically bound hydroxyl, is not beneficial as this may result in release or regeneration of HF. Data presented here show fluoride in dry scrubbed alumina to be bound as a hydrated aluminium-hydroxy-fluoride phase of variable stoichiometry. Water of hydration is relatively weakly bound and could participate in hydrolysis reactions in the fume, during cell feeding. Hydroxyl ions are more strongly bound and may participate in hydrolysis reactions with the electrolyte, or in self-hydrolysis of the aluminium hydroxy-fluoride.

9:50 AM

SO_2 Emission Control in the Aluminum Industry: *Svein Ole Strømmen*[1]; E. Bjornstad[1]; G. Wedde[1]; [1]ABB Environmental Norway, Flakt Div. for the Aluminium Industry, Postboks 6260 Etterstad, Oslo 0603 Norway

SO2 emissions from the aluminum industry are modest on a worldwide and national scale. Large modern smelters using high-sulfur petroleum coke in anodes and smaller smelters with topographical unfavorable conditions may, however, be significant sources locally. This has lead environmental regulators to again review local legislation. Currently SO2 emission legislation is in force in Scandinavia and in some areas in the US. The ongoing review of SO2 emissions might lead to stricter legislation forcing smelters outside these areas to install SO2 control systems. The electrolysis represents approximately 80% of the released SO2 for prebake smelters and approximately 95% for Söderberg smelters. The paper is addressing current SO2 removal legislation as well as current removal technologies applied within the Aluminum industry with focus on investment and operational cost per ton SO2 removed.

10:15 AM Break

10:25 AM

Understanding and Controlling HF Fugitive Emissions through Continuous HF Monitoring and Air Velocity Characterisation in Reduction Lines: *Elaine Yee-Leng Sum*[1]; Chris Cleary[2]; Tseng T. Khoo[3]; [1]Comalco Research & Technical Support, 15 Edgars Rd., Thomastown, Victoria 3074 Australia; [2]Comalco Aluminium Limited, Boyne Smelters Reduction Line 3, Handley Dr., Boyne Island, Queensland 4680 Australia; [3]Boyne Smelters Ltd, Reduction Lines 1&2, Handley Drive, Boyne Island, Queensland 4680 Australia

HF fugative emissions were studied at Boyne Smelters Ltd., an aluminium smelter operating with two different technologies. Fugative emissions from reduction cells, anode butts, bath sows, Pacman skips and open metal crucibles were characterised by an open path FITR spectrometer. The effects of bath chemistry, draught and cell design on fugative cell emissions were investigated. Real time monitoring by a continuous HF gas analyser provided a useful tool for improving work practises and reducing fugative emissions. The air velocity profile in the reduction line roof was characterised using an array of vane anemometers. The diurnal variation in the roof air velocity underlined the importance of a proper sampling proceedure, if intermittent monitoring is employed. A continuous, open path, optical air velocity monitoring system was successfully trailed. A new approach using an ultrasonic anemometer was found to be suitable for monitoring roof air velocity continuously.

10:50 AM

Perfluorocarbon (PFC) Generation during Primary Aluminum Production: *Jerry Marks*[1]; R. Roberts[1]; V. Bakshi[1]; E. Dolin[1]; [1]Alcoa Technical Center, 100 Tech. Dr., Alcoa Center, PA 15069-0001 USA

The primary aluminum industry is continually working to improve production efficiency and enhance environmental performance. As part of EPA's Voluntary Aluminum Industrial Partnership (VAIP) Program, eleven U.S. primary producers are focusing on reducing the duration and frequency of anode effects (AEs), which reduce aluminum current efficiency and generate two perfluorocarbons (PFCs), CF4 and C2F6. PFCs effectively trap heat in the atmosphere, contributing to the greenhouse effect. To better understand PFC emissions and key factors influencing their generation, VAIP sponsored a second, data-intensive measurement program at six aluminum smelters (the results of the first round of measurements were reported at the 1998 TMS meeting in San Antonio and appeared in Light Metals 1998—277-285). At each smelter, PFCs were measured from the potroom exhaust ducts using a continuous real-time mass spectrometer. Fugitive emissions were sampled from roof exhausts and measured by FTIR spectrometry. Emissions were related to facility operational parameters such as AE frequency, AE duration, and AE over-voltage. The real-time measurement capability provides the ability to generate highly time resolved emissions profiles of individual anode effects. This information gives new insights into the factors influencing emissions in addition to estimating overall smelter emissions. Several smelters provided data on the voltage profile of individual anode effects, which has shown correlation to emissions in both bench-scale laboratory studies at MIT and other measurements. The paper reviews the data, provides recommendations for improving PFC emissions predictability and suggests possible means for reducing these emissions.

11:15 AM

Intercomparison of Three Separate Technologies for the Measurement of HF Stack Emissions from the HAW Primary Aluminium Smelter: *Rudolf Heger*[1]; Andre Abbe[1]; John T. Pisano[2]; Matthias Franz[3]; [1]Hamburger Aluminuim-Werk GmbH, Dradenauer Hauptdeich 15, Hamburg 21129 Germany; [2]Unisearch Associates Inc., 96 Bradwick Dr., Concord, Onatrio L4K1K8 Canada; [3]Pier Enterprises GmbH & Co. KG, Voltastrasse 7, Hattersheim 65795 Germany

Tests were conducted at the HAW smelter between three separate technologies with respect to HF measurements. The standard VDI reference measurement procedure for gaseous inorganic fluorine compounds (VDI-2286 Part 1) was used to compare Tunable Diode Laser Spectroscopy (TDLAS) with existing potentiometric based instrumentation. A total number of 30 tests were conducted between all three measurement methods and in all but 5 the TDLAS instrument was in better agreement to the standard method. The TDLAS response also closely tracked the many manual manipulations done to the scrubbing process during the evaluation period and had response times as fast as one second. This fast and accurate response would likely enhance scrubber efficiency control. The TDLAS instrumentation used, provides an effective alternative to other technologies with respect to measuring HF emissions as the overall average deviation between the TDLAS instrumentation and the standard method was around 7% for the 30 tests.

Carbon Technology: Materials Properties and Modeling

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Morten Sorlie, Elkem ASA Research, Vaagsbygd, Kristiansand N-4675 Norway; Christian Dreyer, Aluminium Pechiney, St Jean De Maurienne 73303 France

Wednesday AM Room: Knoxville A
March 15, 2000 Location: Opryland Convention Center

Session Chair: Frank Hiltmann, SGL Carbon Group, Frankfurt/M D-65933 Germany

8:30 AM
The Use of Petrographic Techniques for Evaluation of Raw Material and Process Changes in an Aluminum Smelter: *J. Anthony Ross*[1]; [1]Century Aluminum, Primary Products Div., P.O. Box 98, Ravenswood, WV 26164 USA

The value of routine use of common petrographic technique in support of a process control system in an aluminum smelter is presented. Included is a brief review of the techniques used in petrography and how they proved valuable in defining a petroleum coke quality change, in a case study at Century Aluminum's Smelter. The impact on green-anode paste characteristics from conversion of solid pitch usage to liquid pitch is also presented. Although there have been significant developments in recent years in the areas of microscopic imaging and advanced pore structure/volume measurements of petroleum coke, the basic use of microscopic techniques in most smelter laboratories is minimal. A cursory review of smelter process materials will also show various characteristics valuable to the evaluation of process changes and routine operations.

9:00 AM
The Relation of Reflectance to the Degree of Calcination of Coal Tar Pitch: *J. Anthony Ross*[1]; Ray Patalsky[2]; [1]Century Aluminum of WV, Primary Products Div., P.O. Box 98, Ravenswood, WV 26164 USA; [2]Coal Petrographic Associates Inc., 3100 Braun Ave., Murrysville, PA 15668 USA

Coal rank, or degree of maturation of coal, influences the behavior of coal in various mechanical and thermal processes. Vitrinite reflectance techniques have been used successfully to characterize the degree of maturity of coal. The adaptation of these techniques is proposed for the indirect determination of the calcination level of coke from anode binder pitch. The correlation of the maximum reflectance of pitch coke with calcination temperature and graphite crystalline size (Lc), indicate that coal tar pitch can be effective as a means for indirectly determining the degree of baking of anodes. Green petroleum cokes, presently used for indirect measurements, have varying amounts of isotropic and anisotropic structures which can differ in Lc values, after calcination. Pitch coke is less variable in microstructure and presents a more reliable means for the determination of Lc and reflectivity measurements.

9:25 AM
Evolution of Thermal, Electrical and Mechanical Properties of Graphitised Cathode Blocks for Aluminium Electrolysis Cells with Temperature: *Bénédicte Allard*[1]; J. M. Dreyfus[1]; M. Lenclud[1]; [1]Carbone Savoie, Lab. de Recherche et d'Essais, 30, Rue Louis Jouvet, BP16, Venissieux 69631 France

In order to predict the behaviour of cathodic blocks in aluminium electrolysis pots, measurements of their properties should be made under conditions as close as possible to the real conditions. Tests have been developed to characterize thermal, electrical and mechanical properties versus temperature, and concerning electrical resistivity also under electrolysis. Characterization of various grades of graphite blocks has been performed, through thermal conductivity, electrical resistivity, flexural strength and fracture energy measurements. The evolution of these characteristics between room temperature and 1000°C is studied and compared to the carbon blocks one. Values at 1000°C can be used for pot modelization.

9:50 AM
Thermo-Electro-Mechanical Modeling of the Contact between Steel and Carbon Cylinders Using the Finite Element Method: *Daniel Richard*[1]; M. Fafard[1]; R. Lacroix[1]; P. Clery[2]; Y. Maltais[2]; [1]Laval University, Chem. Eng./GIREF, Pouliot Bldg., Ste-Foy, Quebec G1K7P4 Canada; [2]Alcoa-Lauralco, 1Boul. Des Sources, Deschambault, Quebec GOA1SO Canada

The Hall-Héroult aluminum reduction process requires an enormous amount of electrical power. Energy saving strategies lead to the analysis of the electrical losses at the cast iron/carbon interfaces in the anodic and cathodic assemblies. Numerous experimental studies were made in the past years, both in situ and in laboratories. However, they did not provide any practical means of predicting the interfacial electrical contact resistance. Here, an indirectly coupled thermo-electro-mechanical finite element model of the 1992 experiment of M. Sørlie [1] was built using the commercial code ANSYS. This model was used to obtain a semi-empirical constitutive equation relating nominal contact pressure and temperature to electrical contact resistance. Agreement with experimental data was found to be excellent. The difficulty of predicting contact resistance with existing theories is discussed. Application to stub hole and collector bar slot design is also discussed.

Cast Shop Technology: Melt Quality and Foundry

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Paul Crepeau, General Motors Corporation, GM Powertrain Group, Pontiac, MI 48340-2920 USA; James N. O'Donnell, Commonwealth Aluminum Corporation, Department of Engineering, Louisville, KY 40202-2823 USA

Wednesday AM Room: Mississippi
March 15, 2000 Location: Opryland Convention Center

Session Chair: Elwin L. Rooy, Rooy & Associates, Aurora, OH 44202-8240 USA

8:30 AM Introductory Remarks

8:35 AM
Laboratory and Industrial Validation of an Ultrasonic Sensor for Cleanliness Measurement in Liquid Metals: *Iain D. Sommerville*[1]; [1]University of Toronto, Metallu. and Matls. Sci., 184 College St., Toronto, Ontario M5S3E4 Canada

An ultrasonic sensor for cleanliness measurement in liquid metals has been developed, and is in the process of commercialization for

aluminum melts. In this paper, attention is focussed on practical aspects such as calibration procedure, choice of gate width and location, time setting, the ease of handling and movement of the probes, and the cheapness and ease of operation in the casthouse. Particular attention has been paid to the signal processing and the conversion of the information contained in the CRT trace to a user-friendly format for quick and easy assimilation. The relative roles of counting and attenuation in assessing melt cleanliness in the dynamic situation of casting are explained, and the ability to conduct sampling simultaneously in several locations, and the potential for various probe arrangements are also discussed. Several other ancillary procedures which help to validate the measurements are also described.

9:00 AM

Measurements of the Hydrogen Concentration in Cast Alloys: *Jo Verwimp*[1]; F. De Schutter[1]; G. Mertens[2]; J. Vits[3]; [1]VITO, Process Tech., Boeretang 200, Mol 2400 Belgium; [2]Katholieke Hogeschool Kempen, Campus HIKempen, Kleinhoefstraat 4, Geel 2440 Belgium; [3]Hayes Lemmerz Belgie BVBA, Lage Weg 392, Hoboken 2660 Belgium

Hydrogen is the only important gas in liquid aluminum and can have severe detrimental effect on the properties of the cast product. In order to measure the hydrogen content in liquid aluminum, several tools for on-line measurements have been developed and are used in production facilities. However, a straight correlation between these measurements and the final product is not guaranteed. Moreover, a distinction between pores originating from hydrogen or shrinkage may be important to adjust the production process. In order to measure the hydrogen concentration in solidified aluminum, methods such as hydrogen extraction techniques have been developed. This paper describes the use of such a system (Ströhlein H-MAT 2020) in order to evaluate the hydrogen concentration in cast aluminum wheels. The obtained hydrogen data are compared with calculated solidification profiles and with test results of mechanical and microscopic investigations. The results show a correlation between the hydrogen concentration and the solidification profiles.

9:25 AM

Achieving Low Hydrogen Content in High Purity Aluminum: *Leonhard Heusler*[1]; Werner Kapellner[2]; Ronald Becher[2]; *Jean-Claude R. Terrier*[3]; [1]VAW Aluminium AG, R&D, Cast Shop Tech., P.O. Box 2468, Bonn 53014 Germany; [2]VAW Highpural GmbH, P.O. Box 100664, Grevenbroich 41490 Germany; [3]Péchiney Aluminium Engineering, Alpur and Casthouse Equip., Centr'Alp-B.P. 24, Voreppe 38341 France

Due to the steady increase of the customers' quality requirements the producers of high purity aluminum rolling ingots have to spend much effort on reducing the hydrogen content. By optimization of the melting and casting process, VAW highpural, a german producer of high purity aluminum, already achieved low hydrogen levels. In order to meet even higher requirements as for example for the production of high voltage capacitor foils a degassing system (ALPUR TS 35) was installed additionally. With respect to possible melt contamination with oxides, major selection criteria were a good sealing of the system against ambient air and an additional calming chamber within the box. For the evaluation of the system and the optimization of the degassing parameters, a systematic program of casting trials was carried out using AlSCAN and NOTORP measurements up- and downstream to assess the degassing efficiency, whereas the capability of inclusion removal was evaluated by means of LiMCA and PoDFA measurements. In general, a reduction of the hydrogen content from approx. 0.17 ml/100g Al (AlSCAN) to 0.10 ml/100g Al was achieved while the inclusion content in some cases slightly decreased.

9:50 AM

Using the Prefil-Footprinter Instrument: *Alain A. Simard*[1]; Jasmin D. Proulx[1]; Dany Veillette[1]; François Dallaire[1]; Paul Rochette[1]; [1]ABB-Bomem Inc., 450 St-Jean-Baptiste, Quebec City, Quebec G2E5S5 Canada

Benchmarking is an important aspect of today's Total Quality Management (TQM) and is a requirement for world-class corporations. Quality measurement is also required by standardization programs such as ISO9000 and QS9000. As liquid metal quality is an essential aspect of the quality of final products from casting foundries and casthouses, it is important that world-class operations accurately benchmark metal quality. For decades, and still today, metal cleanliness has been widely monitored using the metallographic analysis of solidified samples. These samples may or may not be filtered. With filtering, the required techniques are time consuming and resource-intensive — analysis results of grab samples are obtained off-line only after significant delays. If no filtering is used, the results suffer from low sensitivity and human interpretation. The following paper proposes a new way of expressing cleanliness measurements of liquid aluminum, and provides benchmark references for common castings and wrought alloys. The paper also provides the measurement conditions and the validity of these benchmarks, and gives a description of the instrument used to generate cleanliness measurements directly at-line.

10:15 AM Break

10:20 AM

Metal Quality Comparison of Alcan Compact Degasser and SNIF at Alcoa Mt. Holly Casthouse: *D. C. Chesonis*[1]; E. Elder[2]; R. O. Wood[2]; D. H. DeYoung[1]; [1]Alcoa Inc., Ingot and Solidification, Alcoa Tech. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA; [2]Alcoa Inc., Alcoa Mt. Holly, P.O. Box 1000, Goose Creek, SC 29445 USA

LiMCA and Alscan measurements of inclusion and hydrogen concentrations were performed at the Mount Holly Casthouse to compare the metal quality obtained with an Alcan Compact Degasser to that obtained with a SNIF degasser. The measurements were conducted at two casting pits that are identical except for the degassers. The in-line metal treatment at one pit has a two stage, R-140 SNIF degassing unit, while the other pit uses a six rotor Alcan Compact Degasser. Identical ceramic foam filters are used downstream of the degassers. Data was obtained from 11 casts of 6xxx alloys over a four day period. This paper will summarize the data and will provide a statistical comparison between the two degassing units. LiMCA inclusion concentrations entering the degassers, inclusion concentrations after the ceramic foam filters, hydrogen concentrations at the exit of the degassers, and the hydrogen removal efficiencies will be compared.

10:45 AM

Mechanical Properties and Heat Treatment of A357 Foundry Alloys: *B. Closset*[1]; S. Khan[2]; [1]Timminco S.A., 44 Chemin Petite-Boissiere, Geneve CH-1208; [2]Shellcast Foundries Inc., Montreal North, Quebec H1G5L4 Canada

Several types of A357 alloy containing different amounts of strontium, berillyum, titanium and zinc were investigated. Test bars were cast in ceramic shell molds to measure the mechanical properties of the different A357 alloys compositions. After heat treatment the microstructures and the mechanical properties were studied. The properties obtained after two new heat treatment cycles were compared to the properties measured after a standard heat treatment cycle. It was shown that a small amount of berillyum (0.040%) added in combination to strontium modification increased significantly the mechanical properties (tensil strength, yield strength, elongation). The effect of the heat treatment on the Al-Si eutectic morphology has also been studied.

11:10 AM

Alloy Refinement Using Computational Thermodynamics: *Ravi Vijayaraghavan*[1]; Jacob W. Zindel[1]; John E. Allison[1]; [1]Ford Motor Company, Ford Rsch. Labs., MD-2122 SRL, Dearborn, MI 48124-2053 USA

The significant volumes of cast components required by the automotive industry provides an important opportunity for alloy refinement. One tool which is available for more efficient alloy design is computational thermodynamics. In this study we have used ThermoCalc, a software tool to calculate thermodynamic phase equilibria in complex multicomponent systems. The objective of this work is to study and demonstrate the potential of phase diagram calculation in refinement of cast aluminum alloys used in engine blocks and heads. Non-equilibrium solidification of a multicomponent 319 aluminum alloy was simulated under the Scheil condition using ThermoCalc. Results were quickly obtained, predicting evolution of fraction solid, latent heat and phase formation. Validation of ThermoCalc predic-

tions with quench experiments will be presented in this talk. The effect of alloying elements on evolution of the iron intermetallics, β-FeSi and script phase, will be summarized. A major goal of this research is the development of a method for systematic alloy refinement using computational thermodynamics.

11:35 AM

Die Soldering in Aluminum Die Casting: Qingyou Han[1]; *Srinath Viswanathan*[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics, Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

Two types of tests, "dipping" tests and "dip-coating" tests, were carried out on small steel cylinders using pure aluminum and 380 alloy to investigate the mechanism of die soldering during aluminum die casting. Optical and scanning electron microscopy were used to study the morphology and composition of the phases formed during soldering. A soldering mechanism is postulated based on experimental observations. A soldering critical temperature is postulated at which iron begins to react with aluminum to form an aluminum-rich liquid phase and solid intermetallic compounds. When the temperature at the die surface is higher than this critical temperature, the aluminum-rich phase is liquid and joins the die with the casting during the subsequent solidification. The paper discusses the mechanism of soldering for the case of pure aluminum and 380 alloy cast in a steel mold, the factors that promote soldering, and the strength of the bond formed when soldering occurs.

Cyclic Deformation and Fatigue of Materials; A Symposium in Honor of Professor Campbell Laird: Crack Initiation, Growth and Fatigue Life (II)

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials

Program Organizers: Zhirui Wang, University of Toronto, Department of Metals and Materials Science, Toronto, Ontario Canada; Charles McMahon, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA; Pedro D. Peralta, Arizona State University, Department of Mechanical and Aerospace Engineering, Tempe, AZ 85287-6106 USA; J. K. Shang, University of Illinois, Department of Materials Science and Engineering, Urbana, IL 61801 USA

Wednesday AM Room: Canal A
March 15, 2000 Location: Opryland Convention Center

Session Chairs: R. O. Ritchie, University of California, Dept. of Matls. Sci. & Min. Eng., Berkeley, CA 94720 USA; Johannes Weertman, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

8:30 AM

Life Prediction of Reactor Pressure Vessels under Cyclic Loading: *Ren Zhi Li*[1]; Jiunn Yuan Huang[1]; Ji Jung Yeh[1]; Ken Feng Chien[1]; Roang Ching Kuo[1]; Peter K. Liaw[2]; Jenn Gwo Huang[3]; [1]Institute of Nuclear Energy Research, Nuclear Fuels and Matls. Div., 1000 Wenhua Rd. Chiaan Village, Chiaan Village Lungtan, Taoyuan, Taiwan 325; [2]The University of Tennessee, Dept. of Matl. Sci. and Eng., Knoxville, TN 37996-2200 USA; [3]Taiwan Power Company, Taipei, Taiwan 100

Surface and internal cracks are the flaws commonly observed in nuclear reactor pressure vessels (RPV), which are subjected to cyclic loading. Nowadays, the flaw analyses are performed using "limit load" as the failure criterion for safety assessment of nuclear power plants. However, there is a substantial conservatism built into the flaw evaluation. In this work, with adopting the concept of "Damage Tolerance Design," a more reliable life prediction model was developed based on linear elastic fracture mechanics. To predict the propagation of part-through cracks in RPV by this model, it is necessary to characterize the shape of a flaw and to estimate stress intensity factors for the evolving crack. It is also known that the stress intensity factor of a surface crack varies along the flaw border, leading to a change in the flaw shape during propagation. However, in-situ measurement of the flaw length along the boundary of the surface crack could be difficult. The initial crack shape, an input for this model, can be obtained by non-destructive tests or prescribed by related regulations. A Paris-law relationship between crack growth rates and stress intensities was derived from the experimental results. Meanwhile, a series of stress intensity factors for surface cracks with different lengths and aspect ratios were estimated by finite element analyses. According to the stress intensity factors and the initial crack shape, the development of these surface cracks and remaining life under the service loading spectrum could be predicted via the block and statistical methods. Experimental results were used to assess the feasibility of this model. The front surface of a progressing crack can be observed by optical or electron microscopy. Scanning electron microscopy (SEM) was also performed to examine the fractographic features of fatigue-tested specimens, from which internal flaw profiles can be inferred. In view of the complex configuration of RPV, fatigue specimens of different designs were tested to characterize the cracking behavior of RPV at various potential sites under simulated loading conditions. With these analysis database, the residual service life of RPV will be predicted while the cracks are observed during routine maintenance.

8:55 AM

Curvilinear Coordinates for Mode III Crack Plastic Zone in a Work Hardening Solid: *Johannes Weertman*[1]; [1]Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL 60208 USA

In antiplane strain the static equilibrium equation in any curvilinear coordinate system is $\delta\sigma_{zF}/\delta F + \sigma_{zF}/R_T + \delta\sigma_{Tz}/\delta T + \sigma_{Tz}/R_F = 0$ and the strain compatibility equation is $\delta\varepsilon_{Tz}/\delta F + \varepsilon_{Tz}/R_T - \delta\varepsilon_{zF}/\delta T - \varepsilon_{zF}/R_F = 0$. Here F and T refer to finger and thumb directions and R_F and R_T are the radii of curvature of the finger and thumb trajectories. Given any particular coordinate system setting $\sigma_{zF} = 0$ and $\sigma_{Tz} \neq 0$ immediately solves some antiplane problem. Given a particular problem a coordinate system can be found (in theory) in which $\sigma_{zF} = 0$ and $\sigma_{Tz} \neq 0$. For a power law hardening law of exponent m the strain components are $\varepsilon_{zF} = 0$ and $\varepsilon_{Tz}/\varepsilon_0 = (\sigma_{Tz}/\sigma_0)^{1/m}$. The equilibrium equation (when $\sigma_{zF} = 0$ and $\sigma_{Tz} \neq 0$) reduces to $\delta\sigma_{Tz}/\delta T + \sigma_{Tz}/R_F = 0$ and the strain compatibility equation to $\delta\sigma_{Tz}/\delta F + m\sigma_{Tz}/R_T = 0$. Using the relationship $\delta^2/\delta F^2 - \delta^2/\delta T^2 = (1/R_F)\delta/\delta F - (1/R_T)\delta/\delta T$ it is easily shown that the radii of curvature of the coordinate system trajectories that gives the solution to a problem must satisfy: $\delta(1/R_F)/\delta F - m\delta(1/R_T)/\delta T + (1-m)(1/R_F)(1/R_T) = 0$. The coordinate system that satisfies this relationship is found for the problem of the plastic zone of a mode III crack in a power law work hardening solid in small scale yielding. It is $y = a(v^m u)^{2/(1+m)}$, $x = 1/2a\{u^{4/(1+m)}(2ar)^{-(1-m)/(1+m)} - v^{4m/(1+m)}(2ar)^{(1-m)/(1+m)} + [(1-m)/(1+m)](2ar)\}$, where x, y are measured from the crack tip, a is the crack half length and r is given by $(v^2/2ar)^{2m/(1+m)} + (u^2/2ar)^{2/(1+m)} = 1$. A finger trajectory is obtained by varying u while holding v fixed and vice versa for thumb trajectories.

9:20 AM

Influence of High Load Ratios on the Fatigue Threshold in Ti-6Al-4V: *B. L. Boyce*[1]; R. O. Ritchie[1]; [1]University of California, Dept. of Matls. Sci. and Mineral Eng., 1 Cyclotron Rd., MS: 62-203, Berkeley, CA 94720 USA

Typically fatigue crack propagation is characterized at low to moderate load ratios (ratio of minimum to maximum load) in the range of 0.1 to 0.8. However, "ripple-loading" conditions such as those experienced in turbine engines under high-cycle fatigue conditions can occur at very high load ratios, R>0.8. For this reason, recent experimental work has focused on the influence of load ratio in the range of 0.1-0.96 on the fatigue threshold in Ti-6Al-4V using both constant-R and constant-K_{max} approaches. At R>0.5, the variation of threshold with load ratio (or K_{max}) can largely be addressed based on closure arguments. However, at R>0.5 (beyond the elimination of macroscopic closure mechanisms), the ΔK threshold decreases linearly with respect

to K_{max}. The possible mechanisms for this behavior include: sustained load cracking (as induced by creep-fatigue or stress-assisted hydride formation), near tip closure, or static modes. Discussion will focus on recent experimental work and evidence in the literature to delineate the mechanism for this K_{max} influence on the threshold.

9:45 AM Break

10:10 AM

Plastic Deformation and Fatigue Crack Initiation in 316L Austenitic Stainless Steel: Crystallographic Aspects: *P. Villechaise*[1]; M. Mineur[1]; J. Mendez[1]; [1]ENSMA, Lab. de Mecanique et Physique des Materiaux, Teleport 2, 1 Avenue C.Ader, Chasseneuil, Cedex BP40109 France

Plastic deformation and crack initiation feature were investigated in an austenitic stainless steel (316L) cycled in tension compression at room temperature under plastic strain control. For these conditions different crack types are observed to initiate at Intense Slip Bands or in twin and grain boundaries. This paper is focused on crystallographic analysis of transgranular cracks (in PSB), the most frequent microcracks in the air environment. For that a population of some hundred of cracks was investigated. Each specimen surface area concerned by these cracks was characterized by EBSD (Electron Back Scattered Diffraction) measurements. Then, the knowledge of crystalline orientations with regard to the stress axis permitted us to identify the activated slip systems in each grain. The nature of the sites for crack initiation was also identified. Crystallographic aspects were studied by taking into account global or local textures: Firstly the influence of the global rolling texture of the material on the cyclic behavior and on total fatigue damage (cracks density) was studied. For that, different sampling directions were defined in the rolled plat to favor <111>, <100> directions corresponding to multiple slip behavior or other directions favoring single slip behavior. Differences described in this paper concern stress-strain curves and crack density. Concurrently, the role of local texture on slip band activity and crack initiation mechanisms was analyzed. Crystallographic and geometric relevant parameters in crack initiation process were then determined.

10:35 AM

A Review of Practical Application to Fatigue Life Prediction under Biaxial Stress Conditions: *F. Lorenzo*[1]; [1]Engineering Systems Inc., 600 Rockmead, Ste. 116, Houston, TX 77339 USA

Technology and materials developed more than thirty years ago is still in service in the United States and Worldwide. Normal wear and tear added to improvements, construction of new pipelines have altered and in some cases damaged old pipelines, causing failures by fatigue in pipeline systems that would have infinite life otherwise. This paper contains a review of current theories for life prediction under biaxial stresses. The basis for the various available theories will be discussed with special emphasis on the strengths and limitations of the various models and techniques to estimate fatigue properties when limited information exists. Data from pipeline rupture case studied by the author will be used to compare life prediction results. Treatment of notches caused by mechanical damage and its effect under biaxial stresses will also be discussed. The paper will also consider the practical limitation of stress based vs. strain based approaches.

11:00 AM

Cyclic Deformation and Crack Growth in Zirconia Ceramics: *M. Matsuzawa*[1]; E. Fujimagari[1]; S. Horibe[1]; [1]Waseda University, Dept. of Matls. Sci. and Eng., 3-4-1 Ohkubo, Shinjuku-ku, Tokyo 169-8555 Japan

Several years ago, it has been found that anelastic behavior is produced in Y-TZP ceramics, i.e., when stress is applied to the specimen abruptly, the strain is not simultaneously produced but gradually formed time-dependently, and after unloading it takes a time to reach the original level. Due to this anelasticity, the curious stress-strain hysteresis behavior and unusual crack growth behavior were observed. In this paper, the change of stress-strain response during cyclic loading and crack growth under monotonic and cyclic loads in Y-TZP in comparison with those in other kinds of zirconia ceramics were investigated and discussed in terms of time-dependent anelastic behavior and stress-induced phase transformation.

11:25 AM

Fatigue Crack Propagation in Underfill Materials in Microelectronic Packages: *Jieping Zhang*[1]; [1]Intel Corporation, CH5-158, 5000 W. Chandler Blvd., Chandler, AZ 85226 USA

Cracks formed in underfill materials during stresses are often observed to continue their propagation into traces in the substrate, which cause electrical failures in microelectronic devices. Hence, a fracture mechanics-based technique was used to characterize the fatigue crack propagation behavior of different underfill materials under two different environmental conditions, i.e., ambient and 85°C/85% RH. Under the ambient condition, there was a well-defined threshold existing in each material studied, while under 85°C/85% RH, near-threshold instability was observed. Crack growth rate at the near threshold region suddenly increased after a period of time during cycling. The near-threshold instability is believed to be the result of interaction between the materials and the environment.

General Recycling of Materials: Topics Related to Light Metals Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee
Program Organizers: Guy Fredrickson, Reynolds Metals Company, Smelter Technology Laboratory, Muscle Shoals, AL 35661 USA; Ilaria Accorsi, Daimler Chrysler, Toledo, OH 43606 USA

Wednesday AM Room: Canal C
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Guy L. Fredrickson, Reynolds Metals Company, Smelter Tech. Lab., Muscle Shoals, AL 35661 USA; Christina Viklund-White, MEFOS, New Tech. Dept., Lulea SE-971 25 Sweden

8:30 AM Introductions and Opening Comments

8:40 AM

New Techniques for Separation of Non-Ferrous Metals from Waste Streams: *Gerrit H. Nijhof*[1]; Peter C. Rem[2]; [1]Nijhof Consultancy, Heemsteedse Dreef 92, KN Heemstede 2102 The Netherlands; [2]Delft University of Technology, Mijnbouwstraat 120, RX Delft 2638 The Netherlands

Separation technologies and upgrading of the non-ferrous metals for remelting purposes is a continuous subject of research. Progress has been reported during the previous four session on General recycling of the Light Metals Conferences. Two items last years presentation in San Diego were further investigated in this year: 1)Image analysis of the material separated by the Eddy Current and 2)Wet Eddy Current sorting. Measurements have been performed on the sorting and monitoring of household waste under industrial conditions using image analysis. The “Wet Eddy Current” technology is further developed. Experiments have been performed on the separation of aluminum and aluminumoxide from dross. In this presentation the actual situation of the running research and the results of recent experiments will be presented.

9:05 AM

Plasma Processing of Waste MgO Dust: *Steven W. White*[1]; Ramana G. Reddy[2]; Banqiu Wu[2]; [1]University of Alabama, Dept. of Chem. Eng., P.O. Box 870203, Tuscaloosa, AL 35487 USA; [2]University of Alabama, Dept. of Metallu. and Matls. Eng., P.O. Box 870202, Tuscaloosa, AL 35487 USA

Plasma technology has been around for a long time, but has just recently been used widely for a number of new processes, including the treatment of waste materials. Current investigation includes the manufacturing of magnesium from waste MgO dust using carbon as a reduc-

ing agent. Plasma energy is ideal for this reaction because of its high enthalpy and improved energy efficiency over conventional methods. Experimental results showed that high magnesium metal recovery was obtained with a 48 kW power input nitrogen plasma. Rapid quenching was employed to minimize back reactions. Thermodynamic and kinetic models were developed. Good agreement was obtained between experimental and theoretical data. Based on these results, a process flow sheet was developed.

9:30 AM

Recycling of Titanium and Ti-6Al-4V Turnings Using Thermohydrogen Processing: *Javaid I. Qazi*[1]; Oleg N. Senkov[1]; Francis H. Froes[1]; Valadimir S. Moxson[2]; [1]University of Idaho, Instit. for Matls. and Adv., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]ADMA Products Inc., 8180 Boyle Park Way, Twinsburg, OH 44087 USA

Commercially pure (CP) titanium and Ti-6Al-4V alloy powders were produced from their respective turnings. Turnings were initially cleaned, hydrogenated and then milled to produce the powders. Parts of CP titanium and Ti-6Al-4V were produced from these powders by a novel loose sinter approach. The presence of hydrogen in the system allowed thermohydrogen processing to be performed to produce a fine-grained structure in sintered parts. The hydrogen was then removed by vacuum annealing. Mechanical properties were studied and compared with the properties of parts made of more expensive powders.

9:55 AM

Recovery of Metal Ions from Process Waste Solutions by Cementation: *Raj P. Singh*[1]; [1]OSRAM Sylvania Inc., Chem. Dev. Dept., Rsch. and Dev. Div., Hawes St., Towanda, PA 14848 USA

This paper would review the applications of cementation in the recovery of metal ions from process waste solutions. Cementation reactions involve electrochemical precipitation of a noble metal from solutions of its salts on a more electropositive metal which, in turn, progressively dissolves. For example, addition of zinc metal powder in a solution containing zinc and cobalt ions would allow for the precipitation (separation) of cobalt according to the following chemical reaction: $Co^{2+} + Zn° = Co° + Zn^{2+}$. Cementation has been frequently used for the separation/recovery of trace or small concentrations of impurity metal ions from advanced electrolytes (product). The method has also been applied for the recovery of precious metals from process streams and toxic metals from process wastewater. In addition to the review of important applications of cementation in hydrometallurgy, the results of our recent work on cementation of cobalt and other metal ions from process waste solutions would also be discussed.

10:20 AM Break

10:45 AM

Gallium Recovery as a By-Product of Bauxites: *Arthur Pinto Chaves*[1]; Alcídio Abrão[1]; Waldemar Avritscher[1]; [1]University of São Paulo (Epusp), Escola Politécnica, Av. Prof. Mello Moraes 2373, São Paulo, SP 05508-900 Brazil

There is no one deposit of gallium all over the world. All this metal is recovered as a by product from the zinc or mainly from the aluminium industry. As Brazil is a major aluminium producer, there is potential for becoming a gallium producer of international expression. This paper describes the effort done to develop a process to provide high grade gallium from Brazilian Bayer liquors. The basic processes of solvent extraction and ion exchange have been tried. The first one was successful but had to be discontinued as the manufacturers of the extractant refused to sell it to us. Ion exchange resins selective to gallium were also successful but it has also been impossible to buy such a product. An effort has been done to synthesize resins similar to those described in the literature. It has been successful and we consider now our “home made” product superior to the commercial samples tested. The refine of the metal is done via electrolisis. Specific problems of oxidation and texture of the deposited metal had to be solved. The gallium market will be described in terms of demand and prices. The unit operations will be discussed, as well as the characteristics of Brazilian raw materials and the problems associated with these characteristics.

11:10 AM

Strong Oxidizing Acid Bath Rejuvenation: *James D. Mavis*[1]; [1]Ch2m Hill, P.O. Box 91500, Bellevue, WA 98009 USA

During manufacture, metals such as titanium, zirconium, and aluminum may undergo surface treatment in strong oxidizing acid baths. Bath life may constrained by the etch or milling rate, by the formation of secondary deposits, or by metal surface quality. Recent development work has shown that baths life could be extended using electrochemical methods to selectively remove metals that accumulate during bath use. Preliminary evaluation indicates these processes might be a cost-effective alternative to periodically replacing high-usage rate baths.

11:35 AM

Reclaiming Salt Flux from Aluminum Salt Slag Wastes Process Design-Product Performance: *Richard Russell*[1]; Jerry Sweeny[2]; [1]Richard M. Russell & Associates, LLC Consulting Eng., 2003 Blair Blvd., Nashville, TN 37212 USA; [2]Tennessee Aluminum Processors Inc., Mt. Pleasant, TN USA

The disposal of aluminum slag wastes continues to challenge an otherwise environmentally beneficial and highly desirable industry-Aluminum Recycling. Slag wastes consists of soluble salts that can be reused in the aluminum smelting. Reclaiming salt is a logical first step in eliminating slag wates. Reclaiming salt flux has been conducted on a very limited basis in the United States for reasons of economy. Practical methods for the production of reclaimed flux as well as rising tipping fees will increase the number of plants which opt to recover flux salts from slag wastes. The authors share their experiences in the design, operation and startup of a commercial scale flux reclamation plant operated by Tennessee Aluminum Processors at Mt. Pleasant Tennessee. This paper identifies three elements that allow practical operations: minimizing evaporator heat duty, simplifying evaporator operations and potassium chloride fortification of the recycled salt.

12:00 PM

Recycling Contaminated Aluminum in a Salt-Free Environment: *Donald M. Martosko*[1]; [1]LOI Inc., 333 Tech. Dr., Ste. 109, Canonsburg, PA 15317 USA

Rising energy prices and the worldwide crisis of available natural resources have made the recycling of aluminum scrap increasingly attractive, from economical as well as an environmental points of view. Until recently, recycling of wet or “dirty” scrap contaminated by oil, grease, paint, or thermal insulation layers (rubber or plastic) has posed significant problems and difficulties with material preparation, the environment, energy consumption, metal loss, and dross accumulation. Our solution consists of a twin-chambered melting furnace, capable of processing up to 18,000 lbs./hr with a capacity of up to 150,000 lbs., in a salt-free environment, using electromagnetic pumping technology and an automatic charging machine. The results include low energy & fuel consumption, minimal metal loss, and reduced dross accumulation in the bath. As the name implies, the twin-chamber melting furnace consists of two (2) chambers: an indirectly heated scrap chamber and a directly heated high-temperature chamber. An air-cooled wall separates the two chambers from each other. The molten metal baths of both chambers are interconnected through an opening located at the bottom of the wall. The scrap chamber is used for clean and contaminated thin-walled, bulk, sheet, thin-walled scrap, and other scrap of such kind. The chamber heated by means of the main burners is suitable for ingots, pigs, and sows, thus allowing furnace charging from sills located at both ends. An electromagnetic pump allows charging of fine turnings with minimal metal loss. This procedure also allows damp material, or scrap with water inclusions, to be charged without any risk. This process is based on low-temperature carbonization and subsequent incineration of the contaminants, making it an environmentally compatible solution. The scrap chamber is heated, using the energy from the flue gases of the heated chamber. The heat value of the organic substances is released for use in melting, thus also offering an effective energy saving over conventional melting practices.

High Resolution Electron Microscopy in Materials Science: Interfaces

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

Program Organizers: Diane E. Albert, Los Alamos National Laboratory, MST-6, The Metallurgy Group, Los Alamos, NM 87545 USA; Martin Allen Crimp, Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Wednesday AM Room: Canal D
March 15, 2000 Location: Opryland Convention Center

Session Chair: Martin A. Crimp, Michigan State University, Dept. of Matls. Sci. and Mech., East Lansing, MI 48824-1226 USA

8:30 AM Invited

HREM Characterization of Hetero-Epitaxial Interfaces: *David Smith*[1]; [1]Arizona State University, Dept. of Phys. and Astro., Tempe, AZ 85287 USA

There is much current interest in structures based on two (or more) dissimilar materials but there are many problems associated with achieving heteroepitaxial growth. In addition to lattice mismatch, which invariably causes strain and possible defect formation, valence mismatch and differences in thermal expansion are factors that can seriously impact the quality of the materials which can be grown. Characterization of microstructure using HREM, in addition to close interaction with the crystal grower, can play a valuable role in optimizing the growth process. This talk will concentrate on three systems of considerable scientific and industrial importance: a) heterostructures based on SiGeC/Si, which offer the intriguing prospect of band-gap engineering in a lattice-matched system; b) CdTe/Si substrates, which could allow the monolithic integration of Si electronics with MCT (mercury cadmium telluride) infrared detectors; and c) alloys of Group III-nitrides, which could totally revolutionize the microelectronics and optoelectronics industries.

9:00 AM

Interface-Related Phenomena of Deformation and Fracture in Two-Phase Titanium Aluminides: *Fritz Appel*[1]; [1]GKSS Research Centre, Instit. for Matls. Rsch., Max-Planck-Strasse, Geesthacht D-21502 Germany

Titanium aluminides with compositions slightly lean in Al are presently being considered for engineering applications. Phase equilibria and transformation in these alloys lead to the formation of a lamellar microstructure comprising of the intermetallic phases alpha 2(Ti_3Al) and gamma (TiAl). This morphology has received much attention since nearly all mechanical properties of g-base alloys are improved when the materials contain a significant volume fraction of lamellar colonies. Several aspects of deformation and fracture might be associated with mismatch structures and coherency stresses of lamellar interfaces. In the present study these structural features will be examined by detailed transmission electron microscopy observations involving in situ heating studies and high resolution imaging techniques. The implications of interface related processes will be discussed with respect to strengthening phenomena and the structural stability of lamellar materials.

9:30 AM

A Few Applications of HRTEM to Hetero-Interfaces: *Pirouz Pirouz*[1]; [1]Case Western Reserve University, Matls. Sci. and Eng., 510 White Bldg., CWRU, 10900 Euclid Ave., Cleveland, OH 44106-7204 USA

A few applications of high-resolution transmission electron microscopy to the study of interfaces between dissimilar materials will be discussed. These include metal/ceramic, ceramic/ceramic, semiconductor/semiconductor and semiconductor/ceramic interfaces. In addition to interfacial defects such as misfit dislocations, the formation of different variants during deposition on a substrate, and the resulting planar defects between these variants will be considered. In particular, the talk will focus on interfaces that occur during phase transformations in wide bandgap semiconductors and look in detail at the intermediate phases that occur during a phase transformation.

10:00 AM

Structure and Morphology of Interfaces in Thin Films of Au on Ge Substrates: *Tamara Radetic*[1]; Urlich Dahmen[1]; [1]Lawrence Berkeley National Laboratory, Nat. Ctr. for Elect. Micro., Matls. Sci. Div., Bldg. 72, 1 Cyclotron Rd., Berkeley, CA 94720 USA

{110} Au mazed bicrystal films have been grown epitaxially on {001} Ge substrates by physical vapor deposition. There are only two equivalent orientation variants in the film, resulting in a "mazed bicrystal" microstructure made of many grains in only two orientations, rotated 90° about a common <110> axis. The misorientation between grains is fixed, while the inclination (grain boundary plane) is variable. Grain boundaries are perpendicular to the film/substrate interface and are therefore are of 90° pure tilt character. These films have been annealed in situ and the effect of annealing on the structure and morphology of grain boundaries and interfaces with the substrate has been investigated by conventional and high resolution electron microscopy. The stability of particular grain boundary inclinations during grain coarsening has been examined and their atomic structure has been studied by HREM. It has been observed that during annealing interdiffusion at the Au/Ge interface leads to diffusion induced grain boundary migration in the film as well as morphological instabilities at the Au/Ge interface. The evolution of the atomic structure of the Au/Ge interface with annealing has been characterized by HREM. This work is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-ACO3-76SFOOO98.

10:30 AM Break

10:50 AM Invited

HREM Studies of Interfaces and Boundaries in CVD Diamond: *John Hutchison*[1]; Dan Schectman[2]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX13PH UK; [2]Technion, Dept. of Matls. Eng., Haifa, Israel

Thin films of diamond grown on Si substrates by CVD techniques have been investigated by high resolution electron microscopy, using a JEOL 4000EX electron microcsope. Specimens were prepared by removal of the Si substrate followed by Ar+ ion milling. Most grains in the films showed heavy twinning on {111} planes, with additional twin structures of increasing complexity. Five-fold twin centres were found in many grains, and it is shown that these centres give rise to incoherent, high-order twin boundaries. The core structure of the five-fold twin centre is shown to be a planar C5 ring, a stable ring configuration of sp3 carbon atoms. The implications of the twin structures on crystal growth processes will be discussed, and it will be shown that the first-order twin boundaries are essential for fast crystal growth, and give rise to many of the features that are observed on the macroscopic scale.

11:20 AM

Substitutional Impurity Segregation to the Sigma 5 (310)/[001] Symmetric Tilt Grain Boundary in Fcc Metals: *Juergen M. Plitzko*[1]; Geoffrey H. Campbell[1]; Wayne E. King[1]; Stephen M. Foiles[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci. Direct., Mailstop L-370, P.O. Box 808, Livermore, CA 94550 USA; [2]Sandia National Laboratories, Comp. Matls. Sci. Dept., Mailstop 9161, P.O. Box 969, Livermore, CA 94550 USA

The Sigma 5 (310)/[001] symmetric tilt grain boundary (STGB) has been investigated in four different fcc metal systems. The metals chosen include pure aluminum, pure copper, aluminum with 1 at.% copper, and copper with 1 at.% silver. These model grain boundaries have been fabricated by ultra-high vacuum diffusion bonding of precisely oriented single crystals. The atomic structure of these STGBs

has been modeled with atomistic simulations using interatomic potentials based on the Embedded Atom Method and with electronic structure calculations within the Local Density Approximation. The theoretical calculations of the interface structure indicate that the Cu and Ag atoms segregate to distinct sites at the interface. High resolution transmission electron microscopy (HREM) and analytical electron microscopy have been used to validate the theoretical models. The HREM images and analytical measurements were performed using a Philips CM300-FEG equipped with an imaging energy filter. The amounts of the segregated species at the grain boundary have been quantified. To determine the atomic positions of the segregated atoms at the interface, HREM coupled with image simulation and reconstruction of through-focal series have been used. Finally, these experimental results are discussed and compared to the theoretical model. This work was performed at Lawrence Livermore National Laboratory under the auspices of the United States Department of Energy under Contract W-7405-Eng-48.

11:40 AM

Electronic Effects on Grain Boundary Structure in BCC Metals: *Geoffrey H. Campbell*[1]; Wayne E. King[1]; James Belak[1]; John A. Moriarty[1]; Stephen M. Foiles[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., Mailstop L-356, P.O. Box 808, Livermore, CA 94550 USA; [2]Sandia National Laboratory, Computat. Matls. Sci., Mailstop 9161, P.O. Box 969, Livermore, CA 94550 USA

The dominant factor in determining the atomic structure of grain boundaries is the crystal structure of the material, e.g. FCC vs. BCC. However, for a given crystal structure, the structure of grain boundaries can be influenced by electronic effects, i.e. by the element comprising the crystal. Understanding and modeling the influence of electronic structure on defect structures is a key ingredient for successful atomistic simulations of materials with more complicated crystal structures than FCC. We have found that grain boundary structure is a critical test for interatomic potentials. To that end, we have fabricated the identical Sigma 5 (310)/[001] symmetric tilt grain boundary in three different BCC metals (Nb, Mo, and Ta) by diffusion bonding precisely oriented single crystals. The structure of these boundaries have been determined by high resolution transmission electron microscopy. The boundaries have been found to have different atomic structures. The structures of these boundaries have been modeled with atomistic simulations using interatomic potentials incorporating angularly dependent interactions, such as those developed within Model Generalized Pseudopotential Theory. The differing structures of these boundaries can be understood in terms of the strength of the angular dependence of the interatomic interaction. This work was performed under the auspices of the United States Department of Energy and the Lawrence Livermore National Laboratory under contract number W-7405-Eng-48.

Honorary Symposium for Professor Oleg D. Sherby: Ultrahigh-Carbon Steels

Sponsored by: Structural Materials Division, Materials Processing and Manufacturing Division, Structural Materials Committee, Shaping and Forming Committee

Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; Chol K. Syn, Lawrence Livermore National Laboratory, Manufacturing & Materials Engineering Division, Livermore, CA 94550 USA

Wednesday AM Room: Bayou E
March 15, 2000 Location: Opryland Convention Center

Session Chair: Eric Taleff, The University of Texas, TX Matls. Instit., Austin, TX 78712-1063 USA

8:30 AM Keynote

The Evolution of Ultrahigh-Carbon Steels-From the Great Pyramids, to Alexander the Great, to Y2K: *Jeffrey Wadsworth*[1]; [1]Lawrence Livermore National Laboratory, Director's Off., P.O. Box 808, L-001, Livermore, CA 94550 USA

Hypereutectoid steels containing between about 1 and 2.1 wt%C, and now known as ultrahigh carbon steels (UHCS), have both a rich history (dating back to ~300 BC) and an interesting, recent, technological period of development (from 1975 to the present). The connections between the modern UHCS and their ancient counterparts, and in particular, Damascus Steels, have received considerable attention. In addition to monolithic products, UHCS have also been used in both ancient and modern times in laminated composites. In the present paper, a summary of the modern development of UHCS and UHCS-containing laminates is given, and parallels are drawn with ancient materials. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

9:00 AM Invited

A History of the Patenting and Licensing of Ultrahigh-Carbon Steels: *Jon C. Sandelin*[1]; [1]Stanford University, Office of Tech. Licen., 900 Welch Rd., Ste. 350, Palo Alto, CA 94304 USA

Only a small number of discoveries made at universities become commercially significant and the road to commercial success may take many years, or sometimes decades. Ultra High Carbon Steels have not yet reached large scale commercial use, but the licensing history has some interesting stories and demonstrates the difficulties in introducing new materials into commercial markets. This paper will describe the patented technologies and review the licensing history of the inventions of Professor Oleg Sherby and his colleagues. We will explore the idea of Ultra High Carbon Steels as a disruptive technology, as the creation of steel minimills is considered to be. We will then look to the future to consider applications and opportunities for Ultra High Carbon Steels.

9:20 AM Invited

Superplasticity and Strength of Ultrahigh-Carbon Steels Extruded at Intermediate Temperatures: *Toshi Oyama*[1]; Chol K. Syn[2]; Donald R. Lesuer[2]; J. Daniel Whittenberger[3]; Oleg D. Sherby[4]; [1]WESGO Metals, Belmont, CA 94002 USA; [2]Lawrence Livermore National Laboratory, L-342, Livermore, CA 94551 USA; [3]NASA-Lewis Research Center, Cleveland, OH 44135 USA; [4]Stanford University, Matls. Sci. and Eng., Stanford, CA 94305 USA

Ultrahigh-carbon steels (UHCSs) are readily extruded at intermediate temperatures, 650 to 820°C, into round bars and T-shape sections. The extruded UHCSs were tested in tension at both ambient and inter-

mediate temperatures. At ambient temperature, all extruded UHCSs showed high tensile strength (ultimate tensile strength of more than 700 MPa) with outstanding elongation (elongation-to-failure of over 30%). At intermediate temperatures, superplastic behavior was observed in the UHCSs extruded below the A_1 temperature. The results are compared with other published data on high-temperaure extrusion of UHCSs (900 to 1100°C).

9:40 AM
Thermomechanical Processing of Austempered Ductile Iron: *Tara Chandra*[1]; [1]University of Wollongong, Matls. Eng., Wollongong, NSW 2522 Australia

High temperature deformation of both unalloyed and alloyed ductile irons was carried out under uni axial compression at an austenitisation temperature of 900°C immediately prior to austempering. It was found that in general deformation increased the transformation kinetics during austempering. For the alloyed ductile irons with high manganese levels (>0.3%), the deformation resulted in a significant reduction on the volume fraction of the deleterious unstabilised austenite in the intercellular regions. The 30% reduction at 900°C resulted in austenite grain refinement by dynamic recrystallization. The refined grain structure in turn provided more nucleation sites for the bainitic ferrite formation and thus accelerating the transformation kinetics during the austempering process.

10:00 AM Break

10:10 AM Keynote
Fracture Toughness of Ultrahigh-Carbon Steel: Pearlite, Spheroidite, and Tempered Martensite: Alberto Fernandez[2]; Manolo Carsi[2]; *Oscar Ruano*[2]; Eric M. Taleff[1]; Oleg D. Sherby[3]; [1]The University of Texas, TX Matls. Instit., Austin, TX 78712-1063 USA; [2]Centro Nacional de Investigaciones Matalurgicas, Av. de Gregorio del Amo 8, Madrid 28040 Spain; [3]Stanford University, Matls. Sci. and Eng., Stanford, CA 94305-2205 USA

Two ultrahigh-carbon steel (UHCS) alloys have been thermal-mechanically processed to obtain various microstructures. The first alloy contains 1.3 C, 0.5 Mn, 0.6 Si, and 0.18 Cr in weight percent, and the second alloy contains 1.5 C, 0.75 Mn, 0.23 Si, 0.09 Cr and 0.31 Mo in weight percent. The microstructures developed by processing include fine-grained spheroidite, coarse-grained spheroidite, pearlite of several different interlamellar spacings, and tempered martensite. Unique thermal-mechanical processing procedures are described for producing several of these microstructures in each alloy from the same fine-grained spheroidized material of the respective alloy. The fracture toughnesses of both UHCS alloys with each microstructure have been experimentally determined using chevron-notch samples. The effects of microstructure and alloy content on fracture toughness are presented and discussed in light of more general trends in the behavior of UHCS materials.

10:40 AM Invited
Microstructural Effects on the Cleavage Fracture Stress of Pearlitic Steels: *John J. Lewandowski*[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. and Eng., 10900 Euclid Ave., Univ. Circle, Cleveland, OH 44016 USA

This presentation will provide a review of the microstructural features which affect the cleavage fracture stress in fully pearlitic steels. Data has been obtained over a wide range of test temperatures on a variety of steels where the pearlite interlamellar spacing, prior austenite grain size, and pearlite lamallae thickness have been varied. The effects of such changes on the magnitude of the cleavage fracture stress will be reviewed.

11:00 AM
Influence of Carbides on the Hot Working of Steel: *Hugh J. McQueen*[1]; C. A.C. Imbert[2]; [1]Concordia University, Mech. Eng. H 54934, 1455 de Maisonneuvre Blvd. W., Montreal, Quebec H3G1M8 Canada; [2]University of the West Indies, Mech. Eng., St. Augustine, Trinidad

Carbides have a strong influence on the hot working behaviour of steels depending on their size, distribution, solubility, hardness and the matrix, either austenite or ferrite. Fine carbides pin dislocations slowing dynamic recover (DRV) and despite the higher dislocation density delay dynamic recrystallization by pinning the grain boundaries. Large carbides raise dislocation density in the surrounding region, thus enhancing nucleation of DRX. Fine carbides reduce ductility by slowing DRV and DRX, whereas large carbides cause stress concentrations and cracks. In tool steels, the carbides are stable up to quite high temperatures causing considerable increase in tendency for cracking, strength and activation energy compared to C steels. In HSLA steels, the fine microalloy carbonitrides tend to precipitate at the low end of the hot working range, which raises the strength and the apparent activation energy while lowering the ductility. For steels with an austenitic phase in which the dissolved carbon enhances DRV and ductility, carbides considerably raise the strength and decrease ductility on coding to the ferritic phase. Pearlitic steels are stronger and less ductile than those in the spheroidized condition; however, they show considerable work softening as the pearlite spheroidizes during hot working. By suitable thermomechanical processing, the high carbon steels can be produced with a micro-duplex structure capable of superplastic deformation. In multistage rolling schedules, large alloy carbides slow interpass recrystallization and hence grain refinement at the hot end. In contrast, fine microalloy carbonitrides delay it at the cool end, resulting in pancaked austenitic grains which yield a fine ferritic grain structure.

11:20 AM Round Table Discussion
From Damascus Steels to Ultrahigh-Carbon Steels-What does the past reveal of future applications?
Moderator: George Mayer

Hume Rothery Award Symposium; Phase Transformations and Evolution in Materials: Session IV

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Alloy Phases Committee

Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA

Wednesday AM — Room: Johnson A/B
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: Kazuhiro Otsuka, University of Tsukuba, Instit. of Matls. Sci., Tsukuba, Ibaraki 305-8573 Japan; Alphonse Finel, ONERA/CNRS, LEM, BP72 Chatillon, Cedex, France

8:30 AM Invited
Mechanical Stability and the Limits of Strength: *John William Morris*[1]; Chris R. Krenn[1]; David Roundy[2]; Marvin L. Cohen[2]; [1]University of California-Berkeley, Dept. of Matls. Sci., 555 Evans Hall, Berkeley, CA 94720 USA; [2]University of California, Dept. of Phys., Berkeley, CA 94720 USA

The upper limit of strength (the "theoretical strength") has been an active subject of research and speculation for the better part of a century. The subject has recently become important, for two reasons. First, given recent advances in ab initio techniques and computing machines, the limits of strength can be calculated with considerable accuracy, making this one of the very few problems in mechanical behavior that can actually be solved. Second, given recent advances in materials engineering, the limits of strength are being approached in some systems, such as hardened or defect-free films, and their relevance is becoming recognized in others, including hard coatings, carbonitrides and diamond-cubic crystals. An elastically strained solid is always at least metastable. Given a kinetically plausible pathway, it

will spontaneously transform into a sheared or broken replica of itself or into a new phase entirely. In that sense, plastic deformation is a structural phase transformation whose onset is governed by the usual criteria. It can be nucleated (and ordinarily is) but, failing that, must commence at the limit of stability of the elastic state. This thermodynamic instability sets the upper limit of strength. The present paper defines the limits of elastic stability (which are surprisingly subtle), shows how those limits reflect the symmetry of the strained lattice, reviews ab initio computations for a number of metals and compounds, and discusses the experimental situations in which they are known or expected to be important.

9:00 AM Invited

Magnetic Ordering: Some Structural Aspects: *David E. Laughlin*[1]; Matthew A. Willard[1]; Michael E. McHenry[1]; [1]Carnegie Mellon University, Matls. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

An overview of some structural aspects of magnetic ordering will be presented. Magnetic symmetry operations, point groups, and Bravais lattices will be utilized to describe the magnetic symmetry of various magnetic materials. Some less common types of magnetism will be introduced and various phase transitions between them will be described. Throughout the talk, the utilization of the theory of magnetic symmetry will be emphasized. The authors acknowledge the financial support of the Air Force Office of Scientific Research, Air Force Materiel Command, USAF, under Grant No. F49620-96-1-0454.

9:30 AM Invited

Thermodynamics of Open Two-Phase Systems with Coherent Interfaces: *Ricardo B. Schwarz*[1]; Armen G. Khachaturyan[2]; [1]Los Alamos National Laboratory, MST-8, Mail Stop G755, Los Alamos, NM 87545 USA; [2]Rutgers University, Dept. Mech. and Matl. Sci., Piscataway, NJ 08855-0909 USA

We develop a theory for the decomposition of a solid containing interstitial atoms into two coherent phases of different interstitial concentration. It is shown that the coherency strain changes the conventional thermodynamics of the phase transformation by producing a macroscopic energy barrier between the transforming phases. This barrier locks the system in metastable states that cannot be surmounted by thermal fluctuations. As a result, the system looses ergodicity, which is a fundamental requirement of Gibbs thermodynamics. Further, two-phase coexistance is no longer possible. The cases where the solid is in equilibrium with finite and infinite reservoirs of interstitial atoms are considered. The theory is applied to the decomposition of metal/hydrogen systems and explains quantitatively the ubiquitous hysteresis in the pressure 'plateaux' of the pressure-composition isotherms. The proposed theory predicts the temperature dependence of the hysteresis and allows one to calculate the critical pressure and temperature where the hysteresis disappears (where the difference between the hydrogenation and dehydration 'plateau' pressures vanishes). Work supported by the U.S. Department of Energy, Office of Basic Energy Sciences.

10:00 AM Invited

Time Evolution of Microstructures in Ferroelastics: *Ekhard K. H. Salje*[1]; [1]University of Cambridge, Dept. of Earth Sci., Downing St., Cambridge CB2 3EQ UK

The experimentally observed time evolution of ferroelastic microstructures in framework structures is compared with results of large scale computer simulations. Quenching a sample through a ferroelastic transition point leads in continuous phase transitions to a characteristic succession of tweed, tartan, needle, comb, and stripe patterns. The computer simulations are based on a hybride method with atomic ordering treated by Monte Carlo and structural relaxations by molecular dynamics methods. The elastic, long-ranging correlations dominate the microstructural evolution in the vast majority of chemical systems which will be discussed. A notable exception is the hexagonal-monoclinic transition in cordierite. Topological twin-type structures are found in addition to traditional twin walls while the later obey the elastic compatibility rule, the former do not. Instead they minimize the local structural relaxation energy. Their appearance leads to an enhancement of thermal fluctuations and a novel type of wall wetting in the low symmetry phase.

10:30 AM Break

10:45 AM Invited

Premartensitic Phenomena in Ti-Ni-Based Alloys Seen through Elastic Constants: *Kazuhiro Otsuka*[1]; Xiaobing Ren[1]; [1]University of Tsukuba, Instit. of Matls. Sci., Tennodai 1-1-1, Tsukuba, Ibaraki 305-8573 Japan

Premartensitic phenomena in b-phase alloys attracted considerable attention in recent years. In these studies attention was paid to the softening of elastic constant cÕ, which represents the resistance to {110}<110> shear. In the present paper we will report a result of systematic elastic constants measurement of various Ti-Ni based alloys, which exhibit three types of martensitic transformations, depending upon composition and heat-treatment. i.e. B2-B19Õ, B2-B19-B19Õ and B2-R phase-B19Õ. In all cases, both cÕ and c44 exhibited softening with decreasing temperature toward the transformation temperatures, where c44 represents the resistance to {001}<100> shear. However, the temperature dependence of the anisotropy factor A=c44/cÕ behaved differently depending upon the types of the transformations. At the conference we will show the importance of not only cÕ but also c44, and the correlation between the elastic softening and the subsequent martensitic transformations.

11:15 AM Invited

Martensitic Transformation under Stress in Ferrous Alloys: *Elisabeth Marie Gautier*[1]; [1]Ecole des Mines de Nancy, LSG2M UMR CNRS 7584, Parc de Saurupt, Nancy, Cedex 54042 France

When martensitic transformation occurs under external stresses, a transformation induced plasticity is observed, as a modification of the kinetics of the transformation and of the morphology of the plates. These modifications, which have to be analysed simultaneously, are dependent on the level of the applied stress. We consider the behaviour for stresses ranging from low external stresses up to stresses larger than the yield stress of the parent austenite. Such a stress range is interesting to consider because stresses generated by the transformation itself have a large contribution to the observed phenomena and cannot be disregarded. Experimental measurements of transformation plasticity deformation in Fe-Ni-C alloys have shown that the two major mechanisms responsible for transformation plasticity i.e. orientation of the martensitic plates, anisotropic plastic accommodation of the transformation strain in the stress direction are occurring in the case of ferrous alloys. The contribution of each mechanism is dependent on the level of the applied stress, the transformation progress and the mechanical properties of the parent austenite. When transformation plasticity deformation increases, a simultaneous change in the plate morphology is observed. The width of the plate is increased, indicating that a further growth of the thin plate is allowed under stress. Based on theoretical analysis this increase can be linked to the occurrence of a change in the stress relaxation process, i.e. the occurrence of plastic deformation in the parent phase. In order to further analyse the effect of the stress, a simple micromechanical model has been established using finite element modelling. The respective role of the applied stresses and the internal stresses has been analysed considering elastic and elastoplastic behaviour of the phases. It is shown that the plate arrangement is modified from self accommodating (for no and low applied stresses) to a single orientation when the applied stress increases, and at the beginning of the transformation. The stress relaxation by plastic deformation leads to a larger efficiency of the applied stress for the plate orientation, giving thus a greater transformation plasticity deformation. This analysis is then able to explain the transformation kinetics observed when the transformation occurs under stress.

11:45 AM Invited

Potency of Heterogeneous Martensitic Nucleation Sites: Gregory B. Olson[1]; *Andrew C. E. Reid*[1]; [1]Northwestern University, Matls. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

The nonlinear, nonlocal continuum elastic fields of various dislocation structures are studied in the context of Martensitic nucleation. The nonlinearity of the model system results from the imposition of a multiple-minimum, Landau-Ginzburg type potential energy, while the dislocations are modeled by the imposition of a topological constraint on the nonlinear elastic continuum, which fixes the Burger's

vector content of the system. The different dislocation structures have differing potencies for nucleation of the Martensitic phase, characterized by the driving force at which the embryo becomes unstable with respect to growth. The dislocation and embryo structures are explored for small 2D and quasi-3D systems in the low-potency limit corresponding to small embryo size.

International Symposium on Global Innovations in Materials Processing and Manufacturing: Structure-Property Evaluations in Solid Free Form Fabrication

Sponsored by: Materials Processing and Manufacturing Division,

Program Organizers: David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; James W. Sears, Lockheed Martin, KAPL Inc., D2, 114, Schenectedy, NY 12301 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; Rob Wagoner, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Wednesday AM Room: Canal E
March 15, 2000 Location: Opryland Convention Center

Session Chair: Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

8:30 AM

Microstructure-Property Evaluations in Fe-25Ni Samples Produced with Directed Light Fabrication: *Dan J. Thoma*[1]; Guleid N. Hussen[1]; Michael L. Steinzig[1]; Bryan R. Lally[1]; Joe C. Fonseca[1]; Frank H. Harlow[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Many freeform fabrication efforts rely on the near-net shape production of parts with structural reliability throughout the component. As a result, significant effort is required to define and control the microstructural development during processing. To gain a better understanding of direct laser fabrication techniques, the solidification behavior and resulting tensile properties have been studied in test bars of a model system, Fe-25Ni. By coupling systematic variations in process parameters with solidification modeling, microstructural investigations, and quasi-static mechanical testing, significant enhancements in the as-solidified properties are possible. For example, by doubling the laser traverse speed, the primary dendrite arm spacings decrease over 15%. Within these limits, the ultimate tensile strength increases 100 MPa (to 850 MPa) with a factor of two increase in ductility (to 6%). With cooling rates being on the order of 10^4 K/s, the current investigation supports optimized properties with microstructural refinement.

8:50 AM

Microstructure and Properties of LENS® Processed 304l Stainless Steel: *J. A. Philliber*[1]; J. E. Smugeresky[1]; B. P. Somerday[1]; S. Robinson[1]; M. Griffith[2]; [1]Sandia National Laboratory, Livermore, CA 94551 USA; [2]Sandia National Laboratory, Albuquerque, NM 87185 USA

The Laser Engineered Net Shaping (LENS®) process has been used to fabricate solid freeform samples of a variety of metals. The smaller grain size resulting from the LENS® processing has produced yield strengths of 56-70 KSI (385-480 MPa) in 316 SS. This represents a two-fold increase over the properties of conventional, annealed 316 with no loss in ductility. Recent experiments on 304L SS have also produced an increased yield strength; but, to a lesser degree than in 316 SS. The LENS® processed 304L had a yield strength of 45-50 KSI (310-345 MPa) when tested perpendicular to the build layers, and ~65 KSI (450 MPa) when tested parallel to the build layers. The microstructural features of the LENS® 304L material, including grain size, second phase particles and dislocation densities, will be related to that of the LENS® processed 316 SS and to the mechanical properties. Work supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

9:10 AM

Microstructure and Properties of Laser Deposited and Wrought Alloy K-500 (UNS N05500): *Patrick W. Hochanadel*[1]; Robert D. Field[1]; Gary K. Lewis[1]; Joe C. Fonseca[1]; Patrick G. Dickerson[1]; [1]Los Alamos National Laboratory, MST-6: Metallu., P.O. Box 1663, MS G770, Los Alamos, NM 87545 USA

Alloy K-500 (UNS N05500) is a corrosion-resistant nickel-copper alloy that contains aluminum and titanium to form γ' age hardening precipitates of ordered $Ni_3(Al,Ti)$. It is used primarily in the chemical process, marine, and oil/gas industries as tubing, fasteners, pump shafts and impellers, etc. The laser deposition process known as Directed Light Fabrication (DLF) was used to produce fully dense specimens of Alloy K-500 for metallographic inspection and mechanical property analysis, and the wrought counterpart of Alloy K-500 was obtained. A series of heat treatments was performed on all specimens, and mechanical properties were recorded as a function of aging time and aging temperature. In addition, the microstructures were analyzed at various aging times and temperatures. A direct comparison of the mechanical properties between the laser material and the wrought product demonstrated that similar properties could be obtained. The kinetics of aging both the wrought and laser deposited Alloy K-500 were investigated by utilizing the mechanical properties data, since determination of the γ' precipitate size proved to be difficult at early times in aging (i.e., before coarsening). The methodology to study the kinetics of aging was similar to the Johnson-Mehl-Avrami-Kolmogorov (JMAK) kinetics approach, and from the results of this analysis, the mechanisms of aging in both wrought and laser deposited Alloy K-500 at early aging times are presented and discussed.

9:30 AM

In-Situ Alloying Using Freeform Laser Processing: *Ralph E. Napolitano*[1]; John E. Smugeresky[2]; Iver E. Anderson[1]; David M. Keicher[3]; [1]Iowa State University, Ames Lab. Usdoe, Metallu. and Cer., 104 Wilhelm Hall, Ames, IA 50011 USA; [2]Sandia National Laboratories; [3]Optomec, R & D, 2701-D Pan American Freeway, Albuquerque, NM 87107 USA

The flexibility with regard to local alloy composition offered by the freeform laser cladding process make this technique potentially viable for the production of net shape parts with spatially distributed microstructural properties. Such microstructural control requires in-situ alloying through simultaneous laser-melting and deposition of multiple pure-metal powders. In the current study, binary and ternary alloys of Cu, Ni, Sn, and Al are produced using this technique. The coupled effects of alloy composition and laser velocity are investigated, and process limits for producing structurally and chemically inhomogeneous components are determined. The primary microstructural features of interest include solidification structures, local composition distributions, and defects. Research avenues for continued advancement are suggested. This work is supported by USDOE Basic Energy Sciences under contract number W-7405-Eng-82.

9:50 AM

Multi-Stream Casting of Ultra High Strength Sub-Millimeter Diameter Wire: *Ayodele Oladimeji Olofinjana*[1]; Hywel A. Davies[2]; James H. Kern[2]; [1]Queensland University of Technology, Schl. of Mech. Manu. and Med. Eng., Gardens Point 4001, Brisbane, Australia; [2]Uni-

versity of Sheffield, Dept. of Eng. Matls., Mappin St., Sheffield S13JD UK

The direct casting of wire of sub-millimetre dimensions have been developed and are now well established using specialised water bath melt spinning process. A number of compositions with exciting engineering properties such as ultra high strength and unique magnetic behaviour have been produced. Despite these interesting properties and the expected savings in cost and energy for production, the wire casting process has not progressed to commercial scale. The main impediment for commercialisation believed to be related to the slow rate of wire casting, which is restricted to approximately 10m/s by the need to optimise the process parameters. Here, we are reporting the experimental work to explore the attempts that we have made at increasing the wire cast rate using Fe-Si-B alloy We report our work on nozzle designs to accommodate multi-streaming as oppose to single stream as is the current practise. Additionally, the effects of temperature control, on the quality and properties of the wire was investigated. It is shown that cast rates could be increased many folds through multi streaming. The optimised process parameters for nozzle design up to 5 streams will be presented. Melt temperature probing during the casting suggest that to retain amorphous structure, melt superheat should be kept below 100K and it was found that excessive superheat was detrimental to the quality of final wire product.

10:10 AM Break

10:20 AM

The Effect of Cyclic Pressure on the Density Distribution in Metal Matrix Composite Compacts: *Yuehong Fu*[1]; Guangbin Jiang[1]; Glenn S. Daehn[1]; John J. Lannutti[1]; Robert H. Wagoner[1]; [1]Ohio State University, Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

One way that rapid free-form fabrication techniques might be used to develop net-shape structural parts is through fabricating die shapes that could then be used in the fabrication of monolithic or composite powders into compacts that then could be sintered to optimize strength. Low compacted density and density gradients lead to component shrinkage and distortion on sintering. Compaction under cyclic load has been shown to produce superplastic-like effects in the consolidation of mixed metal and ceramic powders. Green compacts with increased green density and mechanical properties can be fabricated using pressure cycling. Some limited prior studies also suggest the density gradients can also be reduced. In this study, mixed powders of Aluminum and Alumina were consolidated under static and cyclic pressure at room temperature. Composite compacts of varied aspect ratio and shape were investigated to find the effect of cyclic pressure on the density distribution. X-ray Computed Tomography was used to evaluate the density gradient after compaction. It was found that the uniformity of density distribution inside composite compacts was greatly enhanced under pressure cycling, especially for those with high aspect ratio.

10:40 AM

Experience with Axisymetric Simulation Using Sheet-S: *Robert J. Comstock*[1]; Kaiping Li[2]; Robert H. Wagoner[2]; [1]Armco Inc., Tech. Svcs., 705 Curtis St., Middletown, OH 45044-3999 USA; [2]Armco Inc., Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210-1179 USA

A collaborative research program is underway to understand and improve the forming of high-chromium ferritic stainless steel sheet for automotive exhaust systems. In order to improve current practice, verification of simulation techniques and accuracy was required. Constitutive equations were measured for three alloys: Type 409, Type 304, and ArmcoTM 18CrCb. Friction coefficients for these alloys with two lubricants were measured using the drawbead simulator (DBS) test. Axisymmetric simulations of standard forming tests, especially the Olsen Cup Test, were carried out using SHEET-S, a two-dimensional finite element program. Experiments were performed to assess the accuracy of the simulations. The comparisons show good agreement in both strain distributions and predicted punch-height-to-failure; the latter based on use of a simple failure criterion.

11:00 AM

Measurement of the Bauschinger Effect in Metal Sheets: *Lumin Geng*[1]; Vijay Balakrishnan[1]; *Robert H. Wagoner*[1]; [1]Ohio State University, Dept. of Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210-1179 USA

Direct measurement of the Bauschinger Effect for sheet metals in the sheet plane is difficult because buckling occurs in compression. For this reason, indirect tests, such as bending or in-plane shear have been used. A uniaxial tension-compression device was designed and constructed to stabilize in-plane compression, and used with correction procedures to measure hardening laws following abrupt reversals. In order to verify materials models generated from the test, the hardening laws were implemented into ABAQUS, a finite element code, and the reverse bend test was simulated. Comparison with reverse bend test results shows that isotropic hardening models fit poorly near the reversal, while two anisotropic hardening model reproduce the data well. The strain range obtainable in the bend experiment is small, +/- 2%. Within the scatter of the experiments, the reverse bend test and tension/compression tests are identical. However, interpretation of the bend test results to obtain corresponding uniaxial stress-strain curves is ambiguous.

International Symposium on Iridium: Processing, Refining, and Chemistry

Sponsored by: Structural Materials Division, Refractory Metals Committee

Program Organizers: Evan K. Ohriner, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; H. Harada, National Research Institute for Metals, Tsukuba, Ibaraki 305 Japan; R. D. Lanam, Engelhard-CLAL, Careret, NJ 07008 USA; Peter Panfilov, Ural State University, Ekatrinburg 62001 Russia

Wednesday AM Room: Jackson A/B
March 15, 2000 Location: Opryland Convention Center

Session Chairs: David F. Lupton, W.C. Heraeus GmbH & Co. KG, Heraeusstrasse 12-14, Hanau D-63450 Germany; Alexander V. Yermakov, Ekaterinburg Non-Ferrous Metals Processing Plant, Rsch. Ctr., Ekaterinburg 620014 Russia

8:30 AM

Fundamentals of Iridium Plastical Treatment Technology: *A. V. Ermakov*[1]; A. V. Sedavnykh[1]; N. I. Timofeev[1]; V. A. Dmitriev[1]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, Lenin Ave. 8, Ekaterinburg 620014 Russia

Thanks to their unique properties, iridium and iridium-alloy products are finding an ever increasing use in modern technology. The range of iridium products is continuously extended. The mass of iridium products varies between ten kilograms or more for crucibles and less than 0.01 grams for disks and samples. Iridium articles are produced mainly by the method of plastic deformation. However, it is at this stage that processing engineers encountered considerable difficulties associated with the mechanism of brittle fracture of iridium, which is anomalous for FCC metals.

8:50 AM

Electroforming of Near-Net Shapes in Iridium: *A. Shchetkovskiy*[1]; A. Etenko[1]; V. Sikin[1]; [1]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

Electroforming of iridium is a highly specialized process for manufacturing near-net shapes of iridium, iridium alloys or iridium composites. The advantages of the process include seamless construction, efficient metal use, and purification of the iridium. Wall thickness can vary from a hundred microns up to several millimeters. The technol-

ogy makes it possible to consider the uses of iridium for new products, previously, not considered because of technological or financial reasons. Various examples of products and product applications will be discussed.

9:10 AM

Welding and Weldability of Thorium-Doped Iridium Alloys: *S. A. David*[1]; Evan K. Ohriner[1]; J. F. King[1]; [1]Oak Ridge National Laboratory, Mets. and Cer. Div., P.O. Box 2008, Oak Ridge, TN 37831 USA

Ir-0.3% W alloys doped with thorium are currently used as post-impact containment material for radioactive fuel in thermoelectric generators that provide stable electrical power for a variety of outer planetary missions. Welding and weldability of a series of alloys was investigated using arc and laser welding processes. Some of these alloys are prone to severe hot-cracking during welding. Weldability of these alloys was characterized using Sigmajig weldability test. Hot-cracking is influenced to a great extent by the fusion zone microstructure and composition. Thorium content and welding atmosphere were found to be very critical. The weld cracking behavior in these alloys can be controlled by modifying the fusion zone microstructure. Fusion zone microstructure was found to be controlled by welding process, process parameters, and the weld pool shape. The paper will discuss in detail the inter-relationship between the process-microstructure and weldability of iridium alloys.

9:30 AM

Iridium Refining: *J. D. Ragaini*[1]; [1]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

The superior corrosion resistance and high temperature performance of iridium make it the material of choice for the severe conditions of many industrial processes despite the cost of the metal. Refiners of precious metals strive to satisfy these commercial demands by treating primary ores and recycled scrap to produce purified iridium of acceptable quality. The treatments can be fairly straightforward or quite complex, depending on the nature of the material fed into the refinery. Some of these processes are described.

9:50 AM

Recovery and High Refinement of Iridium: N. I. Timofeev[1]; *A. V. Yermakov*[1]; V. I. Bogdanov[1]; G. F. Kuzmenko[1]; L. D. Gorbatova[1]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia

The new universal technology for extraction of iridium from primary concentrates and refining secondary iridium has been elaborated. Hydrometallurgical means allow increasing contents of iridium in ´poor[a] concentrates up to 98%. After that primary and secondary iridium are directed to the pyrometallurgical processing included itself oxidation melting of scrap in periclase magnesia crucible and electron-beam melting. The final stage of refining is growing massive single crystals by means of electron-beam zone melting. Resulted metal with purity of 99.95% is high pure plastic iridium, which could be processed without considerable troubles.

10:10 AM Break

10:20 AM

The Monitoring of the Speciation of Iridium in the Separation of Iridium from Rhodium: *M. J. Nicol*[1]; [1]Murdoch University, AJ Parker CRC, South St., Murdoch, Western Australia 6150 Australia

The separation of iridium from rhodium in chloride solutions during the refining of platinum metal concentrates is one of the most difficult steps and is generally carried out using either solvent extraction or ion exchange. In both cases, the separation process makes use of the fact that the hexachloroiridium (IV) ion is less strongly hydrated and more polarizable than the hexa- or penta-chlororhodium (III) species. This results in either selective extraction into solvating solvents or selective adsorption onto anion exchange resins of the iridium. It is obvious that control of the speciation is crucial in ensuring efficient separation. This paper will describe the use of relatively simple macro- and micro-electrode cyclic voltammetric techniques for the rapid identification of the iridium species present in solutions obtained during various stages of the conditioning process prior to separation by ion exchange.

10:40 AM

Fluorination of Iridium Metal and Its Application Possibilities in the Synthesis, Analysis and Recovery Technology for Secondary Raw Materials: *V. N. Mitkin*[1]; [1]Institute of Inorganic Chem. SB RAS, 3, Lavrentjeva Ave., Novosibirsk 630090 Russia

The basic regularity in oxidizing fluorination of iridium and its mixes with alkali metal fluorides by various gaseous, liquid or molten reactants-F_2, ClF_3, BrF_3 and $KBrF_4$ has been studied. It is proved that gas-phase processing at 270-350°C or the treatment by molten $KBrF_4$ at 350-450°C always results in formation of Ir(V) hexafluorocomplexes-$MIrF_6$. It has been also determined that the most convenient fluoroxidant in its common sense for transfer of the compact metal into the oxidised state is a liquid BrF_3. The product of Ir interaction with BrF_3 is hexafluoroiridate(V) of difluorobromonuim (III)-$[BrF_2]^+[IrF_6]^-$. The features of Ir dissolution kinetics in a liquid bromine trifluoride at 25-65°C are determined by presence in oxidant of additives of bromium and also availability of the ionogenic forms. Thus, the Lewis acids accelerate the dissolution of iridium whereas the donors of fluoride-ion decelerate the process. The bromium also slows the process at 25-38°C. Apparent activation energies of the processes are 18.9 at 25-38°C and 14.1 kcal/mol at 38-65°C. The specific rate of Ir dissolution in pure BrF_3 is 6.2 x 10^{-7} g-atom/cm_2 min. The processes of iridium oxidizing fluorination were evaluated for analytical purposes. It is proved that the application of bromine trifluoride or potassium tetrafluorobromate can be a unified method of chemical sample preparation. The methods of oxidising fluorination of secondary iridium raw materials permit 98-99% metal recovery and are suitable to practical use.

11:00 AM

Synthesis and Physical-Chemical Investigation of Iridium (III-V) Fluorocomplexes: *V. G. Isakova*[1]; L. M. Levchenko[1]; V. N. Mitkin[1]; [1]Institute of Inorganic Chemistry, Sbras, 3 Lavrentjeva Ave., Novosibirsk 630090 Russia

The processes of iridium metal and iridium (IV) hexachlorocomplexes oxidation by a gaseous F_2 and ClF_3 (p = 1 atm) are studied with an application of the specially designed variants of thermography and thermogravimetry. There are determined an optimal conditions of hexafluoroiridate (VI and V) synthesis. Novel less-known hexafluorocomplexes of Ir(III)-M_3IrF_6 (K, Rb, Cs) have been synthesized under hetero-phase reduction from appropriate complexes M_2IrF_6. All iridium fluorides are characterized by the potentiometry, IR-spectroscopy, X-ray diffraction and elemental analysis. The methods NMR ^{19}F and spectrophotometry have been applied to the study of the transformations of the ions $[IrF_6]^{2-}$ and $[IrF_6]^{3-}$ in solutions under «Ox-Red» and ligand-exchange processes. It has been established that in more labile $[IrF_6]^{3-}$ ions as contrasted to $[IrF_6]^{2-}$ ions there are always taken place the aquatation process with the formation of mixed-ligand complexes $[IrF_{6-n}(H_2O)_n]^{n-3}$ instead the iridium (III) fluorochloro-complexes. The novel iridium (III) crystalline complex $[Ir(H_2O_6]F_3$-3HF has been separated in a solid state for the first time. These results have been applied to preparative chemistry and to the development of original direct high-yield synthesis of the various coordination compounds of iridium using hexafluoroiridate (III and IV) starting materials.

11:20 AM

The Thermoanalytical Study of Ir(III)-Beta-Diketonate's Behaviour in Inert, Oxygen and Hydrogen Atmospheres: *V. N. Mitkin*[1]; V. G. Isakova[1]; [1]Institute of Inorganic Chemistry, Sbras, 3 Lavrentjeva Ave., Novosibirsk 630090 Russia

There are investigated the thermal behaviour of the volatile beta-diketonates of Ir(III) Ir(R'COCHCOR")$_3$ (1) and Ir $(CH_3COCHalCO-CH_3)_{3-n}(CH_3COCHCOCH_3)n_{(2)}$, where «n» = 0,1,2; Hal = Cl, Br, I; R ' = CH^3, CF^3; R " = CH^3, CF^3, C $(CH^3)^3$ in an inert and oxidative or reductive gaseous atmosphere. The thermal decomposition processes of (1) and (2) have been studied by means of Hungarian thermoanalyser Q-1000 in an atmosphere of He, H2, and O2. The resulting and intermediate solid products yielded after thermal «Ox-Red»-transformations at the controlled gaseous media (at atmospheric pressure) and at the selected temperatures have been studied by physical-chemical methods. It has been established that the series of compounds (1) represent a higher thermal stability in hydrogen atmosphere than for thermal decompo-

sition in He. At the same time the temperatures of transformations in oxygen atmosphere are essentially reduced for this series of compounds. The comparative analysis of the volatility and thermal stability of (1) and (2) has been conducted. It has been proved, that (2)-an iridium (III) halogenated acetylacetonates have increased volatility as compared to appropriate acetylacetonates of iridium (III). It has been proved that acetylacetonates of Ir (III)-Ir(R'COCHCOR")3 are thermally decomposed with the selection of metal, but the halogenated acetylacetonates (2) decomposed with the formation of binary iridium halogenides.

11:40 AM

Iridium Oxide-Excellent Electrode Material to Industrial Electrolysis: *T. Shimamune*[1]; [1]Furuyametals Company Limited, MSB-21, Minamiohtsuka Bldg., 37-52, Toshima, Tokyo 170-0005 Japan

Since the great invention of Dr. Beer, in 1965, ruthenium and iridium became popular in the electrochemical field as DSA, where they are used as oxide coating on titanium. The first commercial application of it was to the mercury process chlor-alkali electrolysis, where ruthenium oxide was used with titanium oxide. According to the process conversion to membrane system, higher durability and reaction selectivity became required to DSA, where a combination of ruthenium and iridium oxides was applied. More than 10 years of life and low O1/C12 was attained with lower power consumption of 2,000 kWh/ton-NaOH. With further conversion to the membrane process world iridium demand will become at least 600 kg/year. Several years after the commercialization in chlor-alkali, iridium oxide DSA was commercialized as the oxygen evolution anode. That was successfully applied in Japan at first and proved to be far superior to the conventional platinum based or lead alloy anodes. The application is increasing together with improvements in durability. The service life has increased from 1000 hours in standard sulfuric acid electrolysis to longer than 10,000 hours at present. Application was also made to Cu-foil production, electro-galvanizing, electro-tinning, metal winnings, etc., with tremendous contribution to product quality, energy savings, and environmental impact. Thus, the iridium oxide plays an important role in industrial electrolysis, and the demand for this use is increasing year by year.

12:00 PM

On the Possibility of Electrochemical Separation of Precious Metals during Processing of Heavily Contaminated Starting Materials of Iridium: *A. V. Yermakov*[1]; V. I. Bogdanov[1]; L. D. Gorbatova[1]; K. P. Konik[1]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, Lenin Ave. 8, Ekaterinburg 620014 Russia

An intermediate product-technical-grade ammonium chloroiridate-is formed during the refining of placer platinum. Ammonium chloroiridate contains 20 to 40% iridium, up to 10% of other platinoids, and the same percentage of base elements, mainly copper, lead, iron, and silicon. It was a common practice to transfer these materials to pyrometallurgical processing. The resulting sublimates underwent hydrometallurgical. However, this technological scheme has some drawbacks, primarily inadequate selective extraction of such metals as Pt and Rh. Several hydrometallurgical schemes are known to separate iridium from the aforementioned starting material. Separation of rhodium and iridium presents the most difficult task in refining iridium. Until recently, no satisfactory method for the separation of the two elements existed.

Liquid Metal Atomization: Fundamentals and Practice: Gas Atomization

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee

Program Organizers: Khershed P. Cooper, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5343 USA; Frank Biancaniello, NIST, Gaithersburg, MD 20899-8556 USA; Stephen D. Ridder, NIST, Gaithersburg, MD 20899-8556 USA

Wednesday AM — Room: Bayou B
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: Stephen D. Ridder, National Institutes of Standards & Technology, Gaithersburg, MD 20899-8556 USA; Stephen J. Mashl, Bodycote IMT, Andover, MA 01810 USA

8:30 AM Invited

Gas Atomization Processing with High Efficiency and Size Control: *Iver E. Anderson*[1]; R. L. Terpstra[1]; Jason Ting[2]; C. Yu[3]; [1]Ames Laboratory, 126 Metals Dev., Ames, IA 50011 USA; [2]Crucible Research, Pittsburgh, PA 15205 USA; [3]Delevan, West Des Moines, IA 50265 USA

Many applications for particulate materials demand either the availability of fine (dia. < 45 micron) powders or coarser powders of controlled size. High pressure gas atomization (HPGA), a close-coupled, discrete jet atomization method, has proved to be one of the most effective methods of producing rapidly solidified fine metal and alloy powders with high yields less than 20 microns using Ar, N_2, or He gas. Development of HPGA jets with convergent-divergent (C-D) rocket nozzle designs that have high total kinetic energy was conducted to increase atomization efficiency and uniformity. Efficient use of C-D jets in an atomization nozzle required development of a physical model of the gas dynamics in the atomization zone. An unexpected benefit has been the ability to operate in a nearly constant aspiration mode over an extremely wide pressure range, advantageous for powder size control. Examples of powder size control and comparisons of efficiency will be described. Different aspects of this work were supported by USDOE-BES under contract no. W-7405-Eng-82, the Center for Advanced Technology under DOC contract no. ITA81-02, and the Iowa State University Research Foundation.

9:00 AM Invited

Comparison of the Supersonic Length and Dynamic Pressure Characteristics of Discrete-Jet and Annular Close-Coupled Nozzles Used to Produce Fine Metal Powders: *Steven P. Mates*[1]; Stephen D. Ridder[1]; Frank S. Biancaniello[1]; [1]NIST, 100 Bureau Dr., Stop 8556, Gaithersburg, MD 20899-8556 USA

Well-designed close-coupled nozzles operating at high gas pressures to atomize molten metal into fine powder generally produce a long supersonic gas jet. The magnitude of the dynamic pressure (density times velocity squared) developed in this gas jet, as well as its supersonic length, strongly affect the critical secondary atomization stage, in which molten droplets are disintegrated into fine particles over long distances from the nozzle tip. Higher dynamic pressures and longer supersonic lengths will tend to produce more complete secondary atomization, yielding a smaller average particle size. As such, these two gas dynamic characteristics may be used as benchmarks to compare the potential atomizing capability of one close-coupled nozzle versus another. In this study, the supersonic length and dynamic pressure characteristics of two discrete-jet close-coupled nozzles having different overall diameters are reported for several nozzle pressure ratios. Different nozzle diameters are included to study how nozzle scale affects the supersonic jet behavior. The discrete jet nozzle data are then compared to data obtained from annular converging and converging-

diverging close-coupled nozzles to gauge their relative performance. Finally, the close-coupled nozzle data are compared to round, perfectly expanded supersonic jets, which are the narrowest and longest possible jets at any given pressure ratio and gas flow rate.

9:30 AM Invited

Improvements in Close-Coupled Atomization: An Empirical Approach: *Joseph T. Strauss*[1]; [1]HJE Company Inc., 151-155 Maple St., Glens Falls, NY 12801-3729 USA

The development of a small-scale close-coupled atomization system has evolved incrementally with goals of increasing the reliability, repeatability, predictability, and performance. Reliability and repeatability have been found to be primarily functions of component design and operational parameters. Insight into performance and predictability were gained by a combination of flow bench testing and correlation of particle size data with gas to metal flow rate ratio, gas momentum, and gas energy. This paper will review prior studies investigating the effects of melt and gas properties and operational parameters on particle size. The merit of gas-only aspiration tests and water bench testing will be discussed. Salient design details are reported.

9:55 AM Invited

Understanding the Liquid Metal Atomizer Performance and Behavior: *Leonel L. Núñez*[1]; Rodrigo H. Palma[2]; A. Sepúlveda[2]; [1]Universidad Central de Chile, Facultad de Ciencias Físicas y Matemáticas, Escuela de Ingeniería Civil en Obras Civiles, Sede Parque Almagro Sur, Santa Isabel, Santiago 1186 Chile; [2]Universidad De Chile, Dept. De Ingeniería Mecanica, Casilla, Santiago 2777 Chile

It is recognized that the performance of gas liquid metal atomizers is a function of the following variables: atomization gas flow, liquid metal flow, productivity and size distribution of powders. However, most of the studies of confined and gravity atomizers show that the aerodynamic pressure field, developed by the interaction between gas jets, controls the behavior and performance of them. On the other hand beyond the efforts to make predictions about behavior and performance of atomizers, there are no reliable equations to make design calculations. It is well known that atomizers configured with independent or annular nozzles develop choked flow, a condition suitable to evaluate the gas flow with compressible flow equations. To estimate the particle mass median diameter, the Lubanska equation has been used; however many researchers have demonstrated that this experimental correlation does not give good results, particularly for confined atomizers. It seems that a key for the comprehension of confined and gravity atomizers' performance is the understanding of the aerodynamic pressure field. In this area, in spite of the great quantity of published experimental results for several atomizer types, no efforts have been done to systematize the information in order to propose theoretical models that explain the pressure field formation. In this work, the general equations of gas/liquid metal-atomizer are developed on the basis of fundamental principles and experimental results. The atomization gas flow and the liquid metal flow are evaluated from fluid dynamics principles. It is worth noting that the liquid metal flow equation, an important equation to estimate the atomized powder size, is a function of the aerodynamic pressure field. A procedure to analyze reported experimental pressure fields is developed. The purpose is to contribute to the understanding of the principal controlling variables and to the theoretical prediction of pressure fields. Moreover, a multivariate analysis of powder-size experimental data is conducted to propose corrections to Lubanska's equation. In this case, the objective is the development of an equation to evaluate the particle mass median diameter for several atomization regimes.

10:20 AM Break

10:35 AM

The Possibility of Narrow Size Distribution in Gas Atomised Powders by Nozzle Design Modifications: *Sedat Özbilen*[1]; [1]Gazi University, Metallu. Edu. Dept., Teknikokullar, Ankara 06500 Turkey

Mg powders under low pressure Ar gas and Al powders under Ar gas with nozzle design modifications were produced on a pilot plant gas atomiser. Mg and Al powders produced were characterised by SEM investigation and by laser particle size analysis. It was observed that the powders produced obeyed log-normal size distribution law. Their frequency distribution curves also indicated bi-modal size distribution in the powders. Detailed SEM investigation and particle size distribution analysis indicated narrow size distribution in the powders. The effect of the atomising gas pressure and nozzle design modifications were used to explain the observed tight size distribution both in the Mg and Al powders produced.

11:00 AM Invited

An Investigation into the Processing and Properties of Gas Atomized High Nitrogen Austenitic Stainless Steels: *Frank S. Biancaniello*[1]; R. D. Jiggetts[1]; Stephen D. Ridder[1]; Rick E. Ricker[1]; Mark R. Stoudt[1]; [1]NIST, 100 Bureau Dr., Stop 8556, Gaithersburg, MD 20899-8556 USA

Nitrogen containing stainless steels are known to possess improved properties. The consistent production of alloys with these superior properties is enhanced by rapid solidification processing which eliminates the macrosegregation that inhibits consistently obtaining outstanding properties in these alloys through ingot processing. To evaluate the extent to which gas atomization enables the production of nitrogen bearing alloys with improved properties, a series of alloys was produced using a predictive model to guide alloy and process design, and then, the mechanical, corrosion and stress corrosion properties of these alloys were evaluated. The model used to predict nitrogen solubility and alloy properties also led to a reduced quench rate sensitivity. This property in turn allows for the atomized powders or subsequently extruded wire to be used for thermal spray applications. These applications will be discussed along with the results of the mechanical and corrosion properties.

11:25 AM Invited

NANOVAL Atomizing: A Special Process for Special Products: *Martin Stobik*[1]; [1]NANOVAL GmbH & Company KG, Holzhauser Str. 157-159, Berlin D-13509 Germany

The Nanoval process differs from all other ways of atomizing as a melt stream bursts open by itself when its inner pressure, given by surface tension forces, surpasses the outer pressure of a steadily accelerated gas flow in Laval nozzle. This Nanoval effect is observed in laminar flow of both, melt and gas. Its consequence is fine and ultra-fine spherical powder at particle size distributions narrower than usual. A new autoclave system omits plug rods in using a hydraulic closure unit, which enables continuous or semi-continuous production. Atomizing results are shown for the production of fine and ultra-fine powder of various metals.

Magnesium Technology 2000: Creep Properties and Heat Treating Effects

Sponsored by: Light Metals Division, Reactive Metals Committee, International Magnesium Association
Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA

Wednesday AM Room: Bayou D
March 15, 2000 Location: Opryland Convention Center

Session Chair: Darryl L. Albright, Hydro Magnesium, Hydro Light Metals, Livonia, MI 48152 USA

8:30 AM

Tensile and Compressive Creep Behavior of Die-Cast Magnesium Alloy, AM60B: *S. R. Agnew*[1]; K. C. Liu[1]; S. Viswanathan[1]; [1]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

There has recently been renewed interest, on the part of the automotive industry, in increased usage of magnesium based alloys in an effort to improve efficiency through vehicle mass reduction. One of the limitations of magnesium alloys that has hindered more widespread application is their poor creep resistance. In an effort to determine the best course for developing die-cast alloys with improved creep resistance, the creep behavior of existing commercial alloys is being studied. Of the two most commonly used die-casting alloys, AZ91 and AM60, the former is stronger but the latter is tougher. For many applications under consideration, such as bolted housings and covers, the critical loads are compressive. In spite of this fact, most creep testing has been performed in a tensile mode. In this study we compare the tensile and compressive creep behavior of AM60. Similar to earlier studies of magnesium alloys creep behavior at temperatures in the range of 0.7Tm, the stress dependence of the steady state creep rate, n~5 (stress range 20-60 MPa). Interestingly, the creep strength in compression is significantly higher than that in tension.

8:55 AM

Development of High Creep-Resistant Magnesium Alloy Strengthened by Ca Addition: *Toshio Horie*[1]; Hiroaki Iwahori[1]; Yoji Awano[1]; Yoshiki Seno[1]; [1]Toyota Central R&D Laboratory Inc., Lightwt. & Environ. Matls. Lab., 41-1 Aza-Yokomichi, Oasa-Nagakute, Nagakute, Aichi 480-1192 Japan

By the addition of calcium, mishmetal and zirconium, a low cost Mg-2%Zn-0.8%Ca-2%Mm -0.5%Zr alloy with excellent heat resistance was developed for utilization in automotive parts in the engine compartment. This new alloy shows high creep resistance. The creep property of a high-pressure die-casting of the alloy at 150° under 64 MPa was nearly equal to that of conventional heat resistant alloys such as T6 treated QE22 alloy. The minimum creep rate of the alloy developed in this study was 1/100 of that of AZ91 alloy under the same condition. The ultimate tensile strength was over 200 MPa at 150°. This value is nearly equal to that of T6 treated QE22 and 1.2 times as high as that of AZ91 alloy. The breaking elongation at room temperature was 6%, which is higher than that of AZ91 alloy. Moreover, its mechanical properties are improved significantly by T6 treatment, due to the formation of fine precipitates at low temperatures.

9:20 AM

The Effect of Calcium on Creep and Bolt-Load Retention Behavior of Die-Cast AM50 Alloy: *Keun Yong Sohn*[1]; J. Wayne Jones[1]; John E. Allison[2]; [1]University of Michigan, Dept. of Matls. Sci. and Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]Ford Motor Company, Matls. Sci. Dept., Scientific Rsch. Lab., Dearborn, MI 48121 USA

Magnesium die-casting alloys for automotive transmission case applications require good creep resistance at elevated temperatures. While magnesium alloys such as AE42 have been shown to have acceptable creep resistance, concerns about alloy cost limit wide spread use. Lower cost approaches for creep resistant magnesium alloys are currently under investigation. It has been shown that small additions of calcium greatly improves the elevated creep resistance of magnesium die-castings. In this study, the effect of calcium on creep and bolt-load retention (BLR) behavior at various temperatures and stresses/preloads will be investigated. Four alloys with different calcium content (0, 0.25, 0.50, 0.75%) in base AM50 alloy were die-cast. Creep tests have been carried out at a temperature of 150°C and initial stresses from 30 to 90 MPa. BLR tests have been carried out from 125 to 175°C and preloads from 14 to 28kN. Higher calcium content alloys showed better stress relaxation and creep resistance; AM50 alloy with 0.75% Ca (AMC5007) showed the best BLR property, with a higher BLR resistance than AE42. Similarly, the AMC5007 alloy showed the lowest creep strain of the AMC alloys. TEM investigation of as-cast and crept specimens will be described.

9:45 AM

Creep Resistant Mg-Al-Ca Casting Alloys: *Mihriban O. Pekguleryuz*[1]; Jean Renaud[2]; [1]Noranda Technology Center, 240 Hymus Blvd., Pointe-Claire, Québec H9R1G5 Canada; [2]Intermag Technologies, 357 Rue Franquet, Ste-Foy, Québec G1P4N7 Canada

The automotive use of magnesium is currently restricted to non-structural components. Its use in critical structural components such as transmission and engine parts requires the development of cost-effective alloys that can meet the performance requirements of these components for elevated-temperature (150°C) strength and creep resistance. This study is on the development of a Mg-Al-Ca alloy system that has good creep-resistance at 150°C. The increased creep resistance of the alloy is due to the existence of an Al2Ca intermetallic compound in the as-cast structure. Microstructural investigation of the alloy before and after creep loading shows the role of microstructure in creep resistance. The tensile yield strength and the ultimate tensile strength of the alloy at 150°C both in the permanent-mold cast and diecast state are equivalent to the more expensive rare-earth containing magnesium alloys. Corrosion resistance of the diecast alloys at 0.11- 0.23 mg/cm2/day, as measured through salt-spray corrosion test, falls in the range of high purity magnesium alloys AZ91D and AM60B and the rare-earth containing AE42 alloy.

10:10 AM Break

10:20 AM

Preparation and Solidification Features of As Series Magnesium Alloys: *B. Bronfin*[1]; M. Katsir[1]; E. Aghion[1]; [1]Dead Sea Magnesium Limited, P.O. Box 75, Beer-Sheva 84100 Israel

AS magnesium alloy series are used for high temperature applications that require adequate creep resistance. The aim of the present investigation was to obtain information regarding preparation procedure and phase constituents that are precipitated during solidification of AS21 and AS41 alloys and can affect the performance of these alloys in service conditions. Magnesium alloys are usually alloyed with manganese to remove iron. Hence, the understanding of the factors which influence the mutual solubility of Mn and Fe in molten magnesium is very important for establishing the efficient alloying procedure by Mn and other elements, particularly by Si in AS21 and AS41 alloys. Silicon is added to Mg-Al alloys in order to enhance the creep resistance. It was believed that only Mg_2Si particles are formed through the solidification process of AS21 and AS41 alloys. However, it is evident from the results of present investigation that additional phases are present in the AS21 and AS41 ingots. The sequence of phase transformations occurring through the solidification process is discussed and the factors affecting the microstructure features were disclosed.

10:45 AM

On the Relation between Hardness and Yield Strength in a Sand Cast AZ91 Alloy: Clare L. Bancroft[1]; *Carlos H. Cáceres*[1]; John R. Griffiths[2]; [1]University of Queensland, Dept. of Ming., Min. and Matls. Eng., CRC for Alloy and Solid. Tech., Brisbane, QLD 4072

Australia; [2]CSIRO Manufacturing Science and Technology, P.O. Box 883, Kenmore, QLD 4069 Australia

The effect of aging time at 165°C on the mechanical properties of sand cast AZ91 alloy has been studied. The hardness, Hv, and yield strength, YS, increase with the aging while the tensile ductility decreases. It is possible to fit the flow curves of the material to the equation $\sigma = K \varepsilon^n$, where σ is the true stress and ε the true plastic strain, with a single K-value (570 MPa) and varying the strain hardening exponent, n, according to the alloy temper. The n-value is obtained from the Vickers hardness number with the expression $n = 1.382 - 0.265 \ln(Hv)$ and used to calculate yield strength as $YS = 568.9 (0.002)^n$.

11:10 AM

The Effect of Low-Temperature Aging on the Tensile Properties of High-Pressure Diecast Mg-Al Alloys: *Amanda Lee Bowles*[1]; John R. Griffiths[3]; P. D. D. Rodrigo[3]; Cameron J. Davidson[3]; Tim J. Bastow[2]; [1]The University of Queensland, Co-op. Rsch. Ctr. for Alloy and Solid. Tech., Brisbane, Queensland 4072 Australia; [2]CSIRO, Manu. Sci. and Tech., Private Bag 33, Clayton, Victoria 3169 Australia; [3]CSIRO, Manu. Sci. and Tech., P.O. Box 883, Kenmore, Queensland 4069 Australia

Data are presented which are relevant to the use of magnesium alloys in the engine bay and interior of automotive vehicles where metal temperatures for typical applications can reach 120°C. In this research, high-pressure die-castings of the Mg-Al alloys AZ91D and AM60B have been aged at 120°C for times of up to 5,000 hours. Results are reported for the effect of this aging on the tensile properties and on the microstructure. Increases in yield stress of up to 30 MPa were noted for 5 mm thick castings but the increase for 2 mm thick castings was far less, at about 6 MPa. The ductility of all castings was reduced by more than half. Observations of the microstructure have been carried out by optical and electron microscopy and by nuclear magnetic resonance (NMR). These have shown that precipitation of Mg17Al12 has taken place during aging and it is inferred that this has been the cause both of the increase in strength and the decrease in ductility.

11:35 AM

Study of the Effect of Heat Treatment on the Microstructure and Mechanical Properties of a Thixoformed AZ91Alloy: *Enrico Evangelista*[1]; Marcello Cabibbo[1]; Stefano Spigarelli[1]; Pasquale Cavaliere[1]; Michael Talianker[2]; Volodia Ezersky[2]; [1]University of Ancona, Dept. of Mech., Via Brecce Bianche, Ancona 60131 Italy; [2]University of Negev, Dept. Matls. Sci. and Eng., P.O. Box 653, Beer-Sheva, Negev 84105 Israel

The present study is focused on the structural and mechanical properties of an AZ91 Mg-alloy after thixoforming. The microstructure consisted of large alpha-phase globules separated by quasi-eutectic (alpha+beta). Observations showed that the alpha-Mg areas are developed into individual grains, while the beta-phase (Mg17Al12) particles are present only in the eutectic area. Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) revealed small Mg-rich particles inside the eutectic (divorced eutectic). The heat-treatment response of the alloy was investigated after a solution treatment at 415°C for times ranging 0.5h to 24h. SEM and Light Microscope (LM) showed that the original microstructure produced by thixoforming was almost completely transformed in a conventional structure of equiaxed grains after 2h at 415°C, even if a 4h treatment produced a more homogeneous structure. Solution heat treatment at 415°C produced an equiaxed structure of alpha grains; in particular after 24h aluminium was completely in solid solution, its distribution being substantially homogeneous. The distribution of Al and Zn in the microstructure was investigated by means of X-rays diffraction. The effect of solution treatment-time was investigated by means of tensile tests; the poor ductility typical of the thixoformed alloy was substantially improved by increasing the heat treatment duration over 2h. Additional studies were carried out in order to assess the creep response of the solution-treated material.

Magnesium Technology 2000: Solidification

Sponsored by: Light Metals Division, Reactive Metals Committee, International Magnesium Association
Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA

Wednesday AM Room: Bayou C
March 15, 2000 Location: Opryland Convention Center

Session Chair: Peter Pinfold, Fluor-Daniel, c/o Doe Run Peru, La Oroya Peru

8:30 AM

Eutectic Growth Morphologies in Magnesium-Aluminium Alloys: *Mark Denis Nave*[1]; Arne Kristian Dahle[1]; David Henry StJohn[1]; [1]University of Queensland, Dept. of Ming., Min., and Matls. Eng., CRC for Alloy and Solid. Tech., Brisbane, Queensland 4072 Australia

The Mg-$Mg_{17}Al_{12}$ eutectic exhibits a wide range of morphologies depending on the alloy composition and cooling conditions. An alloy of eutectic composition, Mg-33 wt% Al, exhibits a lamellar morphology at low growth rates and a fibrous morphology at higher growth rates. However, in low aluminium content magnesium-aluminium alloys such as the most common commercial magnesium alloys AZ91 and AM60, the eutectic has a different morphology, described as either completely or partially divorced. This paper reports the results of an investigation into the effect of aluminium content and cooling rate on the morphology of the eutectic in permanent mould cast magnesium-aluminium alloys. Magnesium alloys with aluminium contents ranging from 9 to 33 wt% were cast into steel moulds. The effect of aluminium content was determined by comparing equivalent regions in the different castings while the effect of cooling rate was determined by comparing the microstructures obtained at the edge and the centre of each casting. As the aluminium content of the alloys increased, the dominant eutectic morphology changed progressively from fully divorced to partially divorced, to granular, to fibrous and, finally, to lamellar. Increasing the cooling rate produced a small change in the opposite direction with these transitions occurring at higher aluminium contents. The influence of both aluminium content and cooling rate on eutectic morphology results from the effect of these variables on dendrite morphology and the consequential size of the eutectic regions.

8:55 AM

The Role of Zinc in the Eutectic Solidification of Magnesium-Aluminium-Zinc Alloys: *Mark Denis Nave*[1]; Arne Kristian Dahle[1]; David Henry StJohn[1]; [1]The University of Queensland, Dept. of Ming., Min. and Matls. Eng., CRC for Alloy and Solid. Tech., Brisbane, Queensland 4072 Australia

Previous experimental work has suggested that the addition of zinc to hypoeutectic magnesium-aluminium alloys promotes the formation of a divorced eutectic. However, the amount of zinc required to produce this effect has not been quantified and the mechanism by which it occurs has not been determined. This paper reports the results of a study of the effect of zinc on eutectic morphology in permanent mould cast hypoeutectic magnesium-aluminium alloys, including the commercial alloy AZ91. The results show that the strength of this effect varies with aluminium content and cooling rate. The addition of 1.6 wt% Zn to a Mg-9Al alloy cooled at approximately 80 K/s was sufficient to cause a fully divorced eutectic to form when a partially divorced eutectic formed in the binary alloy solidified at the same rate. An addition of 1.6 wt% Zn to a Mg-15Al alloy cooled at approximately 20 K/s was sufficient to cause a partially divorced eutectic to

form when a granular eutectic was observed in the binary alloy solidified at the same cooling rate. Directionally solidified samples of AZ91 were used to investigate the segregating behaviour of zinc during solidification. The effect of zinc on the solidification behaviour of hypoeutectic Mg-Al-Zn alloys, and its relation to and dependence on cooling rate, is discussed.

9:20 AM

Solidification Induced Inhomogenities in Magnesium-Aluminium Alloy AZ91 Ingots: *Per Bakke*[1]; Carl Fuerst[2]; Hakon Westengen[1]; [1]Norsk Hydro, Rsch. Ctr., Porsgrunn N-3901 Norway; [2]General Motors, Global R&D Operations, 30500 Mound Rd., Warren, MI USA

Magnesium alloys for die casting are commonly based on addition of aluminium as the main alloying element. Due to non-equilibrium solidification, even alloys with aluminium contents well below the maximum solid solubility will contain a certain fraction of eutectic phase. In the later stages of solidification, the presence of low-melting point eutectics, combined with pressure gradients set up by solidification shrinkage, will cause macrosegregation. Element distribution over cross sections of magnesium alloy AZ91 ingots have been investigated. It is found that Al and Zn are strongly depleted in certain regions. Impurities redistribute according to their solid solubilities. Porosity as function of location in the ingots has been examined, and porosity is found to relate closely to the distribution of Al and Zn. The porous Al and Zn depleted regions coincide with the regions solidifying last. This can be explained by inverse segregation as the melt enriched in alloying elements tend to be drawn outwards to colder regions due to shrinkage creating an interdendritic flow, leaving behind an interdendritic porous network depleted in Al and Zn.

9:45 AM

Grain Refinement of Magnesium: *Young C. Lee*[1]; Arne K. Dahle[1]; David H. StJohn[1]; [1]The University of Queensland, CRC for Alloy and Solid. Tech., Ming., Min. and Matls. Eng., Brisbane, Queensland 4072 Australia

Grain formation during solidification of magnesium and Mg-Al alloys has been studied with a focus on grain refinement mechanisms, solute and particle effects. The variation in grain size with increased aluminium content in hypoeutectic Mg-Al alloys showed a continuous decrease in grain size up to 5 wt% Al, and a stabilisation at higher Al contents (above 5 wt%). Strontium additions to both low- and high-aluminium content magnesium alloys showed that Sr had a significant grain refining effect in low-aluminium containing alloys. However, strontium had a negligible effect on grain size in the Mg-9Al alloy. Additions of Zr, Si, or Ca to pure magnesium produced significant grain refinement, probably because these elements have high growth restriction effects during solidification. An attempt was made to identify the grain refinement effect of particles added directly to the melt that are considered to be powerful nucleants in Al based alloys (TiC) and in Mg based alloys (AlN, Al4C3). Most of these particles produced grain refinement, probably because of enhanced nucleation due to the small lattice disregistry between their crystal structures and that of magnesium. However, it is not clear whether the grain refining mechanism of the effective particles was catalysis of primary crystal nucleation or simply restriction of crystal growth during solidification.

10:10 Break

10:20 AM

Stress Induced Defect Formation in DC Cast Magnesium Alloys: *John F. Grandfield*[1]; Arne K. Dahle[2]; [1]CRC for Alloy and Solidification Technology, CSIRO Manufact. Sci. & Tech., Cnr Albert & Raglan Sts., Preston, Victoria 3072 Australia; [2]The University of Queensland, Dept. of Ming., Min. and Matls. Eng., CRC for Alloy and Solid. Tech., Brisbane, Qld 4072 Australia

Magnesium alloys are currently receiving significant interest worldwide for application in automotive components. Because casting is a major production step it is important to understand the solidification of these alloys. Defects in many casting processes are related to the properties and deformation of the partially solidified material. In the case of horizontal direct chill (HDC) casting of magnesium, for example, surface cracks may appear when the partly solid shell ruptures, and classic DC casting hot cracks may also form at the centreline. In order to understand and eliminate these defects the mechanical properties of the partially solidified material, particularly in tension, need to be determined. Work has therefore been undertaken to measure and predict the tensile properties of solidifying, as well as reheated and remelted, pure magnesium and Mg-Al alloys above and below the solidus temperature. The results of surface crack and centreline crack examinations of HDC cast material are compared to the hot tensile test data. The measurements indicate the significance of the mechanical behaviour during solidification on defect formation. Strengthening mechanisms of the mushy zone in magnesium alloys are discussed.

10:45 AM

Casting of Granulated Magnesium and Magnesium Alloys by Centrifugal Spraying of Liquid Metal: Advantages and Limitations: *I. Barannik*[1]; V. Alexandrov[1]; I. Komelin[1]; [1]State Research and Design Titanium Institute, 180 Prospect Lenina, GSP-314, Zaporozhye 330600 Ukraine

Magnesium, the most efficient and environmentally friendly agent for hot metal desulfurization is winning new markets in Northern America, Europe, and Asia. The most economical process of those known in the art is the process for desulfurization based on injecting pure granulated magnesium (without such additives as CaO and CaCl2) to hot metal. The method for casting the granules of magnesium and magnesium alloys developed in the Titanium Institute for this purpose, has been recently improved. In Ukraine, the process has been mastered for the production of magnesium granules from secondary magnesium alloys of Az80 AZ91 grades. Two plants in China operate the units for the production of magnesium granules using the technology elaborated in the Titanium Institute. The paper presents a schematic process flow diagram for the production of granules by a centrifugal spraying of liquid metal. Analysis of industrial operation of the equipment and the quality of magnesium granules coated with fireproof flux has revealed basic advantages and limitations of this material as a reagent for desulfurization of hot metal. Main advantages of granulated magnesium produced by casting a liquid metal mixed with a fireproof flux (salt additive) are as follows: 1. Spheroidal shapes of particles with a diameter of 0.4-1.6 mm; 2. High bulk density; 3. Good fluidity; 4. Fire and explosion safety which permits one to transfer the granulated magnesium by air-operated transport at substantial distance. Main drawback of the product is its hygroscopicity. Casting of granules of liquid metal in a mixture with flux is notable for it low production cost, with the granulation unit located at facilities producing magnesium by electrolysis. The paper also presents main physico-chemical and technological properties of granulated magnesium and formulates the proposals on its application in other metallurgical processes (modification, microalloying, production of modifying agents, etc.).

Materials Issues in Microelectronics: Optical, Electrical, and Thermal: Alpha Particle Issues in Microelectronics Packaging

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Yellapu V. Murty, Carpenter Technology Corporation, Research and Development, Reading, PA 19612-4662 USA; Prasad Godavarti, Motorola, Austin, TX 78721 USA; Sung-Ho Jin, Bell Laboratories, Lucent Technologies, Murray Hill, NJ 07974 USA; Sung Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; John Macwilliams, US Competitors, LLC., Newark, DE 19711 USA; Mark McCormack, Fujitsu Computer Packaging Technologies, San Jose, CA 95134; Martin Weiser, AlliedSignal Electronic Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Wednesday AM — Room: Lincoln C
March 15, 2000 — Location: Opryland Convention Center

Session Chair: Martin Weiser, AlliedSignal Electronic Materials, Spokane, WA 99216 USA

8:30 AM Opening Comments

8:40 AM Invited
The "Discovery" of Alpha Activity in Lead and Solder: *Ron Brodzinski*[1]; [1]Battelle, Pacific Northwest Nat. Lab., Richland, WA 99352 USA

More than 15 years ago the presence of ^{210}Pb activity in lead was found to create significant background in a low-background radiation detection spectrometer. Shortly thereafter, alpha particles emitted from the surface of a solder joint were directly observed with this spectrometer. It was further observed that these alpha particles had been concentrated on the surface of the solder by the melting process, and that they decayed with the energy and half-life of ^{210}Po. Antiquity lead was used to eliminate these sources of radioactive background. The experiments and developments leading up to these "discoveries" will be described. The magnitude of the effect of ^{210}Pb in lead on the experiment will be discussed. The current sensitivity of these spectrometers for detecting ^{210}Pb and other radiocontaminants in lead and other materials will also be presented. The sensitivity of various techniques for measuring ^{210}Pb in lead will be compared, and a practical limit for alpha activity in lead will be proposed.

9:10 AM
From "Clean" Galena to "Contaminated" Lead-Why?: *Glenn I. Lykken*[1]; Ben Ziegler[1]; Berislav Momcilovic[2]; [1]University of North Dakota, Grand Forks, ND 58202-7129 USA; [2]Institute for Medical Research and Occupational Health, P.O. Box 291, Zagreb, Croatia

Lead ore (galena) is originally very low in 210Pb whereas lead processed from galena may have amounts of 210Po high enough to generate soft errors in computer chips. We processed a low 210Pb galena (Doe Run, Co., Herculaneum, MO) in a small-scale standard smelting method (Heraeus, patented). To reduce the melting point of galena, 2.5 g of galena were mixed with 2.5 g Na_2CO_3 and 0.2 g of powdered graphite as a reducing agent in a graphite boat. Both Na_2CO_3 and powdered graphite contained 210Po and so does the smelted lead from previously low 210Pb galena. Polonium-210 alpha particle emissions were measured with a 676A Alpha-King Spectrometer, EG&G Ortec, Nashville, TN. Supported in part by DEPSCoR under Research Proposal No. 40072-EL-DPS.

9:40 AM Break

9:55 AM
The Detection and Analysis of Alpha Particle Emitting Contaminants in Semiconductor Packaging Materials: *Don Weeks*[1]; Mike Tucker[1]; [1]Spectrum Sciences, 3050 Oakmead Village Dr., Santa Clara, CA 95051 USA

Semiconductor alpha particle induced soft error was discovered by Tim May and Murray Woods at Intel in the late 1970's. Since that time, there have been several nuclear particle counting techniques developed to monitor semiconductor packaging materials for alpha emitting radioisotopes. Methods of surface analysis, bulk analysis and alpha spectroscopy will be discussed. Emphasis will be placed on acceptable measurement techniquesfor evaluating Flip Chip and BGA lead solder related processes.

10:25 AM
Ensuring Alpha Counting: *Guenter Schindlbeck*[1]; [1]Infineon Technologies, MPQ, Balanstrasse 73, P.O. Box 800949, Munich 81609 Germany

Alpha counting rates follow Poisson distributions. The slope of the cumulative density function (CDF) of a Poisson distribution only depends on its mean value. This fact can be used for simple graphical checks of counting rates. The first example demonstrates, how to check a series of counting rates with mean values above 20. With the second example a systematical error was detected, which was added to the purely statistical fluctuations of a Poisson distribution. Analysing the chronological distribution of the counting rates, daily alterations and a superpositioned weekly cycle was found. The tubes of the gas supply had caused the problem. By replacing the tubes, this problem could be solved. For mean values below 5 to 10 a modified method is presented for checking series of counting rates. A table of alpha rates is presented. All measurements were checked with the methods described above. The alpha rates vary from about 0.0005 to more than 100 alphas per square centimeter per hour. One counting tube was investigated in detail. Its background counting rate showed a different dependency on bias voltage, than the counting efficiency. A clear minimum of the relative background (background divided by efficiency) showed up near the lower edge of the alpha plateau. This bias point represents the optimum operating condition.

10:55 AM
Micron Slices of Lead for Assessment of Alpha Particle Emissions in Computer Chip Manufacturing: *Berislav Momcilovic*[1]; Glenn I. Lykken[2]; Cody Nitschke[2]; [1]Institute for Medical Research and Occupational Health, P.O. Box 291, Zagreb Croatia; [2]University of North Dakota, Grand Forks, ND 58202-7129 USA

Soft errors in computer memory chips arise when alpha particles from the radon daughter 210Po in the lead solder bumps change the charge state of individual transistors in the memory. Certification of the 210Pb concentration in low alpha lead (LAL) is difficult, time consuming and expensive at the levels required by the industry. We used a microtome (American Optical Co., Buffalo, NY) to produce thin lead samples (1 to 5 microns) of relatively large area (> 7 cm2) so that a alpha flux from thin samples of large surface area could be measured. Attenuation of 210Po alpha particles (collimated beam) from a plated source (0.1 microcurie, Spectrum Technologies, Oak Ridge, TN) m were used to measure peak smearing and energy shift dependence upon lead thickness (676A Alpha-King Spectrometer, EG&G Ortec, Oak Ridge, TN). Lead slices one, and two micron thickness had respective peak broadening of 7 to 70 times and energy shifts of 0.5 to 1.4 MeV. Lead slices thicker than 2 microns produced broad, low intensity peaks with poor resolution. These data demonstrate the necessity to standardize the optimal lead thickness for uniform standardization of the measurement techniques among different laboratories. Supported in part by DEPSCoR under Research Proposal No. 40072-EL-DPS.

11:25 AM
The Relative Counting Efficiencies of Zinc Sulfide and Gas Proportional Alpha: *T. H. Zabel*[1]; [1]IBM, T. J. Watson Rsch. Ctr., P.O. Box 128, Rt. 134, Yorktown Heights, NY 10598 USA

Several different methods of measuring alpha particle emission from materials used to manufacture semiconductors have been developed.

These different methods often produce inconsistent results for measurements made on the same sample. Smaller devices and more intimate contact with the potential alpha particle emitters such as solder bumps require materials with lower alpha emission rates. As a result, it has become more important to understand the discrepancies in reported alpha emission between different measurement techniques. This talk will discuss the physics behind both zinc sulfide and gas proportional counters and how this impacts the measurements. It will then compare the physical models to experimental data to determine the relative counter efficiencies. Finally, I will discuss how to correlate the results from these two measurement techniques so that the users and suppliers of low alpha Pb and related materials can obtain more reliable results.

Materials Processing in the Computer Age III: Solidification and Process Modeling

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizers: Vaughan Voller, University of Minnesota, Saint Anthony Falls Laboratory, Minneapolis, MN 55414-2196 USA; Hani Henein, University of Alberta, Edmonton, AB T6G 2G6 Canada; Sulekh Jain, Ge Aircraft Engineering, MId M-89, Cincinatti, OH 45215 USA

Wednesday AM Room: Lincoln A
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Christoph Beckermann, University of Iowa, Dept. Mech. Eng., Iowa City, IA 52242 USA; John M. Krane, Purdue University, Sch. of Matls. Eng., West Lafayette, IN 47907 USA

8:30 AM
Modeling of Semisolid Metal Casting: *Andreas N. Alexandrou*[1]; Gilmer R. Burgos[1]; Vladimir M. Entov[2]; [1]Worcester Polytechnic Institute, Semisolid Matl. Process. Lab.-Met. Process. Instit., 100 Institute Rd., Worcester, MA 01609 USA; [2]Institute for Problems in Mechanics of Russian Academy of Science, Pr. Vernadskogo 101, Moscow 117526 Russia

Processing of metal alloys in their mushy state represent a new trend in metal processing. As part of the process (thixocasting), specially prepared billets are reheated to a temperature in the mushy zone, and then injected into a die. Parts produced using this technology offer better mechanical properties than those produced by casting and characteristics comparable to those of forged alloys. During thixocasting, semisolid slurries are highly concentrated suspensions of rounded rosette-like crystals in eutectic liquid. The mechanical behavior of the slurry is determined by the structure and properties of the skeleton formed by the alpha phase particles. The structure of the skeleton is almost never at equilibrium. It depends on the mechanical and thermal history of the material, and its evolution is governed by a number of kinetic phenomena of different characteristic time-scales. As a result of these kinetic processes, the rheological properties of the material, such as effective viscosity and yield stress, decrease with structure breakdown and increase with its development. In the present investigation, the behavior of semisolid slurries during processing is modeled using conservation equations and the Herschel-Bulkley fluid model. The rheological parameters are assumed to be functions of the solid volume fraction, and of a structural parameter that changes with processing history. The evolution of the structural parameter is described by a first order kinetic differential equation that relates the rate of build-up and break-down of the solid skeleton. The model is implemented into a computer code to predict die filling. An extensive parametric study is performed with different rheological constants and their effect on processing is analyzed.

8:50 AM
A Computer Model for Simulation of Multi-Scale Phenomena in the Centrifugal Casting of Metal-Matrix-Composites: *Laurentiu Nastac*[1]; Juan J. Valencia[1]; Junde Xu[1]; Hao Dong[1]; [1]Concurrent Technologies Corporation, Manu. Tech. Direct./Proc. Anal. Dept., 100 CTC Dr., Johnstown, PA 15904-1935 USA

A comprehensive computer model was developed to understand and optimize the centrifugal casting process for manufacturing TiC/Al-bronze friction drums. Performance of these components requires proper distribution of TiC particles, which is shown by dimensional analysis to be dominated by centrifugal buoyancy effects and solidification kinetics. The model addresses the following: (1) a rheology-viscosity model of the interference between moving particles, (2) engulfment (entrapment) or pushing of particles in the mushy region by the advancing solid/liquid interface, (3) nucleation and growth of equiaxed and eutectic phases, (4) effects of particle size, particle concentration, and cooling rate on the final grain size, and (5) the impingement effect of particles on the nucleation and growth kinetics of solidifying microstructure. Parametric studies were conducted using the computer model to investigate the effects of various process and material parameters on the distribution of TiC particles. The effects of the volume fraction, size, and morphology of the particles (including clusters and agglomerates) on the particle distribution were evaluated in detail. The complex interaction between the solidifying structure and insoluble ceramic particles in centrifugally-cast metal-matrix-composites was also investigated. The model predictions of particle distribution and microstructure were validated with experimental data for centrifugally-cast A356/SiC and TiC/Al-bronze alloys. This work was conducted by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation under contract No. N00140-92-C-BC49 to the U.S. Navy as part of the U.S. Navy Manufacturing Technology Program.

9:10 AM
Microsegregation in Ternary Alloys with an Open System: Michael R. McLane[1]; *Matthew John M. Krane*[1]; [1]Purdue University, School of Matls. Eng., 1289 MSEE Bldg., West Lafayette, IN 47907 USA

A model is developed to simulate microsegregation during the solidification of ternary alloys. Composition profiles of each component of several aluminum rich ternary alloys are obtained for the primary and secondary phases. The model addresses the complications associated with an open system and possible remelting due to mixture composition and temperature variations during solidification. Calculated profiles are presented for the aluminum rich region of the Al-Cu-Mg system and compared with published results.

9:30 AM
Large Eddy Simulation of Turbulent Flow in Continuous Casting of Steel: *Sivaraj Sivaramakrishnan*[1]; *Brian G. Thomas*[1]; Pratap Vanka[1]; [1]University of Illinois, Mech. & Indust. Eng., 1206 W. Green St., Urbana, IL 61801 USA

During the continuous casting of steel, transient flow events may be very important to the generation of quality problems, such as surface level fluctuations, inclusion and bubble entrainment. Conventional Reynolds-averaged models of turbulent flow, such as K-e, are inherently inaccurate at predicting the intermittent, transient flow events that arise due the chaotic turbulent motion. To improve the ability to predict these phenomena, large eddy simulation models are being developed, that directly simulate the important large-scale structures. These models are being applied to simulate three-dimensional, transient, asymmetric flow in the continuous slab-casting mold and the associated quality problems.

9:50 AM
Comparison of the Transient Start-Up Phase in an Aluminum Ingot-Differences in: *Daniel Paul Cook*[1]; *W. Kinzy Jones*[2]; [1]Reynolds Metals Company, Corp. Rsch. and Dev., 13203 N. Enon Church Rd., Chester, VA 23831 USA; [2]Motorola Inc., Phys. Proto. and Tool. Ctr., 8000 W. Sunrise Blvd., Ft. Lauderdale, FL 33324 USA

With the ever-increasing power of desktop computers, simulation has become an indispensable tool for today's metallurgist. Few researchers, however, have the time or luxury to develop their own numerical code and this has lead to a large increase in the number, and complexity, of the commercially available software packages. These commercial software packages have not yet reached the "black-box" stage, i.e. where they are both bug-free and relatively easy-to-use for researchers new to the field of simulation. In this paper, we will discuss bench marking of two popular general purpose CFD codes distributed by Fluent, Inc., FIDAP, which is based on the finite element method, and FLUENT-UNS, which uses finite volumes to discretize the computational domain. Both of these codes were used to model the transient start-up phase in vertical, direct chill casting of aluminum. Temperature data was taken during multiple casts of a Al-1%Cu alloy and a thermal profile of the ingot was developed. This data was then used to validate the mathematical models and compare the solution methodologies used in each code. Finally, comparisons of computational resources necessary for each code will be presented.

10:10 AM Break

10:30 AM
Virtual Aluminum Castings: A Tool for Process and Product Optimization: *John E. Allison*[1]; Ravi Vijayaraghavan[1]; [1]Ford Motor Company, Ford Rsch. Lab., MD 3182 SRL, Dearborn, MI 48124-2053 USA

In the next ten years the materials developments which will have the most significant economic impact on the automotive industry will be those which lead to refinements and improvements in existing materials and processes. Advances in computational materials science will play a pivotal role in this optimization of materials, processes and components. This talk will overview an integrated analytical approach to optimization of aluminum castings as well as progress in achieving this goal. Our vision is that virtual aluminum castings will be designed, cast, heat treated and complete durability testing, all on a workstation. In addition to a robust knowledge of molten metal flow and thermal history, models which accurately predict microstructural evolution during casting and heat treatment are required along with models relating these microstructures to mechanical properties and failure criteria. Recent progress in each of these areas will be reviewed for the Al-Si-Cu alloys typically used in automobile engine structures, including models for phase equilibria and microsegregation, aging response, and the influence of microstructure on tensile and fatigue properties. A number of unsolved problems have been identified and will be discussed.

10:50 AM
Modeling of Materials Synthesis in Thermal Plasma Reactor: *Sutham Niyomwas*[1]; Banqiu Wu[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metallu. and Matls. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

A mathematical model was developed to describe the plasma gas and particle dynamics and conversion yields. The velocity and temperature fields in the plasma were calculated by solving the Navier-Stokes equations and the thermal energy balance equations. Conservation equations for the system species consider the multi-component diffusion and chemical reactions. The model was used to study the thermal decomposition of ilmenite in the non-transferred arc plasma reactor. Through the application of these models, concentration, temperature and flow fields were computed.

11:10 AM
Scaling Laws and Instabilities in Electric Field Enhanced Smelting and Refining of Iron: *Adam Clayton Powell*[1]; Uday B. Pal[2]; [1]MIT, Matls. Sci. and Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139-4307 USA; [2]Boston University, Manu. Eng., 15 St. Mary's St., Boston, MA 02446 USA

In electric field-enhanced smelting and refining of iron and steel, reaction rate is controlled by iron ion diffusion through slag to the cathode, thus understanding of the kinetics at the cathode is important to improving the process. In addition, under some conditions, molten iron forms a conductive path through the slag, resulting in vastly lower efficiency; this underscores the importance of understanding the phenomena here. However, the evolution of molten iron in slag gives rise to multiple coupled instabilities: the Mullins-Sekerka instability leads to protrusion of iron fingers from the cathode into the slag, capillary instabilities accelerated by uneven Lorentz force breaks the fingers into droplets, and dense iron suspended in slag produces a Rayleigh instability. These instabilities and their time/length scales are explored in order to understand the regimes of operation and limitations to overall reaction rate, and to pave the way to future numerical modeling of the process for cathode design optimization.

Packaging & Soldering Technologies for Electronic Interconnects: Reliability of Bulk Solders

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Hareesh Mavoori, Bell Laboratories, Murray Hill, NJ 07974 USA; Srini Chada, Motorola, Plantation, FL 33322 USA; Gautam Ghosh, Northwestern University, Department of Materials Science, Evanston, IL 60208-3108 USA; Martin Weiser, AlliedSignal Electronic Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Wednesday AM Room: Lincoln D
March 15, 2000 Location: Opryland Convention Center

Session Chairs: S. Jin, Lucent Technologies, Murray Hill, NJ 07974 USA; R. A. Fournelle, Marquette University, Milwaukee, WI 53201 USA

8:30 AM Invited
Dimensionally Stable Solders for Microelectronic and Optoelectronic Applications: *Hareesh Mavoori*[1]; Sungho Jin[1]; [1]Bell Laboratories, Lucent Tech., 700 Mountain Ave., Murray Hill, NJ 07974 USA

Solders are generally prone to time-dependent deformation caused by phenomena such as creep and stress-relaxation due to their low melting points and high homologous operating temperatures. Such dimensional instabilities could lead to failures in certain optoelectronic and microelectronic devices through loss of optical alignment, electrical isolation, or mechanical failures. In light of the ever-increasing device miniaturization and complexity, it is desirable to have solders that are resistant to changes in dimensions due to creep and microstructural instabilities. We present two approaches towards minimizing creep in solder bonds: (1) an oxide-dispersion based approach to improve the creep-resistance and microstructural stability of the solder itself, and (2) an alloying approach that dramatically improves the interfacial bonding, thereby eliminating many of the stability problems related to solder bonding of optical or microelectronic components.

9:00 AM Invited
Thermomechanical Fatigue Behavior of Sn-Ag Solder Joints: S. Choi[1]; J. Lucas[1]; *K. N. Subramanian*[1]; [1]Michigan State University, Dept. of Mat. Sci. & Mech., East Lansing, MI 48824-1226 USA

Solder joints in electronic packages experience thermal cycling due to temperature fluctuations encountered in service. In automotive under-the-hood applications extreme temperatures could range between -40 to 150°C. Thermomechanical fatigue (TMF), caused by stresses developed from CTE mismatch during thermal cycling, is one of the most important contributor to solder joint failures. Microstructure evolution, and the fracture behavior, in Sn-Ag solders under thermal cycling conditions will be discussed.

9:30 AM
A Study of Lead Free Solders: *David K. Suraski*[1]; *Karl F. Seelig*[1]; [1]AIM Inc., Tech. Dept., 25 Kenney Dr., Cranston, RI 02920 USA

With the ongoing concern regarding environmental pollutants, lead has been targeted in the electronics assembly arena. One result of the push to find lead-free solder alternatives is that there now are many options to the board assembler. Much development, patterning, and research have gone into finding a viable solution for those who want to eliminate lead from their processes. However, each lead-free alloy is different in significant ways and background information about each of these is necessary. This paper shall discuss these various alloys and compare them to one another, as well as to the traditional tin-lead alloy. Highlighted in this will be a comparison of the tin-silver, tin-copper, and tin-silver-copper alloys. Included in these comparisons will be new data on creep at four different temperatures and two different alloys comprised of tin-copper-silver.

9:55 AM Break

10:15 AM Invited

Analysis of Ring and Plug Shear Strengths for Comparison of Lead Free Solders: *James C. Foley*[1]; Alan Gickler[2]; Larry LeProvost[2]; [1]Ames Laboratory, Metallu. & Cer. Pgm., 122 Metals Dev., Ames, IA 50011 USA; [2]Johnson Manufacturing Company, 114 Lost Grove Rd., P.O. Box 96, Princeton, IA 52768 USA

The global drive to replace the use of toxic lead metal and its alloys in industrial applications has spurred the development of new Lead-Free solder alloys. In addition to the toxicity of lead, there are other problems concerning the mechanical properties of Sn-Pb and Pb-based solders. Current leaded solders lack shear strength and resistance to creep and to thermal-mechanical fatigue. A solder which exhibits enhancements of these properties and retains solderability is crucial in avionics, automotive electronics, and industrial applications where the solder joints are subjected to many thermal cycles, severe vibrations, and sustained temperatures of up to 125°C. Modified ring and plug joints were made with 12 selected lead-free solders and 2 well characterized lead containing solders. The results of the mechanical tests under varying temperature and strain rate conditions provide a basis for selection of the optimum lead free solder for elevated temperature applications.

10:45 AM

Characterization of Microstructural Evolution in Sn-3.5Ag Solder Joints during Creep: *Vladimir I. Igoshev*[1]; *Jacob I. Kleiman*[1]; Ulysse Michon[2]; Donkai Shangguan[3]; Stephen Wong[4]; [1]Integrity Testing Laboratory Inc., 4925 Dufferin St., North York, ON M3H5T6 Canada; [2]ESPEO, Orleans, Cedex France; [3]Visteon Automotive Systems, Dearborn, MI USA; [4]Visteon Automotive Systems, Markham, ON Canada

In this work, specially designed Cu coupons were soldered with Sn-3.5Ag solder to model real solder joints. The samples underwent creep tests at different temperatures and applied stresses. The kinetic data on the thickness of Cu-Sn intermetallics layer and the distribution of Sn-Ag intermetallics in the matrix are presented. The results of the creep test (the kinetic data as well as microstructural changes) are compared with the data obtained for samples made of Sn-3.5Ag bulk solder alloy. The role of the intermetallics (Cu-Sn and Ag-Sn) in the reliability of the solder joints is discussed in view of the creep test results.

11:10 AM

Reliability of In-Situ Composite Solder Bumps Produced by an In-Situ Process: *Jong-Hyun Lee*[1]; Dae-Jin Park[1]; Jong-Tae Moon[2]; Yong-Ho Lee[1]; Yong-Seog Kim[1]; [1]Hong Ik University, Metallu. and Matls. Sci., Mapo-Gu Sangsu-Dong 72-1, Seoul 121-791 Korea; [2]Hyundai Electronics Company, Device and Semicond. Rsch. Div., Ichon 467-701 Korea

With the ever-increasing heat release rate per unit volume of the electronic device associated with the employment of smaller size solder balls in recent packages such as in Chip Scale Packages, thermal stability of the solders is crucial for the reliability of the electronic devices. In this study, Sn-Pb or Sn-Ag matrix composite solders reinforced with Cu_6Sn_5 dispersoids, which was formed via an in-situ reaction process between Cu powder and the molten solder, were produced. The ingots were hot rolled into thin sheets and from which disks were punched-out. The solder disks were remelted in a column of silicon oil to form solders balls. Microstructural observation of the solder balls indicated that the size of the reinforcements is about 2mm. Reflow soldering of the balls on a BT-resin substrate was conducted in a furnace of convection heating and the shear strength were measured after various thermal histories, which include reflow soldering up to 4 times, aging at 120°C for 210 hours, and thermal cycling between +150 and -65°C. In those tests, the composite solders showed consistently better properties than those of matrices. Microstructural observation of the solders showed very little growth of the reinforcements after those thermal treatments and the Pb-rich layer, which forms near the interface of the eutectic solder/substrate interface, was not developed with the composite solders.

Process Synthesis and Modeling for the Production & Processing of Titanium & Its Alloys: Session III

Sponsored by: Materials Processing and Manufacturing Division, Structural Materials Division, Titanium Committee, Shaping and Forming Committee

Program Organizers: James A. Hall, Oremet-Wah Chang, Albany, OR 97321 USA; F. H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026 USA; Isaac Weiss, Johnson Matthey, USA; Kuang Oscar Yu, RMI Corporation, R&D, Niles, OH 44446-0269 USA

Wednesday AM Room: Knoxville B
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Isaac Weiss, Johnson Matthey, USA; Sam Froes, University of Idaho, Moscow, ID 83844-3026 USA

8:30 AM

Modeling of the Mechanochemical Process for the Synthesis of Ti Based Materials: *Swati Ghosh*[1]; E. G. Baburaj[1]; K. Prisbrey[2]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]University of Idaho, Dept. of Metallu. and Min. Eng., Moscow, ID 83844-3026 USA

A mechanistic model has been developed to study the kinetics of formation of titanium-aluminum alloys in nanocrystalline form by mechanochemical synthesis. The model is based on Courtney's phenomenological model for mechanical alloying and has been extended by hypothesizing an intermediate activated complex to take into account the chemical reactions that occur simultaneously with solid-state displacement reactions during the mechanochemical synthesis. The present model takes into consideration the liquid-solid reactant mixture (Liquid TiCl4 + Solid AlCl3 + Solid Reducing Agent CaH2 and Mg) in place of the ideal solid-solid combinations considered in Courtney's model. The kinetics of the synthesis are expressed in terms of 'event probabilities' which relates to the fractions reacted with time. In addition, the effect of charge ratio, pre-milling and ball-to-powder ratio on the reaction kinetics have been evaluated and experimentally verified. The model gives an approximation of the actual process inside the reactor and allows us to predict changes in reaction kinetics with changes in processing parameters.

8:55 AM

Synthesis of a Low Density Ti-Si-Al Alloy: *Mutlu Cavusoglu*[1]; Oleg N. Senkov[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg. R321, Moscow, ID 83844-3026 USA

A low density Ti-Si-Al alloy was synthesized by blended elemental (BE) powder approach in conjunction with mechanical alloying (MA) and heat treatment. Some amount of titanium hydride (TiH1.924) was blended with titanium in order to avoid the use of process control agent (PCA) and contamination caused by it. Nature of phase transformations taking place in the system during heating was studied utilizing differential thermal analysis (DTA) and X-ray diffraction technique

(XRD). It was found that after annealing the blended elemental powder even at 1150°C, some silicon, aluminum and titanium still remained in elemental form in addition to the Ti5Si3 and TiAl phases present in the system. However after 15 hours of MA, TiH1.924, Ti, Si and Al were present in an amorphous phase which resulted in wide, fused XRD peaks. Formation of Ti5-Si3 was observed around 425°C while heating up to 500°C. Decomposition of the amorphous phase was observed after heating up to 660°C and resulted in formation of TiAl and further formation of Ti5Si3 phases. Some amount of titanium was still present in the system. After annealing at 1150°C, only two stable phases, Ti5Si3 and TiAl were present in the alloy and were stable during subsequent cooling or heating of the alloy.

9:20 AM
Effect of Cr Substituted for Fe on Resistivity and Tensile Properties of Ti-Fe-Cr Alloys: *Masahiko Ikeda*[1]; Shin-ya Komatsu[1]; Koichiro Inoue[1]; Hiroyuki Shiota[1]; Toru Imose[1]; [1]Kansai University, Dept. of Matls. Sci. and Eng., 3-3-35, Yamate-cho, Suita, Osaka 564-8680 Japan

Beta phase stability and tensile properties were investigated on Ti-Fe-Cr alloys having constant e/a, 4.28 in solution treated and quenched (STQed) state through resistivity measurement and tensile test. In the STQed state, resistivity and Vickers hardness decreased with increasing Cr substituted for Fe, though maintaining negative temperature dependence of resistivity. From these results, it is considered that beta phase stability increased with addition of Cr as a substitute for Fe. With addition of Cr, though tensile strength slightly decreased down to about 900MPa, elongation increased up to about 20%. Balance between the tensile strength and the elongation was improved by addition of Cr.

9:45 AM Break

10:00 AM
Microstructural Properties of Low Density Ti-Mg-Si Alloy: *Mutlu Cavusoglu*[1]; Oleg N. Senkov[1]; F.H. (Sam) Froes[1]; [1]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

A low density Ti-Mg-Si alloy was produced by hot isostatic pressing (HIP) of mechanically alloyed (MA'd) powders and blended elemental (BE) powders. Ti, Mg and Si were blended in the weight proportion of 20:3:2 respectively. In some experiments a mixture of Ti and TiH1.924 was used in order to eliminate the use of process control agent (PCA), stearic acid, and contamination caused by it. HIP'ing was performed at 700°C, 30 ksi for two hours. After MA, a supersaturated solid solution of Mg and Si in Ti or Ti + TiH1.924 (in batches where TiH1.924 was used) was produced and a nanocrystalline structure was formed. XRD phase analysis of HIP'd compacts of BE powders revealed Ti and Mg-2Si as the major phases. However only a titanium phase was present in HIP'd compacts of MA'd powders. Annealing up to 1150°C was carried out using DTA, resulted in Ti, Ti5Si3 and MgO in both compacts. Optical (OM) and transmission electron microscopy (TEM) was used for microstructural analysis. TEM analysis showed very fine nanometer sized grains structure in HIP'd and annealed compacts produced from MA'd powders. The results obtained showed a possibility of production of a low-density titanium alloy with high concentrations of magnesium and silicon with a nanocrystalline structure. Mechanical properties of the alloy are being investigated.

10:25 AM
Mechanical Properties of a Nanocrystalline TiAl Alloy Produced by Mechanical Alloying and Hot Isostatic Pressing: *O. N. Senkov*[1]; M. R. Shagiev[2]; G. A. Salishchev[2]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]Institute for Metals Superplasticity Problems, Russian Acad. of Sci., Ufa 450001 Russia

A Ti-47Al-3Cr (in at.%) alloy with a grain size of 140nm was produced by hot isostatic pressing at 950°C of an amorphous mechanically alloyed powder. The compact was then annealed at 900°C to decrease internal stresses and cut in flat samples for tensile testing. Tensile mechanical properties were studied within a temperature range of 800°C to 1200°C and strain rate range of $8x10^{-5}$ to $1.7x10^{-1}$ s^{-1}. The deformation curves were typical to the high temperature behavior of the material, with hardening, softening and steady-state stages. Elongation of the samples increased with an increase in temperature and went through a maximum when strain rate decreased. The elongation of 165% was achieved at 900°C and 405% at 1200°C. The strain rate sensitivity of the flow stress increased with temperature from 0.16 to 0.30 within the temperature range studied, and it only slightly depended on strain rate. The activation energy of the plastic flow was determined to be Q=347 kJ/mol. Because of microstructural features resulted from mechanical alloying, the deformation was non-homogeneous that led to the non-superplastic behavior of the alloy at low deformation temperatures. With increasing the deformation temperature up to 1100-1200°C improvements in the microstructural homogeneity occurred resulting in substantial ductility improvement.

10:50 AM
Mechanical Modeling of Diffusion Bonding at a Periphery Part of Two Flat Disks Out of Ti-6Al-4V Alloy: *V. K. Berdin*[1]; E. Evantelista[2]; [1]Institute for Metals Superplasticity Problems, 39 Khalturin St., Ufa 450001 Russia; [2]University of Ancona, Mech. Dept., Anacona Italy

It is known that Diffusion Bonding (DB) mainly depends on temperature, time and stress applied on local volume of joining surface. In addition, the interrelations between loading scheme and stress state resulted on the area to be joined, affects microstructure and mechanical properties of the bonded product. Diffusion bonding at periphery of two Ti-6Al-4V flat discs, 190.0 mm in diameter and 4.0 mm in thickness, was conducted in a vacuum furnace at 930°C. Finite element modeling, FEM, (ANSYS 5.3), was used to predict the stress-strain state on the joining zone. Material properties were determined by a visco-plastic constitutive law derived from mechanical tests. On the basis of FEM analyses, microstructural and fractography and mechanical investigations of the welded zone, the interrelation between loading scheme and properties of the formed joint was established.

Rare Earths and Actinides; Science Technology and Applications IV: Rare Earths I Extraction

Sponsored by: Light Metals Division, Reactive Metals Committee

Program Organizers: Renato G. Bautista, University of Nevada-Reno, Department of Chemical and Metal Engineering, Reno, NV 89557-0136 USA; Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA

Wednesday AM Room: Lincoln E
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Renato G. Bautista, University of Nevada, Metallu. and Matls. Eng., Reno, NV 89557 USA; K. Osseo Asare, Pennsylvania State University, Dept. of Matls. Sci. and Eng., University Park, PA 16802 USA

8:30 AM
Robert Bunsen and the Rare Earth Industry: *Fathi Habashi*[1]; [1]Laval University, Dept. of Min. & Metallu., Quebec City G1K7P4 Canada

Robert Bunsen (1811-1899) the German chemistry professor most famous for the burner, he invented and now used in most laboratories, played an important role in initiating the rare earth industry. Bunsen's laboratory was a meeting place for chemistry students from all over Europe. Two students who came to Bunsen played a particular role in the history of the rare earths. They were Jons Fridrik Bahr (1815-1875) from Uppsala and Carl Auer (1858-1929) from Vienna. The first brought with him samples of rare earths' minerals for analysis by Bunsen's newly invented spectroscope. The second was asked by Bunsen to study further the different spectra of rare earths extracted from

gadolinite. On his return to Vienna, Carl Auer took with him samples of the minerals to continue his research. Few years later he started the first rare earth industry. Details of the Swedish contribution, Bunsen's work, and Carl Auer's discoveries that led to the industry are outlined.

9:00 AM

Recovery of Rare Earths from a Bastnaesite Preconcentrate: *Yavuz Topkaya*[1]; [1]Middle East Technical University, Dept. of Metallu. and Matls. Eng., Ankara, Turkey

A bastnaesite type rare earth mineral, located in Beylikahir, Turkey, with an estimated ore deposit of 1 million tons, and an average concentration of 3.42 total REO was used in this study. The other constituents of the ore are calcium fluoride (52.5%), barite (25.4%), calcite (2.8%) and minor amounts of thorium, etc. The bastnaesite mineral occurs either as cement material between fluoride and barite particles or is intimately associated with these minerals. The rare earth elements are enriched considerably in sub-sieve sizes. The traditional concentration and hydrometallurgical methods were not suitable for the production of marketable bastnaesite concentrate and other products. A rare earth preconcentrate with 23.5% REO with a recovery of 77.5% was prepared by attrition scrubbing and desliming by cyclones. The beneficiation of fluoride and barite minerals was carried out by applying various physical concentration methods. Sulphuric acid baking and subsequent water leaching were used for the extraction of rare earths from the preconcentrate. Rare earth leach recoveries up to 90% were readily obtained.

9:30 AM

Decomposition of Xenotime with Carbon Tetrachloride: Kinetic and Microstructural Studies: Evandro Batista Augusto[1]; *Herenilton Paulino Oliveira*[1]; [1]São Paulo University, Chem. Dept., Faculdade de Filosofia Ciências e Letras de Ribeirão Preto, Av. Bandeirantes 3900, Ribeirão Preto, São Paulo 14040-901 Brazil

Among the rare earth minerals, fluorides, phosphates and oxides have been deserving attention for rare earth industry. Traditional methods of decomposition of these minerals, usually alkaline or acid processes, involve several operations. Another possibility to obtain lanthanides chlorides or oxychlorides is reacting the mineral with chlorinating agents (chlorination), like gaseous chlorine, hydrogen chloride, thionyl chloride and carbon tetrachloride, reducing the operations costs and making the process less complicated. In this context, we investigated the decomposition of xenotime (YPO_4) using carbon tetrachloride at temperatures from 873 K to 1173 K, and kinetic and mechanistic studies have been performed. The experimental apparatus for the essays included horizontally electric tubular furnace and a system to control the gases fluxes. Powder X-ray diffraction, SEM, EDX, UV/Vis transmission spectroscopy, FTIR, thermal analysis techniques were used in this study. The results show that the reaction follows the shrinking-unreacted-core model with a formation of a product layer (YOCl, confirmed by powder XRD). Moreover, significant microstructural changes of xenotime grains during the chlorination reaction were not verified. This work was supported by FAPESP under grant 1997/05779-1.

10:00 AM Break

10:30 AM

Direct Production of Mixed, Rare Earth Oxide Feed for High Energy-Product Magnets: *Fiona M. Doyle*[1]; Mark G. Benz[2]; Juliana C. Shei[2]; Ding Shan Bao[3]; Ni De Zhen[3]; [1]University of California at Berkeley, Dept. of Matls. Sci. and Eng., 551 Evans Hall #1760, Berkeley, CA 94720-1760 USA; [2]GE Corporate Research and Development, One Research Circle, Building K-1, Niskayuna, NY 12309 USA

Conventional rare earth purification processes produce pure, single elements or compounds. For manufacturing high energy-product magnets containing Pr, Nd, and some Ce, it would be preferable to produce mixed rare earths of the appropriate composition directly. A new solvent extraction configuration allows this. After separating Sm and higher rare earths, aqueous feed containing light rare earths undergoes solvent extraction with cation exchange or solvating extractants. Pr, Nd and a fraction of the Ce appropriate for the final application is extracted. La and residual Ce are recovered from the raffinate. The loaded organic undergoes selective stripping, yielding an aqueous stream containing Ce, Pr, and some of the Nd. The Nd remaining in the partially stripped organic can be recovered as a high purity product. The proportions of Nd reporting to the mixed feed and the pure stream can be adjusted by operating parameters, allowing flexible response to product specifications and market economics.

11:00 AM

Waste Reduction in Solvent Extraction Processes Utilizing Precipitation Stripping for the Heavy Lanthanides: *Peter M. Smith*[1]; George K. Schweitzer[1]; [1]University of Tennessee, Dept. of Chem., 552 Buehler Hall, Knoxville, TN 37996 USA

Solvent extraction is widely used for the purification of the lanthanides. Many industrial processes require large quantities of concentrated mineral acids to remove the lanthanide ions from the organic extractant, typically di-(2-ethylhexyl) phosphoric acid (D2EHPA)or mono-2-ethylhexyl-(2-ethylhexyl)-phosphonic acid (MEHEHP). The aqueous lanthanide solutions are subsequently neutralized and the lanthanides are precipitated as the oxalates. This process generates large volumes of neutralized acid waste. Precipitation stripping has the potential to reduce the acid consumption in solvent extraction processes, thereby greatly reducing the quantities of waste produced. Stripping by precipitation has been investigated for the removal of lanthanide ions from D2EHPA and MEHEHP. Previous studies have demonstrated the feasibility of oxalate precipitation stripping for the light lanthanides. This study describes the precipitation stripping characteristics of the heavy lanthanides, particularly lutetium, from D2EHPA and MEHEHP solutions using saturated aqueous solutions of oxalic acid. The study also addresses the feasibility of a precipitation stripping circuit in an industrial solvent extraction plant.

11:30 AM

Cell Design for the Electrolysis of Neodymium Oxide: *Rudolf Keller*[1]; [1]EMEC Consultants, 4221 Roundtop Rd., Export, PA 15632 USA

The electrolysis of neodymium oxide is an effective way to produce high-quality neodymium metal and alloys. The process chemistry is similar to that of the commercial electrolysis of aluminum oxide, but there are also significant differences, such as increased tendencies to product reoxidation, sludge formation and emission of perfluorocarbon gases. Related difficulties have been controlled successfully in 100-A experiments which extended over 96 hours and produced acceptable yields of neodymium-iron alloy, without the emission of any CF4 or C2F6. Water model studies suggested special cell design features to keep the electrolyte vigorously agitated in the anode area, while keeping agitation in the cathode area low.

12:00 PM

Cerium Oxide Particles in Aqueous Slurries for Chemical Mechanical Polishing (CMP): Solution and Surface Chemical Considerations: *K. Osseo-Asare*[1]; P. Suphantharida[1]; [1]Penn State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA

Ceria (CeO_2), in the form of dispersed particles in aqueous solution, is an important abrasive material for chemical-mechanical polishing (CMP) of glass and silicon nitride surfaces. Recently, the use of ceria has been extended to CMP applications in microelectronics technology. In spite of the growing use of ceria slurries, the polishing action of this material is still little understood. In this presentation, aqueous stability diagrams for the systems Ce-H_2O, Si-H_2O, and Si-N-H_2O, coupled with zeta potential data derived from the electrophoretic mobility of ceria particles in the presence and absence of silicate ions, are used to elucidate the interaction between ceria particles in CMP slurries and surface films on silica and silicon nitrides.

Research and Development Efforts on Metal Matrix Composites: Mechanical Behavior of MMCs

Sponsored by: Joint ASM-MSCTS/TMS-SMD Composites Committee ; Young Leaders Committee

Program Organizers: John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; Warren H. Hunt, Aluminum Consultants Group Inc., Murrysville, PA 15668 USA

Wednesday AM Room: Bayou A
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Daniel B. Miracle, US Air Force, Matls. and Mfg. Direct., Wright Patterson AFB, OH 45433 USA; Carl Boehlert, Johns Hopkins University, Dept. Mech. Eng., Baltimore, MD 21218 USA

8:30 AM Invited

Mechanical Behavior of Particle Reinforced Metal Matrix Composites-An Overview: *Nikhilesh Chawla*[1]; [1]Arizona State University, Dept. of Chem., Bio, and Matls. Eng., Tempe, AZ 85287-6006 USA

Metal matrix composites (MMCs) offer several advantages over conventional monolithic alloys. The enhanced mechanical properties, which result from incorporation of high modulus particles into a metallic matrix, are of interest for many applications. This talk will provide a broad overview of the mechanical behavior of particulate reinforced metal matrix composites, with an emphasis on SiC particle reinforced Al alloys. In general, strengthening in these materials stems from a combination of classical composite strengthening (direct strengthening) and changes in matrix microstructure and deformation characteristics (indirect effects) which arise from the presence of the reinforcement. The influence of reinforcement volume fraction and size on the mechanical behavior of the composites will be described. The effects of matrix microstructure (e.g., precipitate size, spacing, and distribution) on the mechanical response of the composites, as well as mechanical behavior at elevated temperatures will be discussed.

9:00 AM

Plastic Strain-Controlled Fatigue of SiC Particulate Reinforced Al Composites at Room and Elevated Temperature: *Jenn-Ming Yang*[1]; Nanlin L. Han[1]; Z. G. Wang[2]; [1]University of California, Matls. Sci. and Eng., Los Angeles, CA 90095-1595 USA; [2]State Key Laboratory of Fatigue and Fracture for Materials, Instit. of Met. Rsch., Chinese Acad. of Sci., 72 Wenhua Rd., Shenyang 110015 PRC

The low-cycle fatigue lives and cyclic stress response characteristics of SiC-particulate reinforced aluminum composites with different particulate volume fraction have been investigated at room and elevated temperature. The specimens were cyclically deformed with fully-reversed loading under plastic-strain amplitudes. The dislocation substructure developed during cyclic deformation was examined by TEM. The results show that the unreinforced aluminum exhibited initial cyclic hardening, cyclic stability and secondary hardening at room temperature and cyclic softening elevated temperature, while the composites showed gradual softening over most of the fatigue life both at room and elevated temperature. The differences in dislocation substructure obtained from processing and its developments under cyclic strain loading were considered to give rise to the observed phenomena.

9:20 AM

Microstructural and Mechanical Characterization of Carbon Coatings on SiC Fibers: *Dr. Kevin L. Kendig*[1]; Dr. Ronald Gibala[2]; Dr. Daniel B. Miracle[1]; Dr. Robert A. Shatwell[3]; [1]Air Force Research Laboratory, Matls. and Manufact. Direct., 2230 Tenth St., WPAFB, OH 45433-781 USA; [2]University of Michigan, Dept. of Matls. Sci. and Eng., 2300 Hayward St., 2026 H.H. Dow Bldg., Ann Arbor, MI 48109 USA; [3]Defense Evaluation and Research Agency, Sunbury on Thames, Middx TW167LN UK

Continuous SiC fibers used in titanium matrix composites often have an outer carbon coating of less than 5 mm in thickness. This coating has been identified as the common initiation location for composite failure in tension transverse to the fiber axis. A series of three carbon coatings were deposited using chemical vapor deposition while varying the SiC monofilament temperature. The microstructures of these fibers were examined using optical microscopy, scanning electron microscopy, and transmission electron microscopy. Transverse tensile tests on single-fiber composite samples revealed that the stress required to cause debonding of the fiber from the matrix was not significantly affected by deposition temperature. Adhesion experiments showed an increase in bond strength of the SiC-C interface with temperature. The increase in deposition temperature correlates with increased crystallographic texturing of the carbon coatings. In transverse tension, the carbon coating with the lowest SiC-C interface strength failed at this interface, and the coatings with more highly textured carbon failed within the coating.

9:40 AM

Tribological Evaluation of Various Aluminum Metal Matrix Composites: *Paul J. Huang*[1]; Wen-Sheng Chu[2]; [1]US Army Research Laboratory, Weapons & Matls. Rsch. Direct., AMSRL-WM-MC, Aberdeen Proving Ground, MD 21005-5069 USA; [2]Johns Hopkins University, 3400 N. Charles St., Baltimore, MD 21218 USA

The military is currently facing substantial hurdles as it seeks to improve fuel economy, reduce weight and increase performance of its ground vehicles. Advanced technology and materials will be required to meet these challenges. In particular, a number of potential engine applications where wear and weight are critical issues can be addressed with the introduction of aluminum metal matrix composites (Al MMC's Aluminum metal matrix composites reinforced with ceramic particulates have higher wear resistance and higher temperature capability than monolithic aluminum alloys. Unfortunately there is limited wear data on these new materials. Block-on-Ring tests, and other mechanical and thermal properties measurements have been made on various Al MMC's (i.e. 6061/SiCw/20%, 2014/SiCp/15%, A359/SiCp/10%, 2014/Al2O3p/15%), and monolithic aluminum alloys (i.e. 6061 and 2014). The scope of work is to develop a database of wear properties on Al MMC's and monolithic alloys to assist future system designs where wear resistance is a critical factor.

10:00 AM

Fracture and Fatigue of Nb5Si3/Nb Composites: D. Padhi[1]; S. Solv'yev[1]; W. Zinsser[3]; *John J. Lewandowski*[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. and Eng., The Case School of Eng., Cleveland, OH 44106 USA; [3]Cessna Aircraft, Wichita, KS USA

The fracture toughness and fatigue crack growth behavior of Nb5Si3/Nb composites have been determined over a range of test temperatures and conditions. The effects of changes in test temperature from 196°C to 500°C on the fracture toughness have been determined on both binary Nb-Si alloys as well as multi-component Nb-Si alloys. In addition, the effects of changes in R-ratio and test temperature on the fatigue crack growth behavior have been determined. Quantitative fractography has been utilized to relate the effects of changes in Δ K, R-ratio and test temperature on the fatigue crack growth rate.

10:20 AM Break

10:30 AM

Deformation and Fracture of a WNiFe Alloy in the Temperature Range—100°C to 300°C: *Robert G. O'Donnell*[1]; [1]Alloy Development Group, CSIRO Manu. and Sci. Tech., Private Bag 33, Clayton Sth MDC, Victoria 3169 Australia

Liquid phase sintered W-Ni-Fe alloys comprise a semicontiguous, spheroidal, tungsten rich phase surrounded by a Ni-Fe rich matrix phase. In this work, the fracture behaviour of a 95wt%W 3.5wt%Ni 1.5wt%Fe alloy, deformed in tension at temperatures in the range 100°C to 300°C, is investigated. The four principal fracture mechanisms within this alloy are: (i) intergranular fracture between tungsten

spheroids, (ii) interfacial fracture between the tungsten spheroids and the matrix material, (iii) rupture of the matrix material and (iv) transgranular fracture of the tungsten spheroids. The contribution to the fracture surface from each of these fracture mechanisms is determined to vary with test temperature. This variation is explained in terms of the relative strengths of the individual fracture mechanisms, and how these are related to the temperature dependence of the flow stress of each phase. Below the ductile to brittle transition temperature for the tungsten phase, all fracture mechanisms are well represented in the fracture surface whilst above the ductile to brittle transition temperature for this phase, fracture is primarily through tungsten cleavage. Intergranular fracture between tungsten spheroids during straining prior to final rupture results in the presence of cracks within the specimen. The role of these internal cracks in determining the final rupture mechanism in these alloys is also discussed.

10:50 AM
Damage Evolution by Interface Decohesion during Tensile Deformation of an Al2080/20 vol.% SiC Metal Matrix Composite: Robert David Evans[1]; *James Douglas Boyd*[1]; [1]Queen's University, Matls. and Metallu. Eng., Kingston, Ontario K7L4N6 Canada

An MCC of Al2080 reinforced with 20 vol.% of 3 um SiC particulate was prepared by powder processing and hot extrusion. This allowed the effects of interface structure and decohesion to be studied independent of the effects of particle cracking. Damage evolution during tensile testing to the point of fracture was studied by measuring 1) density, 2) area fraction of voids resulting from interface decohesion and 3) fraction of decohered particles. The latter 2 measurements were made on samples sectioned and imaged by focused ion beam microscopy (FIB). Transmission electron microscopy (TEM) of the near-interface region revealed that plastic strain is concentrated in 1-2 grain widths adjacent to the particles, and the decohesion mechanism is failure through a 20-40 nm thick amorphous Si-Mg-Al-O layer at the interface. The damage measurements were fitted to existing models as follows: 1) area fraction of voids to the Seetharaman and Semiatin model up to a maximum value of 1-2% at fracture, 2) fraction of decohered particles to the Whitehead and Cline model up to a maximum value of ~ 40% at fracture, and 3) number of decohered particles/area to the Sun model to calculate an interfacial strength of 200-300 MPa.

11:10 AM
Experiments and Numerical Calculations on Damage in Al/Al2O3 Model Composites: *Partha Ganguly*[1]; Warren James Poole[1]; [1]University of British Columbia, Dept. of Met. and Matls. Eng., 6350 Stores Rd., Vancouver, BC V6T1Z4 Canada

The goal of this work was to systematically study the initiation and accumulation of damage in a model two-phase material. The damage process was examined as a function of the spacing and the geometric arrangement of the reinforcing phase. A model composite system was fabricated from AA6061 and sapphire fibers (alumina fibers, 1 mm in diameter). The composite was prepared by infiltrating an array of sapphire fibers with the liquid aluminum alloy. The volume fraction and geometric arrangement of the fibers was maintained during casting by graphite spacers. The resulting composites were loaded in compression perpendicular to the fiber axis and the pattern of damage was monitored during deformation. In addition to the experiments, finite element method calculations were conducted using LS-DYNA. In these calculations, the effect of damaged fibers on the local stress and strain distribution was examined. Experiments showed that extensive damage occurs in this system either by fiber cracking or interfacial decohesion. At a given volume fraction of sapphire fibres, the geometric arrangement was found to strongly influence the damage process.

11:30 AM
Some Aspects of Hydrostatic Extrusion of Metal Matrix Composites: D. Lahaie[2]; *J. D. Embury*[1]; Francis W. Zok[3]; [1]McMaster University, Matls. Sci. and Eng., 1280 Main St. W., Hamilton, Ontario L8S4L7 Canada; [2]University of Quebec, Chicoutimi, Quebec, Canada; [3]University of California, Matls. Dept., Santa Barbara, CA USA

Hydrostatic extrusion represents a method of producing stress states which suppress damage formation in composites and thus allow the attainment of significant plastic strains during forming processes. This paper will deal with analysis of the local stress states in hydrostatic extrusion and their relation to damage accumulation. The results will be considered in terms of experimental data both for Cu-W composites and Al based MMC's.

11:50 AM Closing Remarks

12:00 PM
MMC Symposium Poster Session and Luncheon
Box Luncheon tickets required.

Research and Development Efforts on Metal Matrix Composites: Poster Session on MMCs

Sponsored by: Joint ASM-MSCTS/TMS-SMD Composites Committee; Young Leaders Committee
Program Organizers: John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; Warren H. Hunt, Aluminum Consultants Group Inc., Murrysville, PA 15668 USA

Wednesday AM Room: Bayou A
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Warren H. Hunt, Aluminum Consultants Group, Murrysville, PA 15668 USA; John J. Lewandowski, Case Western Reserve University, Dept. of Matls. Sci. and Eng., Cleveland, OH 44106 USA

Aging Characteristics of Al-Sc-Mg Alloy and Its Composite: *Awadh B. Pandey*[1]; Daniel B. Miracle[2]; Cory A. Smith[3]; Thomas J. Watson[4]; [1]UES Inc., Matls. and Process. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, Matls. and Manu. Direct., 2230 10th St. Ste. 1, Wright-Patterson Air Force Base, Dayton, OH 45433 USA; [3]DWA Aluminum Composites, 21130 Superior St., Chatsworth, CA 91377 USA; [4]Pratt & Whitney, 400 Main St., East Hartford, CT 06108 USA

Considerable effort has been made in the past to understand the aging response of discontinuously reinforced aluminum (DRA) composites with precipitation hardened aluminum alloys as a matrix material. It has been well documented that the composites show accelerated aging behavior as compared to the unreinforced alloy due to the enhanced dislocation density resulting from the coefficients of thermal expansion (CTE) mismatch between matrix and ceramic reinforcements. In this study, attempts are being made to understand the aging characteristics of a relatively new class of materials: Al-Sc-Mg alloy and Al-Sc-Mg/15 vol.% SiC composite, made by a powder metallurgy approach, to provide improved strength-toughness relationships. The influence of aging on the microstructure, hardness, and tensile properties of these materials will be presented and discussed. Differential scanning calorimetry (DSC) and transmission electron microscopy will be used to evaluate the phases present in the materials.

In-Situ Synthesis of Al-Si Alloy and SiC Composite: *Banqiu Wu*[1]; Ramana G. Reddy[1]; [1]University of Alabama, Metallu. Eng., A129 Bevill Bldg., P.O. Box 870202, Tuscaloosa, AL 35487 USA

Experimental investigation on synthesis of Al-Si alloy and SiC composites using methane was carried out in the temperature range of 950-1150°C. A kinetic model for the rate process was developed. The gas-liquid contact method and temperature have significant effect on the silicon carbide content in the composite. The Al-Si alloy composite with 25 wt.% SiC was obtained. The particle size of SiC formed was in the range of 1-10 µm. Reaction products were characterized using optical microscope, SEM and electron microprobe, and micro image

analyzer. Calculated SiC formation rate agrees well with experimental results.

Wear Behaviour, Microstructure and Dimensional Stability of As'Cast Zinc-Aluminum/SiC(MMC) Alloys: Rafael Auras[1]; *Carlos Enirique Schvezov*[1]; [1]University of Misiones, Faculty of Sci., Azara 1552, Posadas, Misiones 3300 Argentina

Zinc-Aluminum alloys and composites were cast and their microstructure, dimensional stability and wear properties were determined and analyzed. Five different alloys containing Silicon, Copper and Silicon Carbide particles were employed. The cast alloys were tested for a period of 1000 hr at 165°C and the results show that the five different alloys and composites were dimensional stable during the whole test. The wear test were performed using a pin-on-disc apparatus under dry and lubricated conditions. The charges used were 5 and 8 Kg, the velocity was 250 rpm (2m/s) and the test time was 1 hr. The wear test results show that under dry conditions there was considerable lost of material, particularly in the non-reinforced alloys. In addition, the non reinforced alloys presented substantial local plastic deformation and transfer of elements from the disc to the sample.

Thermo-Mechanical Characterization of 2080 Al/SiCp Composites by Mechanical Spectroscopy Technique: *Efrain Carreño-Morelli*[1]; Nikhilesh Chawla[2]; Robert Schaller[1]; [1]Ecole Polytechnique Fédérale de Lausanne, Institut. de Génie Atomique, Lausanne CH-1015 Switzerland; [2]Hoeganaes Corporation, Rsch. and Dev., 1001 Taylors Ln., Cinnaminson, NJ 08077 USA

The thermo-mechanical behavior of 2080 Al/SiCp composites was investigated by mechanical spectroscopy. The thermal fatigue behavior, between 100 K and 400 K, was studied by mechanical loss and dynamic shear modulus measurements. A transient mechanical loss maximum was observed during cooling near 150 K, that originates from relaxation of thermal stresses due to the differential thermal expansion between matrix and reinforcement. Decreasing particle size resulted in a decrease in the damping maximum and in improved dimensional stability during thermal cycling. Finally, by keeping the volume fraction and particle size constant, the matrix microstructure was varied to study the effect of precipitate and dislocation distribution on stress relaxation around the reinforcement. The effect of inclusion size and matrix microstructure on the microplasticity near the interfaces induced by thermal and mechanical stresses will be discussed.

Experimental and Numerical Examination of the Consolidation of Composite Powders in Pressure Cycling: *Guangbin Jiang*[1]; Weidong Wu[1]; Robert H. Wagoner[1]; Glenn S. Daehn[1]; [1]The Ohio State University, Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Powder consolidation of MMC's under cyclic pressure has been previously shown to produce significantly higher densities in mixed powders when the two powders have dissimilar compressibilities. Smaller effects when the reinforcement content is small, and large enhancements are possible at high ceramic loadings. This is a complex process that involves many mechanisms including pressure-change induced plasticity, powder rearrangement and frictional sliding. In order to remove the effects of powder rearrangement and consider the deformation behavior only, closely-packed macro-sized aluminum and steel rods studied in cyclic compaction. The finite element code, ABAQUS was employed to simulate the densification behavior under static and cyclic pressure. From simulations it is shown friction between particles has great effect on densification under cyclic pressure. Data obtained from the simpler arranged rods are compared with powder consolidations and both are compared to simulations. These are discussed in the context of determining the relative contributions of plasticity, friction and rearrangement in powder consolidation.

Fabrication Process and Thermal Properties of High Volume Fraction SiCp/Al Metal Matrix Composites: *Hyo Soo Lee*[1]; Soon Hyung Hong[1]; [1]Korea Advanced Institute of Science and Technology, Matls. Sci. and Eng., 373-1 Kusung-dong, Yusung-gu, Taejon 305-701 Korea

Fabrication process and thermal properties of high volume fraction ranged 50~71vol% SiCp/Al metal matrix composites (MMCs) for electronic packaging applications have been investigated. Preforms composed of 50~71vol% SiC particles were fabricated by ball milling and pressing method. SiCp/Al MMCs were fabricated by an infiltration of Al melt into SiC particle preforms using the pressure infiltration casting process. Thermal conductivities and coefficients of thermal expansion (CTE) of SiCp/Al MMCs were characterized with varying the volume fraction of SiC particles, content of inorganic binder and porosity using laser flash method and thermomechanical analysis, respectively. Thermal conductivity decreased from 177W/mK to 120W/mK and CTE decreased from 10ppm/K to 6ppm/K with increasing the volume fraction of SiC particles in SiCp/Al MMC from 50vol% to 71vol%. The measured coefficients of thermal expansion were in good agreement with the calculated coefficients of thermal expansion based on Turner's mode.

Direct Deposition of Laminated Metal-Matrix Composites by Laser Cladding Process: *Kali Mukherjee*[1]; Y. P. Hu[1]; C. W. Chen[1]; [1]Michigan State University, Matls. Sci. & Mech., and Comp. Matls. and Struct. Ctr., High Energy Laser Process. Lab., East Lansing, MI 48824 USA

Laser cladding technique has been employed to directly deposit laminated metal matrix composites (LMMCs). By using optimized processing parameters associated with 3-D geometric components, alternate 304L stainless steel, and CPM10V tool steel layer components have been successfully produced. The prototypes on steel substrates are found to be relatively smooth and metallurgically sound. Optical micrographs displayed that LMMC samples are free of pores and cracks, and have a good metallurgical bonding with the substrate, but with a low dilution. Laminated layers have discrete and serrated interface to maintain both original chemical compositions, and mechanical and physical properties for each cladding layer. The hardness of LMMC layers of stainless steel 304L and tool steel CPM10V is about 300 Hv and 750 Hv, respectively. Processing parameters, and properties associated with this study will be discussed in detail.

Fracture and Fatigue of Al-Be Composites: S. Solv'yev[1]; J. Larose[1]; R. Castro[2]; *John J. Lewandowski*[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., The Case School of Eng., Cleveland, OH 44106 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

The fracture toughness and fatigue crack growth behavior of Al-Be composites are being determined under a variety of test conditions. Fracture toughness is being determined on both notched and fatigue precracked specimens, while fatigue crack growth behavior is being measured at different R-ratios. The effects of test conditions on the fracture toughness and fatigue crack growth behavior will be summarized in addition to both optical and SEM examination of the fracture path and fracture surfaces.

Densification and Flow Stress Evolution Processing Model For Discontinuously Reinforced Aluminum (DRA) Composites: Erik J. Hilinski[2]; Thomas J. Rodjom[3]; Paul T. Wang[3]; *John J. Lewandowski*[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. and Eng., Case School of Eng., Cleveland, OH 44106 USA; [2]U.S. Steel Research Center, Monroeville, PA USA; [3]Alcoa Technical Center, 100 Tech. Dr., Alcoa Ctr., PA 15069-0001 USA

The current processing technology in the realm of discontinuously reinforced aluminum (DRA) materials generally employs a two step forming sequence whereby a powder compact (powder-void aggregate) is consolidated to full density and then extruded or forged into a desired component shape. In order to combine the consolidation and shaping operation into one processing step, an analytical description of the thermomechanical behavior of porous materials that provides an understanding of the key densification mechanisms and processes in powder consolidation is desired. A constitutive model of porous material densification and plastic flow, imbedded within a finite element model, would prove to be a useful tool in aiding the design of both die and preform; a key element in consolidating and shaping porous material into void-free products with one operation. This poster presents the efforts to develop the densification and flow stress evolution processing model. A porous yield criterion based on the Gurson micromechanical model, as modified by Tvergarrd, Richmond and Smelser, and Wang, is used to predict the densification response of the powder processed composite material. Ex-situ density measurements,

via the archimedean densitometry method, have been used to track the variation of porosity and have also been used to determine porosity evolution in open die uniaxial compression specimens. Yield surfaces of the monolithic and composite materials used in this investigation are presented. An attempt at predicting the flow stress evolution of the DRA material during uniaxial compression testing using single internal state variable theory has been unsuccessful to date. It appears that the aging characteristics of the matrix material must be taken into consideration in order to develop an accurate flow stress evolution description of the material.

Pressure Effects on Flow and Fracture of Monolithic and Composite Materials: P. Lowhaphandu[1]; *John J. Lewandowski*[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., The Case School of Eng., Cleveland, OH 44106 USA

A review of the effects of high pressure on the mechanical behavior and deformation processing of both monolithic metals and composites has recently been completed. This presentation will summarize the experimental observations that have been made on a variety of different materials systems, including composites.

Neutron Diffraction Study on the Effect of Reinforcement Volume Fraction and Temperature on Residual Stresses in Fiber Reinforced Metal Matrix Composites: *Partha Rangaswamy*[1]; Hahn Choo[2]; Mark A.M. Bourke[1]; Anil K. Saigal[3]; [1]Los Alamos National Laboratory, Matls. Sci. and Tech., MST-8, LANSCE 12, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Lujan Ctr., Lansce 12, H 805, Los Alamos, NM 87544 USA; [3]Tufts University, Dept. of Mech. Eng., Medford, MA 02155 USA

Pulsed neutron diffraction was used to determine residual elastic strain and stress in two continuous fiber reinforced metal matrix composites (MMC's): tungsten fiber reinforced Kanthal, and silicon carbide (SCS-6) reinforced titanium alloy. Residual strains/stresses were determined in tungsten/Kanthal MMC's containing 10, 20, 30 and 70 percent of fibers by volume (V_f). The focus of this study was to determine the effect of varying V_f of tungsten fibers on the residual stresses at the fiber matrix interface using simple micro-mechanics in the tungsten/Kanthal MMC's. High temperature measurements ranging from 20-1050°C were conducted on a SiC/Ti-6Al-4V MMC to study stress relaxation effects in the fiber and matrix. In addition, the relaxation of stresses in both the alpha(α) and beta (β) phases of the α-β Ti-6Al-4V matrix material was also determined as a function of temperature. These results are presented in order to demonstrate the use of neutron diffraction as a technique that is unique in studying this class of materials.

Creep Deformation and Rupture Behavior of Laminated Metal Matrix Composites: *S. B. Biner*[1]; [1]Iowa State University, Ames Lab., Metallu. and Cer., Ames, IA 50011 USA

In this study, the creep behavior at 250°C of laminated composite consisting of 6061Al alloy layers and 2014Al-20vol%SiC particulate reinforced composite layers was investigated. In spite of the absence of delamination between the layers, the observed creep rupture times of the laminated composite were much shorter than those seen for its constituent phases. This behavior is explained with a model based on the laminate theory. This work was performed for the United States Department of Energy by Iowa State University under contract W-7405-Eng-82.

Mixed-Mode Fracture Behavior of Laminated Metal-Matrix Composites: *S. B. Biner*[1]; [1]Iowa State University, Metallu. and Cer., Ames Lab., Ames, IA 50011 USA

In this study, the mixed-mode fracture behavior of laminated composite consisting of 6061Al alloy layers and 2014Al-20vol%SiC particulate reinforced composite was investigated. During the fracture tests, initial fatigue cracks were in the crack divider orientation in respect to orientation of the layers. Although, there was a significant increase in the fracture toughness value under pure mode-I loading, due to presence of relatively ductile 6061Al layers, this improvement was diminished with increasing mode-II component for larger mode mixities. The detailed fractographic studies and numerical analysis indicate that the observed fracture behavior is associated with the interfacial failure behavior between the layers under mixed loading conditions. This work was performed for the United States Department of Energy by Iowa State University under contract W-7405-Eng-82.

The Al2O3p/Zn-Al Composites Fabricated by Ultrasonic Treatment: *Wang Jun*[1]; Sun Baode[1]; Shu Guangji[2]; Zhou Yaohe[1]; [1]Shanghai Jiao Tong University, School of Matls. Sci. & Eng., Shanghai 200030 PRC; [2]Southeast University, Dept. of Matls. Sci. & Eng., Nanjing PRC

Although many techniques have been used for producing MMC's, the low cost liquid-metal process may be utilized mostly. However, it is usually very difficult to disperse ceramic particles homogeneously in liquid metal because of the poor wettability of the particles. High intensity ultrasonic treatment has been used to fabricate Al2O3p/Zn-Al composites in this paper. The minimum diameter of reinforcement used is 0.5 micrometer. The microstructure and mechanical properties are studied. The results show that particles disperse homogeneously with good bonding with matrix and the mechanical properties depend on the volume fraction rather than the size of reinforcement. The effective range of high intensity ultrasonic treatment is studied by experiment and numerical simulation. The mechanism of the ultrasonic treatment is believed to be the combined effects of the cavitation and acoustic streaming.

Experimental and Numerical Examination of Static Compaction Using Model Geometries: *Weidong Wu*[1]; Guangbin Jiang[1]; Glenn S. Daehn[1]; Robert H. Wagoner[1]; [1]The Ohio State University, Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The deformation behavior of metal matrix composites in cold uniaxial compaction was investigated both experimentally and numerically using aluminum and steel rods as a model plane-strain system. Static uniaxial die compaction was carried out with sets of rods in both close-packed and cubic arrays with both mixtures of hardened steel and aluminum as well as pure aluminum rods. ABAQUS was employed to simulate the densification behavior, inter-particle friction effects and springback behavior during compaction and ejection. The pressure-density curves were measured using both closed-hexagonal and square-packing arrangements of aluminum and steel rods, eliminating the effects of particle rearrangement and revealing the effects of plastic deformation evolution up to full density. Investigation showed that inclusions inhibited the densification during compaction. Particle-interface friction retards the densification in composite compaction, but shows no effect in pure material compaction. Contact pressure causes produces significant springback that should be accounted for in the modeling of powder consolidation. The density-pressure curves from simulation and experiments are very close as are the deformed shapes of the aluminum. Based on this work, our future work will attempt to calibrate the effects of particle rearrangement, morphology, size distribution and surface condition. Particular focus will be given to composite-powder compaction, an area many experiments have been done, but careful comparisons between experiment and simulation have been limited.

Explicit FEM Simulation of Powder Consolidation Under Pressure Cycling: *X. J. Xin*[1]; P. Jayaraman[1]; R. H. Wagoner[2]; G. S. Daehn[2]; G. Jiang[2]; [1]Kansas State University, Dept. of Mech. and Nuclear Eng., 338 Rathbone Hall, Manhattan, KS 66506 USA; [2]The Ohio State University, Dept. of Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

Pressure cycling has been shown experimentally to be a viable method to consolidate composite powders (i.e. metal/ceramic) to higher densities than can be obtained by normal consolidation procedures. Such processing can produce composites with higher volume-fraction ceramic content, improved homogeneity and better net shape precision. Explicit FEM (finite element modeling) was used to simulate systems of over 50,000 degrees of freedom and tens of loading cycles on a Pentium workstation in a few days. Particle re-arrangement during compaction can also be handled without extra difficulties. Various combinations of materials, particle arrangement, interfacial properties, and loading conditions have been investigated systematically. There is good agreement between simulations and experiments under monotonic loading conditions, while larger discrepancies exist for cyclic loading. In the range of 0 to 0.3, increasing friction coefficient leads to slightly decreased consolidation density for a given pressure,

but the effect of friction coefficient becomes negligible above 0.3. The effect of Poisson's ratio (from 0.01 to 0.495) is also small. A higher strain hardening rate, however, results in significantly lower compaction density under the same applied pressure.

Dry Sliding Wear Behavior of Al-3 WT.% Mg Matrix Composites Reinforced with SiC Particles Manufactured by a Pressureless Infiltration Technique: *Yong-Suk Kim*[1]; Seung-Hyun Kim[1]; Hyung-Il Kim[1]; [1]Kookmin University, School of Metallu. and Matls. Eng., 861-1 Chongnung-dong Songbuk-ku, Seoul 136-702 South Korea

Dry sliding wear behavior of Al-3 wt.% Mg matrix composites reinforced with SiC particles manufactured by a pressureless infiltration technique was investigated. Pin-on-disk wear tests under various applied load conditions were carried out using a hardened steel ball as a counterpart. The effect of the varying size and volume fraction of the carbide particle on the wear was studied. Worn surfaces of the composite together with wear debris were also examined with optical and electron microscopy. Wear resistance of the composite increased with the increase of the size and volume fraction of the reinforcing particle. Mild abrasive wear was accompanied with low wear rates, while massive severe delamination wear resulted in high wear rates. Subsurface strain of the wearing surface was estimated to correlate the subsurface deformation and cracking with the wear rate of the composite. Special attention was paid to the role of iron oxide layers formed on the wearing surface.

Correlation between Hardness and Tensile Properties of Discontinuously Reinforced Metal Matrix Composites: *Yu-Lin Shen*[1]; Nikhilesh Chawla[2]; [1]University of New Mexico, Dept. of Mech. Eng., Albuquerque, NM 87131 USA; [2]Hoeganaes Corporation, Rsch. and Dev., 1001 Taylors Ln., Cinnaminson, NJ 08077 USA

Hardness tests are used extensively in quantifying the mechanical properties of metallic materials. Good correlation between hardness and tensile strength have been well documented for many alloys. As metal matrix composites are generating increased interest from industry, traditional hardness testing may serve as a simple and useful means of characterizing the composite strength. In this study we attempt to develop a baseline understanding of the correlation between hardness and strength of discontinuously reinforced metal matrix composites. Aluminum alloys reinforced with various amounts and sizes of silicon carbide particles were used. The matrix microstructure in all composites was kept relatively constant by a combination of heat treating and rolling operations. Tensile tests and the Rockwell B scale hardness tests were performed. It was found that for composites with small reinforcement particles, the hardness value correlated well with tensile strength. For composites with larger reinforcement particles, the hardness test overestimated the tensile strength. It is believed that the larger silicon carbide particles tend to be fractured during extrusion and tensile testing, which reduces the composite's overall load carrying capacity. In hardness tests the predominantly macroscopic compressive load during indentation resulted in the particles being pushed and significant matrix flow. Thus, particle fracture during indentation was not observed. Detailed analyses using finite element modeling were performed to support the experimental results. Other artifacts regarding applying hardness testing to determining the tensile properties of metal matrix composites will also discussed.

Dynamic Deformation and Fracture Behavior of Novel Damage Tolerant Discontinuously Reinforced Aluminum Composites: Dr. M. Irfan[1]; Dr.V. Prakash[1]; Prof. John J. Lewandowski[3]; Dr. Warren H. Hunt, Jr.[3]; [1]Case Western Reserve University; Dept. of Mech. and Aero. Eng., Cleveland, OH 44106 USA; [2]Case Western Reserve University, Dept. Mats. Sci. and Eng., Cleveland, OH 44106 USA; [3]Al Consultants Group, 4530 William Penn Hwy, Murrysville, PA 15668 USA

Extrinsically toughened DRA composites have been processed to enhance their damage tolerance. The dynamic compression behavior of the composites is examined by employing the split Hopkinson pressure bar. The measured dynamic stress-strain response of the composites is correlated with the macro- and micro- damage mechanisms inferred from post examination of the impacted specimens. The dynamic fracture characteristics of the composites are obtained by impact loading pre-cracked three point bend specimens in a modified Hopkinson bar apparatus. The measured load-point force versus load-point displacement curves are used to, (a) estimate the energy required for dynamic crack initiation, and (b) understand the interaction of the dynamically propagating crack tip with the ductile phase reinforcements. The results indicate that the extrinsically toughened DRA composites absorb significantly greater energy during crack propagation. The level of extrinsic toughening is affected by the location, volume fraction and mechanical properties of the ductile phase reinforcements.

Surface Engineering in Materials Science I: Coating/Films Properties Evaluation (PE)-I

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee

Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816 USA; Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA; John Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA

Wednesday AM — Room: Canal B
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: V. H. Desai, University of Central Florida, Adv. Matls. Process. and Ana. Ctr., Orlando, FL 32816 USA; A. Kar, University of Central Florida, Schl. of Optics and Ctr. for Rsch. and Edu., Orlando, FL 32816 USA

8:30 AM

Characterization of Fe-Al Weld Overlay Coatings for Use in High Temperature Sulfidizing Environments: *S. W. Banovic*[1]; J. N. DuPont[1]; A. R. Marder[1]; [1]Lehigh University, Dept. of Matls. Sci. and Eng., 5 E. Packer Ave., Bethlehem, PA 18015 USA

Iron-aluminum alloys are currently under investigation for use as corrosion protective weld overlay coatings in reducing environments. These materials are relatively inexpensive, do not exhibit macro- or microsegregation, and have better corrosion resistance compared to convention Ni-based and stainless steel-type compositions presently in use. However, their use is limited due to weldability issues and their lack of corrosion characterization in very aggressive environments. Therefore, the objective of this research was to examine the sulfidation behavior of weldable Fe-Al compositions in highly aggressive reducing atmospheres. The high temperature corrosion behavior in environments containing oxygen and sulfur was characterized by thermogravimetric techniques. As-solidified Fe-Al alloys, with 0-20 wt% Al, were isothermally held at temperatures between 500-700°C for up to 100 hours in a reducing environment. Specially tailored gases maintained the partial pressure of oxygen and sulfur at each temperature [$p(O2) = 10^{A-25}$ atm, $p(S2) = 10^{A-4}$ atm]. Post-exposure characterization of the corrosion reaction products consisted of surface and cross-sectional microscopy in combination with energy dispersive spectroscopy and electron probe microanalysis. The corrosion behavior of weldable compositions of Fe-Al alloys in an oxidizing/sulfidizing atmosphere was found to be directly related to the aluminum content of the alloy. For high aluminum compositions (above 7.5 wt% Al), protection was afforded due to the development of a thin, continuous gamma alumina scale that inhibited rapid degradation of the alloy.

Increasing the aluminum content of the alloy was found to promote the formation and maintenance of this scale. For low aluminum alloys (less than 7.5 wt% Al), thick scales of sulfide phases were found. Growth of these scales was diffusion controlled and the addition of 5 wt% Al was observed to decrease the rate of the outer FeS scale development by an order of magnitude when compared to pure iron at 700°C. Intermediate aluminum contents (7.5 wt% Al) exhibited the initial formation of the protective scale that encountered mechanical failure at later times and the subsequent growth of corrosion product nodules. The results from this study indicate that weldable compositions of Fe-Al alloys (10 wt% Al) show excellent corrosion resistance to aggressive reducing environments. With the potential promise for applications requiring a combination of weldability and corrosion resistance in moderately reducing environments, these alloys are viable candidates for further evaluation for use as sulfidation resistant weld overlay coatings.

8:50 AM
Accelerated Liquid Metal Corrosion Response of Laser Surface Engineered VC Coating on Structural Steel: *Narendra B. Dahotre*[1]; Arvind Agarwal[1]; Lalitha R. Katipelli[1]; [1]University of Tennessee Space Institute, Dept. of Matls. Sci. & Eng., Ctr. for Laser Appl., B. H. Goethert Pkwy., Tullahoma, TN 37388 USA

VC coating on structural steel has been deposited using laser surface engineering (LSE) technique. The LSE technique involved spray deposition of the coating precursor material in a water-based organic vehicle followed by manipulation of laser beam for scanning of the surface to synthesize required coating material. The present work employed high power Nd-YAG laser with fiber optic beam delivery for synthesis/deposition of the coating. The coating has been evaluated for accelerated corrosion response to liquid A356 Al by immersing the coated samples for various durations. Optical and scanning microscopies were employed to observe topographical features along with SEM/EDS for elemental distribution and x-ray diffraction for phase identification. The effects of interaction between molten aluminum and VC are observed and evaluated in terms of types and quantities of reaction products. Such coatings are intended for protecting tool and die materials in casting industries and the present work will discuss their effectiveness for the applications.

9:10 AM
Oxidation and Wear Performance of Laser Surface Engineered TiC Coating on Al: Lalitha R. Katipelli[1]; Arvind Agarwal[1]; *Narendra B. Dahotre*[1]; [1]University of Tennessee Space Institute, Dept. of Matls. Sci. & Eng., Ctr. for Laser Appl., B.H. Goethert Pkwy., Tullahoma, TN 37388 USA

Refractory ceramic coatings are desirable for their physical and chemical natures, which make them suitable for many applications involving wear and corrosion. Such refractory coating if deposited on lightweight and soft material can extend their usefulness as structural materials in many commercial applications. In view of this possibility, synthesis of TiC coating on Al-alloy was achieved using laser surface engineering (LSE) technique. The LSE technique involved spray deposition of the coating precursor material in a water-based organic vehicle followed by manipulation of laser beam for scanning of the surface to synthesize required coating material. The present work employed high power Nd-YAG laser with fiber optic beam delivery for synthesis/deposition of the coating. In addition to microstructural evaluation of the deposited samples, the coated samples were also studied for their performance under dry sliding wear test and in elevated temperature ambient environments. Observations and evaluations will be presented.

9:30 AM
Effects of Nitrogen on the Electrochemical Passivation of Metal Nitride Coatings Produced by Ion Beam Assisted Deposition: *John Derek Demaree*[1]; Wendy E. Kosik[1]; Gary P. Halada[2]; Clive R. Clayton[2]; [1]Army Research Laboratory, Weapons & Matls. Rsch. Direct., AMRSL-WM-MC, Bldg. 4600, Rodman Matls. Rsch. Lab., Aberdeen Proving Ground, MD 21005-5069 USA; [2]State University of New York at Stony Brook, Dept. of Matls. Sci. & Eng., Stony Brook, NY 11794-2275 USA

Hard nitride coatings produced by ion beam assisted deposition (IBAD) are candidates to replace electroplated chromium (EHC) in a number of tribological applications, but the differences in the corrosion behavior of EHC and IBAD nitrides have not been fully characterized. In this study, coatings of Cr-N have been synthesized with IBAD, using 1000 eV nitrogen ions and e-beam vapor deposition. The chemical and phase composition of the coatings were examined using Rutherford backscattering spectrometry (RBS), the aqueous corrosion behavior of the alloys was studied by electrochemical techniques, and the chemistry of the passive oxide was examined using angle-resolved x-ray photoelectron spectroscopy (XPS). The effect of nitrogen (both in the coating and dissolved in the electrolyte) on the formation of oxyanions in the passive oxide will be discussed, as will the consequences of this oxyanion formation on corrosion resistance.

9:50 AM
Properties of DLC and a-C:N:H Films Layers Grown by PECVD and MWCVD Techniques: *S. Jonas*[1]; *T. Stapinski*[1]; E. Walasek[1]; [1]University of Mining and Metallurgy, Dept. of Elect., Al. Mickiewicza 30, Cracow PL 30-059 Poland

Diamond-like carbon (DLC) and amorphous carbon-nitrogen-hydrogen (a-C:N:H) films have been deposited by Microwave Plasma Enhanced Chemical Vapour Deposition (MWCVD) at 2.45 GHz and by Plasma Enhanced CVD (PECVD). The layers, owing to high hardness and excellent corrosion resistance at elevated temperatures have found application in manufacturing of cutting tools and materials for nuclear reactors and in microelectronics. The materials were examined by means of FTIR, SEM, optical spectroscopy and X-ray diffractometry. The surface morphology, chemical composition, chemical bonding structure, optical and mechanical properties of films were investigated. The authors optimized the technological parameters of CVD processes to obtain high quality materials for future applications.

10:10 AM Break

10:25 AM
Synthesis and Properties of BN, BCN and B/BN Thin Films Deposited by Ion Beam Sputtering Method: *S. Kurooka*[1]; T. Ikeda[1]; N. Iwamoto[2]; [1]Joint Research Consortium of FCT, Japan Fine Cer. Ctr., C/o NIMC 1-1, Higashi, Tsukuba, Ibaraki 305-8565 Japan; [2]Ion Engineering Research Institute Corporation, 4-4-24, Tsudayamate, Hirakata, Osaka 573-0128 Japan

Many workers have given much attention on c-BN and BCN coatings because of their prominent hardness as well as oxidation resistance at high temperature. In order to do clear the reason why c-BN and BCN coating induce crack occurrence after deposition, we have compared the difference of behavior among c-BN, ternary B-C-N and multilayer of B/BN. The hardness and oxidation resistance of films at high temperatures were investigated. The films were prepared by the ion beam sputtering method and crystal structure was investigated by FT-IR and ESCA. Also nanoindentation test was carried out. Moreover the thermal behavior at high temperatures up to 923K was studied in air.

10:45 AM Invited
Laser Surface Alloying of Ferritic Steel to Enhance Oxidation Resistance: *I. Manna*[1]; K. Kondala Rao[1]; S. K. Roy[1]; K. G. Watkins[2]; [1]I. I. T., Kharagpur, Dept. of Metallu. and Matls. Eng., Kharagpur, WB 721302 India; [2]University of Liverpool, Laser Grp., Liverpool L693GH UK

An attempt has been made to enhance the high temperature oxidation resistance (above 873 K) of 2.25Cr-1Mo ferritic steel by laser surface alloying (LSA) with co-deposited Cr using a continuous wave CO2 laser. The main process variables chosen for optimizing the LSA routine were laser power, scan speed of the sample-stage and powder feed rate. A detailed investigation indicates that the microstructure (studied by optical and scanning electron microscopy) and composition (determined by energy dispersive spectroscopic analysis) of the alloyed zone (AZ) are strong function of the LSA parameters. Following LSA, microhardness of the AZ increases to 450-550 VHN as compared to 220 VHN of the underlying substrate. Isothermal oxidation studies in air by thermogravimatric analysis at 973 and 1073 K for up to 100 h reveal that LSA may significantly enhance the oxidation resistance of ferritic steel during exposure to 100/200 K above the

current upper limit of use of the same steel for heat exchangers in thermal reactors. Post oxidation microstructural analysis suggests that an adherent and continuous Cr2O3 layer is responsible for the improvement in oxidation resistance. Finally, a detailed structure-property-LSA parameter correlation will be reported.

11:10 AM
Microstructural Evolution in Laser Surface Alloying of Ti with Ir for Developing Neural Stimulation Electrodes: *I. Manna*[1]; W. M. Steen[2]; K. G. Watkins[2]; [1]I. I. T., Kharagpur, Dept. of Metallu. and Matls. Eng., Kharagpur, WB 721302 India; [2]University of Liverpool, Laser Grp., Liverpool L693GH UK

Electrodes for neural stimulation in cochlar implants necessitate an extremely high charge injection/emission capacity and good drawability. Multi-layered activated Ir is known to possess one of the largest charge carrier density. However, Ir is expensive and brittle. On the other hand, Ti is relatively cheap, ductile and bio-compatible. Recently, an attempt was made to develop a Ti-based electrode with an Ir-rich Ir-Ti alloyed zone (AZ) by laser surface alloying (LSA). The present study is aimed at a detailed characterization of the microstructure, surface-chemistry and phase-distribution in the AZ of such an electrode prepared by LSA of Ti with Ir. LSA with an earlier determined optimum processing conditions appears to develop an AZ that can be conveniently divided into three regions with distinctly characteristic microstructure and composition. The influence of the LSA parameters on the morphology, identity and distribution of phases are discussed. Accordingly, a metastable phase diagram is proposed to account for the observed microstructure. Potentiodynamic polarization tests reveal that the amount of charge injected increases significantly following a special etching. Finally, an attempt was made to correlate the microstructure and composition of the AZ or electrode-tip with the electrochemical response, and assess its suitability for neural stimulation.

Ultrafine Grained Materials: Mechanical Behavior and Strengthening Mechanisms: I

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee, Shaping and Forming Committee

Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; S. L. Semiatin, Wright Laboratory, Materials Directorate, Dayton, OH 45440 USA; C. Suryanarayana, Colorado School of Mines, Department of Metal and Materials Engineering, Golden, CO 80401 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Wednesday AM — Room: Polk A/B
March 15, 2000 — Location: Opryland Convention Center

Session Chair: Rajiv S. Mishra, University of Missouri, Dept. of Metallu. Eng., Rolla, MO 65409 USA

8:30 AM Invited
Processing, Characterization, and Properties of Nano-crystalline Zinc: Xinghang Zhang[1]; *Carl C. Koch*[1]; [1]North Carolina State University, Matls. Sci. and Eng. Dept., Raleigh, NC 27695 USA

Zinc is an interesting metal to study deformation behavior of nanoscale microstructures. A study of nanocrystalline(nc) Zn prepared by a gas condensation method with grain sizes of 8-30 nm exhibited plastic deformation in compression at room temperature but failed in the elastic regime in tension. This is behavior typical of many nc materials at room temperature, but room temperature is 0.42 Tm for Zn. Superplasticity has been reported for nc Ni at 0.36 Tm. Dilute Zn(Al) alloys with a lower melting temperature such that room temperature is 0.45 Tm show superplastic behavior for 1 micron grain size samples at room temperature. We report here preliminary results of a study of nc Zn prepared by mechanical attrition. Zn powder milled at room temperature forms 3-6 mm size pellets due to its high ductility and extensive cold-welding. These pellets have an internal grain size of 25-30 nm. Milling at -180°C results in powder with a grain size of about 20 nm. Differential scanning calorimetry and x-ray diffraction were used to follow the structural changes with milling time and subsequent annealing. Maxima in both stored enthalpy and microstrain with milling time are observed, which implies a change in deformation mechanism with decreasing grain size. Results of preliminary mechanical property measurements on the nc Zn will be presented.

8:55 AM Invited
Strengthening Mechanisms in Ultra-Fine Scale Metallic Multilayers: *Amit Misra*[1]; Marc Verdier[1]; Harriet Kung[1]; Michael Nastasi[1]; J. D. Embury[1]; T. E. Mitchell[1]; J. P. Hirth[1]; [1]Los Alamos National Laboratory, Matls. Sci. and Tech. Div., MS K765, MST-CMS, Eniwetok Rd., Los Alamos, NM 87545 USA

Ultra-fine scale metallic multilayers may be synthesized to have strength levels close to the theoretical strength. These materials are also ideal for investigating the effects of length scales in plastic deformation of metallic materials. Refinement of the microstructure from the micron-scale to the nanometer-scale may give rise to different deformation modes involving continuum pile-up (classical Hall-Petch), discrete pile-up (modified Hall-Petch) and single dislocation (Orowan). Diffusion-based mechanisms such as Coble creep may be operative causing softening below a critical microstructural-scale in the nanometer range. Mechanical property results from a range of fcc/fcc and fcc/bcc Cu-based multilayers having different residual stresses, shear moduli mismatch between layers and lattice misfit strain between layers will be presented. The relative effects of layer thickness and grain size within the layers will be elucidated through the construction of two-dimensional maps that show layer thickness and grain size ranges over which different deformation mechanisms operate. By correlating the deformation mechanism maps with the experimental data, we show that these maps serve as guidelines for interpreting the scale-dependent strengthening or softening mechanisms in multilayers The effects of factors besides length scale that may influence the transition from one mechanism to another are discussed. This research is sponsored by DOE-OBES.

9:20 AM
Quasistatic and Dynamic Properties of Ultrafine-Grained Fe and W Alloys: D. Jia[2]; K. T. Ramesh[1]; M. Trexler[2]; *E. Ma*[1]; [1]Johns Hopkins University, Dept. of Matls. Sci. and Eng., Baltimore, MD 21218 USA; [2]Johns Hopkins University, Dept. of Mech. Eng., Baltimore, MD 21218 USA

Fe and W-based alloy powders with nanoscale grain sizes were produced by mechanical milling at room temperature. These powders were subsequently consolidated to full density bulk samples. The consolidated samples have grain sizes in the 50 nm to 1 micron range. Quasistatic and high-strain rate (Kolsky bar) tests have been conducted on these samples to determine the dependence of strength, ductility, strain hardening, and strain-rate sensitivity on grain size. The consolidation behavior with and without second-phase additions, the mechanical response under quasistatic versus dynamic conditions, and the potential of such alloys for kinetic energy penetrator applications are discussed.

9:40 AM
Ductile-Brittle Transition of Partially Crystallized Amorphous Al-Ni-Y Alloys: *Sun Ig Hong*[1]; Hyoung Seop Kim[1]; [1]Chungnam National University, Dept. of Metallu. Eng., Taedok Science Town, Taejon 305-764 Korea

In this study, a model on the ductile-brittle transition of partially crystallized amorphous Al-Ni-Y alloys is proposed. The strength of the partially crystalline Al-Ni-Y alloys was found to exhibit the maximum when the size of Al particle is 10 nm. Partially crystallized Al-Ni-Y alloys were also found to be brittle when the size and the volume fraction of the Al particles exceeded the optimum values. On the assumption that the diffusivity of Y is so low that Y atoms rejected

from FCC-Al particles are limited near interface region and the diffusivity of rejected Ni atoms is so high that the perfectly mixed solution is readily attained in the matrix, the reaction in three regions, Al particles, Y accumalated interface layers and amorphous matrix, were derived by balancing the quantity of each element. Al-Ni-Y alloys were assumed to become brittle when the solute content of interface layers reached the critical value (20%). The predictions based on the Y accumulation model are in good agreement with the published data available.

10:00 AM

Mechanical Properties of Nanocrystalline WC-Co Hardmetals: *Soon Hyung Hong*[1]; Seung Il Cha[1]; B. K. Kim[2]; G. H. Ha[2]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matl. Sci. and Eng., 373-1 Kusong-dong, Yusong-gu, Taejon 305-701 Korea; [2]Korea Institute of Machinery and Materials, Dept. of Matls. Processing, 66, Sangnam-dong, Changwon, Kyungnam 641-010 Korea

The mechanical properties of nanocrystalline WC-10Co-X hardmetals were investigated. Nanocrystalline precursor powders were prepared by spray drying of solution containing salts of W and Co. The precursor powders were reduced and carbonized into WC/Co powders by following mechano-chemical process. The initial WC powder size was about 100nm and was mixed homogeneously with Co binder. The powders were ball-milled in n-Hexane with ball-to-powder ratio of 5:1 for 24hrs and dried for 24hrs in drying oven. The mixed powders were sintered at 1375°C under pressure of 1mtorr. To compare the microstructures and mechanical properties with nanocrystalline hardmetals, the commercial WC with 0.57-1.27mm powders size were mixed with Co powders and followed by sintering at 1375°C under pressure of 1mtorr. Varying amount of Cr3C2, TaC and VC were added into nanocrystalline WC-10Co hardmetals as grain growth inhibitors. The microstructural parameters such as WC size, Co mean free path and WC/WC contiguity were sensitively dependent on addition of inhibitors. It was observed that Co phase was precipitated within faceted WC grains in sintered nanocrystalline hardmetals. The precipitated Co phase was fcc crystal structure and the average size was about 10nm. In case of the addition of 0.7% TaC/VC inhibitors, the WC size was about 300nm and the WC/WC contiguity was measured as 0.7. However, in case of the addition of 0.7% Cr3C2/VC inhibitors, the WC size was about 600nm and the WC/WC contiguity was lower value of 0.4. The transverse rupture strength was sensitively dependent on Co mean free path and WC/WC contiguity. The hardness of hardmetals was dependent on the WC particle size.

10:20 AM Break

10:30 AM Invited

Developing of SPD Processing for Enhancement of Properties in Metallic Materials: *R. Z. Valiev*[1]; [1]Ufa State Aviation Technical University, Instit. of Physics of Adv. Matls., K. Marks str., 12, Ufa 450000 Russia

Severe plastic deformation (SPD), i.e. intense plastic straining under high imposed pressure is the new technique for fabrication of ultrafine-grained metals and alloys. Methods of SPD processing can lead to a strong refinement of microstructure and formation of nanostructures in metallic materials; therefore, there is a potential to achieve their new and extraordinary properties. However, attaining such properties is a complex problem, which depends on different processing and microstructural parameters. This paper focuses on the relationship: SPD processing-nanostructures-new properties, for several pure metals and alloys. It has been shown that for microstructures of SPD materials it is typical to have a presence of not only very small grain sizes, but also specific defect structures, high internal stresses, crystallographic texture and often a change of a phase composition. From the other hand, these microstructural parameters are associated with details of SPD processing (applied method, processing routes, temperature, strain and strain rates and others). The examples of attaining very high tensile and fatigue strength in SPD materials are demonstrated. Special attention is stressed on development of the processed nanostructured materials for structural use. Enhanced superplasticity at low temperatures and high strain rates in SPD alloys is shown. The examples of practical applications of SPD materials are considered and discussed as well.

10:55 AM

Aging Effects in Bulk Metastable Nanostructured Alloys: *V. V. Stolyarov*[1]; R. Z. Valiev[1]; [1]Ufa State Aviation Technical University, Instit. of Physics of Adv. Matls., K.Marks str., 12, Ufa 450000 Russia

By now it is well established that severe plastic deformation has an ability in either strongly refine microstructure till up nanometer range and change essentially a phase composition forming highly metastable states in bulk samples of various alloys. This paper focuses on SPD processing by severe plastic torsion straining (SPTS) of several Ti and Al-based alloys and hard magnetic $Nd(Pr)_2Fe_{14}B$ based alloys. It is shown by TEM, X-ray studies and magnetic measurements that SPTS processing has resulted in formation of supersaturating solid solutions and amorphization of intermetallics phases in these alloys. During further heating aging effects take place and processed alloys demonstrate unique mechanical (very high strength, superplasticity) and magnetic hysteretic properties. The origin of metastable states and aging effects are discussed and explained using results of modeling defect structures of SPTS-processed alloys.

11:15 AM

Effect of Dispersed Oxide Particles on the Strength and Ductility of Ultrafine-Grain Steels: *Yoshikazu Sakai*[1]; Minoru Ohtaguchi[1]; Yuuji Kimura[1]; Kaneaki Tsuzaki[1]; [1]National Research Institute for Metals, Frontier Rsch. Ctr. for Structural Matls., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

Strength of alloys increases strongly with decreasing grain size. On the other hand, ductility, especially uniform elongation, decreases with decreasing grain size, namely, increasing strength. Strong plastic instability occur at room temperature and normal strain rates if the grain size is sufficiently fine. Therefore, it is necessary for keeping high strength-elongation balance of ultrafine-grain materials to be improved the rate of strain hardening. Dispersion of fine particles in the matrix might be effective for improving strain hardening. In this study, ultrafine-grain steels with a grain size less than 1 micron were produced using powder metallurgy method. Several kinds of iron powders with compositions in the range of 0.2-1.5 wt% oxygen were mechanically milled in an argon atmosphere and then hot rolled into bars, 600 mm long and 11 mm in diameter at 973K. The ultrafine-grain steels consist of a ferrite phase and fine particles of Fe3O4. Tensile test and microstructure observation for the steels were carried out. In the paper, we will discuss relationship between (1) formation of ultrafine-grain structure and oxygen content, (2) strength and grain size, (3) strength and ductility, (4) oxide particle size and milling time, and (5) effect of dispersed oxide particles on the strength and the ductility.

11:35 AM

Structure/Property Relations of Rapid Solidified Aluminum Alloys for High Temperature Applications: *S. G. Song*[1]; A. F. Giamei[1]; [1]United Technologies Research Center, MS 129-22, 411 Silver Ln., East Hartford, CT 06108 USA

Dispersion strengthened aluminum alloys via rapid solidification process are of increasing interest to materials researchers for elevated temperature applications because of their lightweight and low costs. Several newly developed dispersion strengthened aluminum alloys were investigated for potential high temperature applications. The bulk samples of the alloys were prepared by P/M process with high cooling rates of gas atomization. Mechanical testing and microscopy examination of the alloys were conducted and the results were analyzed to establish structure/property relationship. The metastable structure of the alloys resulting from rapid solidification was seen to vary with processing and heat treatment conditions. Optimal properties can be achieved through proper heat treatment processes.

12th International Symposium on Experimental Methods for Microgravity Materials Science: Session 4

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Thermodynamics & Phase Equilibria Committee

Program Organizers: Robert Schiffman, R.S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica, Bologna I-40126 Italy

Wednesday PM Room: Memphis A
March 15, 2000 Location: Opryland Convention Center

Session Chair: Michael R. Fiske, Morgan Research Corporation, Matls. Sci., Huntsville, AL 35805 USA

2:00 PM

Pore Bifurcation and Migration During Liquid Phase Sintering in Microgravity: *Yubin He*[1]; Saiyin Ye[1]; James E. Smith[1]; [1]University of Alabama, Dept. of Chem. and Matls. Eng., Huntsville, AL 35899 USA

Samples from Fe-Cu and Co-Cu systems processed in microgravity showed considerable pore formation and metamorphosis. Pore filling, coarsening and pore migration was found in most samples. Pores showed bifurcated behaviors based on their liquid volume fraction. These behaviors result from particle rearrangement, particle growth and different diffusion patterns that associated with interfacial energy differences, instabilities, and grain coarsening along the interface between phases. Volume diffusion exists throughout the entire process and dominates in high liquid volume-fraction samples. However, low liquid volume fraction and the presence of the agglomeration, which results in high local solid volume fraction, enhances the surface diffusion during the process which causes the pore breakup. Both volume diffusion and surface diffusion contributes to the pore migration. In this paper, a pore bifurcation and migration model will be presented to monitor the trends of shape changes of a pore in a microgravity.

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Transient Effects in Dendritic Solidification: *M. B. Koss*[1]; J. C. LaCombe[1]; M. E. Glicksman[1]; A. Chait[2]; V. Pines[2]; [1]Rensselaer Polytechnic Institute, CII 4225, 110 8th St., Troy, NY 12180-3590 USA; [2]NASA Glenn Research Center, Computational Microgravity Lab., Cleveland, OH 44135 USA

Dendritic solidification is a common mode of solidification. It is also an important model problem in non-equilibrium physics and pattern formation physics. Current theories couple the transfer of latent heat with selection mechanisms at the interface. Measurements of succinonitrile (SCN) dendrites in microgravity show reasonable agreement between heat transfer predictions and experiment. However, data and analysis for assessing interfacial physics theories are less definitive. We are studying, and will present data on, transient effects in dendritic growth of SCN. We employ the Clapeyron pressure/melting temperature effect to make a rapid change in a sample's hydrostatic pressure, and thereby rapidly change the specimen's melting temperature, forcing the dendrite to select a new steady-state. These initial measurements show some surprising and non-intuitive effects.

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Droplets Coarsening in Copper-Cobalt Metastable Immiscible Alloys: *Delin Li*[1]; Mike B. Robinson[2]; Tom J. Rathz[3]; [1]NRC, SD47, MSFC/NASA, Huntsville, AL 35812 USA; [2]NASA, SD 47, MSFC, Huntsville, AL 35812 USA; [3]UAH, SD47, MSFC/NASA, Huntsville, AL 35812 USA

Droplet growth in Cu-Co metastable immiscible alloys were studied by undercooling experiments and numerical modeling of the population dynamics equations. Increasing undercooling resulted in droplet coarsening during continuous cooling. From the isothermal treatment at a certain undercooling, the time evolution of droplet size distribution has been determined which can be divided into two regions: dispersed and non-uniform structure. For the droplet growth in the former, there is agreement between experiments and modeling of gravitational-induced coalescence, whereas for the latter, experiments deviate from calculations because the system is out of dispersion. Calculations also show that droplet coalescence caused by Stokes settling considerably prevails over that by Marangoni migration on the ground-based experiments, even though mass density of liquid Cu and Co is very close.

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Solidification of Fe-Ni-Mo Alloy from Undercooled Melt: *M. Murata*[1]; T. Aoyama[2]; I. Jimbo[1]; K. Kuribayashi[2]; [1]Tokai University, Instit. of Space & Astro. Sci., c/o Kazuhiko Kuribayashi, 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan; [2]The Institute of Space and Astronomical Science

The 18%Ni maraging steel is well known as one of the structural materials that have well balanced mechanical properties in strength and toughness. However, there has been often observed the degradation of these mechanical properties in the weld joint. One of the reasons for the degradation is the metastable δ phase inherited into the ambient temperature. In the present study, rapid solidification behavior from undercooled melt of Fe-Ni-Mo ternary alloy that is the base alloy of the maraging steel was precisely observed. The drop of the alloy, $Fe_{76}Ni_{18+X}Mo_{6-x}$ where x is from -2 to +2, was successfully levitated by the electromagnetic levitation furnace. More than 300 K of undercooling was achieved. Although the alloy whose Ni content is higher than that of the eutectic composition has the γ phase as their equilibrium primary phase, undercooling higher than T_c, the critical temperature where the activation energies for nucleation of both phases are equilibrated, promotes the nucleation of the δ phase showing the pronounced recalescence. The δ phase was nucleated secondary after the first recalescence. These behaviors were observed clearly by the high-speed video camera.

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Effects of Directional Solidification Rates on Aluminum Matrix Structures Reinforced with 23% Oriented SiC Whiskers: *C. Patuelli*[1]; R. Tognato[1]; [1]Universita di Bologna, Dept. di Fisica ed Instituto Nazionale di Fisica della Materia, Alma Mater Studiorum, Viale Berti Pichat 6/2, Bologna I-40127 Italy

TEM, SEM and AFM observations were carried out in order to investigate the SiC-Al interface after different unidirectional solidification rates. The morphology of the interface is examined taking into account the important role played by the SiC whisker surface on the faceted or not faceted growth of the Al Matrix.

4:00 PM

Solute Diffusion in Dilute Liquid Metals and Metalloids: *Reginald W. Smith*[1]; [1]Queen's University, Dept. of Matls. and Metallu. Eng., Kingston K7L 3N6 Canada

It is now well known that the diffusion coefficient (D) measured in a laboratory in low earth orbit (LEO) is less than the corresponding value measured in a terrestrial laboratory. However, all LEO laboratories are subject to transient accelerations (g-jitter) superimposed on the steady reduced gravity environment of the space platform. This paper reports recent measurements of the diffusion coefficients for dilute binary alloys of Pb-(Ag, Au, Sb), (Sb-(Ga, In), Bi-(Ag, Au, Sb), Sn-(Au, Sb), Al-(Fe, Ni, Si) and In-Sb in which g-jitter was suppressed.

It was found in all alloy systems that $D\chi T$ (temperature) if g-jitter was suppressed rather than $D\chi T^2$ as observed by earlier workers with g-jitter present. The significance of these results will be reviewed.

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Characterization of Equiaxed Microstructures in Refined Al-3.5Wt%Ni Directionally Solidified Under Diffusive Transport Conditions: *S. Verrier*[1]; [1]DEM/SPCM, CEA-Grenoble, 17 Rue Des Martyrs, Grenoble, Cedex 9 38054 France

Directional solidification of a refined Al-3.5%Ni alloy was performed with several velocity steps during the AGHF6 experiment in the STS-95 mission (November 98). Homogeneous multigrain equiaxed microstructures were obtained and their dependence on the solidification rate evidenced. The quantitative characterization of these microstructures by using original Image Analysis tools and several complementary metallographic technics (optical microscopy under polarized light, SEM), is presented here. The following morphological features were systematically determined on sections taken both parallel and perpendicular to the solidification direction: envelope of the grain, internal and external eutectic fractions, developed surface of the crystal, and distribution of the orientations of dendritic branches. Emphasis is put on the importance of measuring these different quantities in order to provide benchmark data for the validation of the models describing the formation of this type of microstructures and their rheological behaviour.

4:40 PM

Materials Science Experiment Module Accommodation within the Materials Science Research Rack 1 (MSRR-1) on the International Space Station: *Dinah B. Higgins*[1]; Kevin S. McCarley[1]; Robert R. Jayroe[2]; [1]NASA/MSFC, Microgravity Matls. Sci. Appl. Dept., MSFC Alabama, SD42, Huntsville, AL 35812 USA; [2]Pace & Waite Inc., NASA/MSFC, Microgravity Sci. & Appl. Div., SD42, Huntsville, AL 35812 USA

The Materials Science Research Rack 1 (MSRR-1) of the Materials Science Research Facility (MSRF) is a modular facility designed to accommodate two Experiment Modules (EM) simultaneously on board the International Space Station (ISS). One of these EMs will be the NASA/ESA EM being developed collaboratively by NASA and the European Space Agency. The other EM position will be occupied by various multi-user EMs that will be exchanged in-orbit to accommodate a variety of materials science investigations. This paper discusses the resources, services, and allocations available to the EMs and briefly describes performance capabilities of the EMs currently planned for flight.

Alumina and Bauxite: Bayer Process Chemistry Optimization

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Vito Cedro, Alcoa World Alumina, Pittsburgh, PA 15219 USA; Joe Anjier, Queensland Alumina Limited, Gladstone, Queensland 4680 Australia

Wednesday PM — Room: Jefferson B
March 15, 2000 — Location: Opryland Convention Center

Session Chair: K. I. Verghese, Alcan International, Ltd., Montreal, Quebec, Canada

2:00 PM

ClickSim-Bayer Process Simulation Model: *Karek Fort*[1]; [1]KF Engineering Services, Weiherweg 19, Volketswil 8604 Switzerland

The Bayer process simulation model is a valuable tool for the optimization of design, operation and energy issues in an alumina plant. Successful utilization of such a model was often restricted to a few "experts." The simulation model "ClickSim" provides a broad range of users with an easy-to-handle tool. It combines Bayer process know-how with object-oriented software. Some features of this software are: easy-to-use Graphical Uner Interface; mass and energy balance; streams contain main chemical solid and liquid components; all significant chemical reactions; steam and power generation; the possibility for users to develop their own units. The paper presents the model based on examples of problem solving such as plant water balance.

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The Equilibrium Approach to Causticisation for Optimising Liquor Causticity: *Gerald I. D. Roach*[1]; [1]Alcoa World Alumina, Rsrch. & Dev., Cockburn Rd., Kwinana, Western Australia 6167 Australia

The causticisation reaction using lime to convert sodium carbonate to sodium hydroxide, with the formation of calcium carbonate and calcium aluminate, has a thermodynamic equilibrium that depends upon liquor composition. The causticisation reaction has been studied from both a theoretical and practical standpoint; the reaction involves an intermediate that is formed almost instantaneously and has the approximate composition 3CaO.Al2O3.CaCO3.11H2O (commonly called monocarbonate or hydrocalumite). The equilibrium data have been used in carbonate balance models to help optimise causticisation in Alcoa World Alumina plants. Opportunities to increase plant causticity were identified, especially for Western Australian Refineries where the majority of the carbonate added to the liquor circuit is in the bauxite and the causticity of the liquor is relatively low. Some of the opportunities were difficult to realise because of the interactive effect on phosphate and calcia in the liquor that can affect both plant operations and product quality. Studies of the kinetics of decomposition of the monocarbonate species to either calcium carbonate or tricalcium aluminate are reported. The kinetic information has assisted in both understanding and optimising lime efficiency.

2:50 PM

Scale Control and Prevention of Hydrate Precipitation in Red Mud Filters: *John D. Kildea*[1]; Sophy Gotsis[1]; Anna Thomas[1]; [1]Nalco Australia, 2 Richardson St., Kwinana, West Australia 6167 Australia

Typical Bayer operations involve separation of waste solids through thickening/settling with remaining mud solids in the overflow liquor removed by a filtration step. One of the problems often encountered in this filtration step is the precipitation of small masses of trihydrate alumina. While such precipitation results in only a small product loss it often leads to substantial operation and maintenance problems for the filters. Application of Nalco 85711 to settler overflow liquor has been shown in a variety of test regimes to improve the stability of the aluminate in solution under filter conditions. This improvement can reduce hydrate precipitation in the filters. This reduces the operational and maintenance issues associated with filtration. Many plants prevent precipitation in the filters by maintaining a less-than-optimum aluminate concentration in the liquor. An improved stability of aluminate in filters can directly prevent precipitation and this may allow increased uptake of alumina values in digestion and directly lead to increased production yield. The dose rate of 85711 required to stabilise aluminate in solution in filtration is under 10 ppm while the impact of this product on trihydrate crystallisation under precipitation conditions is orders of magnitude greater (up to 1000 ppm). As a result, should any product pass beyond the filtration step it is highly unlikely that any detrimental impact on precipitation yield will be observed.

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Process Control in Alumina Refining-An Automated Plant: *Pierre Castelli*[1]; Bernard Bosca[2]; Christos Apostolakis[2]; Nikos Costis[2]; [1]Aluminium Pechiney, Aluval-BP 07, Voreppe, Cedex 38341 France; [2]Aluminium De Grece, Paralia Distomou, Saint Nicolas, Beotie 32003 Greece

Nowadays, to create added business value, it is imperative to modify workforce organization systems and automate processes whenever possible and cost-effective, by means of controlled investment projects. The following presentation will address the 3 year project under development at "Aluminium de Greece", concerning the automation of its alumina plant at Saint Nicolas (Greece). It will describe in three parts the new Production Organization (started up in parallel with the

project), the global architecture of the system and the project structure.

3:40 PM Break

3:50 PM
Optimization of Tricalcium Aluminate Use to Enhance Filtration in the Bayer Process: Sana U. Khan[1]; Winston Rennick[2]; Scott Barham[1]; *John T. Malito*[1]; [1]Nalco Chemical Company, 2 Anderson St., Botany, NSW 2019 Australia; [2]Alcoa Australia, Alcoa Wagerup, P.O. Box 84, Waroona, Western Australia 6215 Australia

Alumina refineries generally remove solids by a combination of sedimentation and filtration. In such processes 99.5% of the mud solids are removed by flocculation in settling vessels and less than 1% of the mud solids are removed by filtration where the settler overflow liquor is passed through pressure and less commonly, sand filters. Tricalcium aluminate hexahydrate (TCA) is used within the Bayer process as a filtration aid during filtration of the sodium aluminate liquors. This paper details studies which show that the filtration performance can be maintained with reduced TCA to mud ratios when Green Liquor Filtration Aid (GLFA) is used. The savings resulting from reduced TCA significantly outweigh the cost of using GLFA. Alternatively, improved filtration performance may be realized by adding the GLFA at constant TCA to mud ratios. In either case, substantial benefits are available. Mathematical analysis of the laboratory results allows the development of an algorithm, which adequately models filtration performance as a function of TCA/Mud ratio and GLFA dose. Laboratory pressure filtration tests were used to determine the effects of TCA and GLFA on cake compressibility and cake resistance. TCA was produced under a variety of adverse process conditions with the resulting product assessed for filtration efficiency. XRF, XRD and SEM analysis of the TCA produced correlated with filtration results.

4:15 PM
Control of Calcium Contamination in Alumina: The Ouro Preto Experience: *Paulo Marcio Figueiredo*[1]; Paul Hackett[1]; Steve Ostap[2]; [1]Alcan Aluminio do Brasil Ltda., Av. Americo R. Gianetti, Saramenha P.O. Box 1, Ouro Preto, MG Brazil; [2]Bayer Consultant, R. R. 3, Bath, Ontario K0H 1G0 Canada

In recent times the calcium content of alumina from the Ouro Pretor refinery has increased steadily to as high as 0.10% (as CaO). The bulk of this contamination arose from soluble calcium entering the liquor across the polishing filter presses. Investigations showed that the monocarbonate (3CaO.Al203.CaCO3.11H2O) content of the filter precoat was dissolving to levels up to 100 mg/l, probably due to the high organic carbon content of the liquor. In contrast, a precoat composed of tricalcium aluminate (TCAS) dissolved to only 15 mg/l. Unfortunately, while giving a better coverage of the filter cloth plus a low soluble calcium in liquor, the fine TCAS resulted in significantly lower filtration rates. The rates were largely restored by significantly reducing precoat thickness and by taking advantage of the improved cloth permeability afforded by the greater protection offered by the new precoat material. As a result of these changes, CaO in product has returned to less that 0.04% while maintaining production at target levels.

4:40 PM
Technology Roadmap for Baurite Residue Treatment and Utilization: Summary of Workshop Sponsored by the Aluminum Association: F. W. Williams, Alcoa World Alumina, Pt., Comfort, TX

The treatment and use of bauxite residue was the focus of a recent workshop sponsored by the Aluminum Association. Industry representatives were joined by several external technical experts to discuss various approaches and set priorities for possible collaborative research. Key technical, economic, environmental, and market factors were used to rank a large number of approaches. Priority areas targeted for additional research were to further evaluate the potential recovery of metals from the residue, explore the removal of desilication product (DSP) before it becomes residue, and to develop new separation technology to enable bauxite beneficiation. These and other results from the technology roadmap will be more fully discussed.

Aluminum Reduction Technology: Cell Operation/Innovations

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: John Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland, New Zealand; Georges J. Kipouros, Dalhousie University, Department of Mining and Metallurgical Engineering, Halifax, NS B3J2X4 Canada

Wednesday PM Room: Sewanee
March 15, 2000 Location: Opryland Convention Center

Session Chair: Mark P. Taylor, Comalco Aluminium Ltd., Brisbane, Queensland, 4001 Australia

2:00 PM
Thermodynamics of Electrochemical Reduction of Alumina: *Warren Haupin*[1]; Halvor Kvande[2]; [1]2820 Seventh Street, Lower Burrell, PA 15068 USA; [2]Hydro Aluminium Metal Products, Oslo N-0246 Norway

An updated calculation is presented of the theoretical minimum energy required to electrochemically reduce alumina to aluminum using either carbon anodes, which are consumed in the process, or inert anodes. Knowledge of this energy (the standard enthalpy of reaction, or deltaH°) is required for making heat balance calculations. The reversible cell potential, or Nernst potential, is calculated from the standard Gibbs energy of reaction, deltaG°, and the activities of reactants and products. This value is needed for calculating cell voltage. The present calculations update the 1976 thermodynamic analysis by Bratland, Grjotheim and Krohn. It was based upon floury alpha. Today the cell feed is sandy, largely gamma alumina. This changes deltaH°. However, the greatest difference is in the calculation of the reversible potential.

2:30 PM
Carbon Consumption and Current Efficiency Studies in a Laboratory Aluminium Cell Using the Oxygen Balance Method: *Jan Hives*[1]; Sverre Rolseth[2]; Henrik Gudbrandsen[2]; [1]Slovak University of Technology, Inorganic Tech., Radlinskeho 9, Bratislava 81237 Slovakia; [2]SINTEF Materials Technology, Electrolysis Grp., Trondheim N-7465 Norway

A method of continuous current efficiency measurements has been developed for use in a laboratory cell. The cell is a semi-large on laboratory scale, with a 42 mm diameter anode. To achieve minimal back and side reactions the cell was designed based on the following: all metal parts of the cell (holders, supporting wires, etc.) were covered with alumina shielding, used graphite crucible, with inner alumina lining, was covered with TiB2 paste and copper cathode was used to form Cu-Al alloy with a low aluminium activity in the melt. Electrolytic carbon consumption tests have been performed with PB-anodes samples. The tests were carried out in a newly developed apparatus where the anode gases were analysed continuously for CO and CO2 during the whole experiments, lasting 12 hours. Argon with known flow rate was used as inert carrier gas. This enabled us to make a total mass balance with respect to the amount of CO and CO2 evolved in the experiment. This amount was compared to the weight loss of the anode sample determined after the experiment. In the calculations of the weight loss of the anode corrections were made for the ash content and bath that had penetrated into the anode during electrolysis.

2:55 PM
Laboratory Experiments with Low-Temperature Slurry-Electrolyte Alumina Reduction Cells: *Craig W. Brown*[1]; [1]Northwest Aluminum Technologies, Rsrch. Dept., 3950 Sixth Ave. NW, Seattle, WA 98107 USA

The research presented addresses a novel total system concept as an alternative to conventional Hall-Heroult technology for aluminum

smelting. The electrolyte comprises fluoride salt mixtures with low-temperature melting compositions. The low temperatures require that the electrolyte be maintained as a slurry with undissolved alumina particles. This allows the use of metal alloy anodes. These are inert in that the main process offgas is oxygen, not carbon oxides. The concept includes an "inverted" cell in which the liner and cell bottom are anodic, and aluminum-wetted cathodes that are suspended vertically in the electrolyte. The system thus offers the advantages of both inert anode and wetted cathode technologies. The system concepts are presented, and ongoing laboratory experiments are described.

3:20 PM Break

3:40 PM Panel Discussion-Chair: Nolan Richards
"Aluminium Reduction-Where to from here?"
Invited Panelists: To be advised

Carbon Technology: Raw Materials

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Morten Sorlie, Elkem ASA Research, Vaagsbygd, Kristiansand N-4675 Norway; Christian Dreyer, Aluminium Pechiney, St Jean De Maurienne 73303 France

Wednesday PM Room: Knoxville A
March 15, 2000 Location: Opryland Convention Center

Session Chair: Trygve Foosnas, Hydro Aluminium A.S., Technology Center Ardal, Ovre Ardal N-6882 Norway

2:00 PM
Petroleum Derivatives as an Alternative to Binder Coal-Tar Pitches: M. Perez[1]; *Marcos Granda*[1]; R. Garcia[1]; E. Romero[2]; R. Menendez[1]; [1]Instituto Nacional del Carbon, CSIC, La Corredoria s/n, Apartado 73, Oviedo 33080 Spain; [2]Repsol Petroleo, S.A. Valled de Escombreras, Cartagena, Murcia 30350 Spain

Coal-tar pitches have been traditionally used as binder materials for the production of carbon anodes and graphite electrodes. However, the reduction in demand for metallurgical coke and the increasing concern about exposure to carcinogens at work, have led to the search for new binder materials for anodes and electrodes. Petroleum derivatives seem an attractive and interesting alternative for replacing, or at least competing with, binder coal-tar pitches. However, due to the different chemical composition of coal-tar and petroleum derivatives a very different behavior and properties can be expected. This paper focuses on the study of the composition and pyrolysis behavior of several petroleum residues, commercial binder coal-tar pitches and their blends with special emphasis on possible interactions on carbonization.

2:30 PM
Developing Coal Tar/Petroleum Pitches: *Robert H. Wombles*[1]; Melvin D. Kiser[2]; [1]Koppers Industries Inc., 1005 William Pitt Way, Pittsburgh, PA 15238-1362 USA; [2]Marathon Ashland Petroleum Company, P.O. Box 911, Catlettsburg, KY 41129 USA

Over the years the aluminum industry has evaluated the potential use of petroleum derived binders for producing anodes. Many of these evaluations produced less than desirable results. Most of these evaluations were driven by the potential of producing a more economical binder. In the future the evaluation of petroleum material may be driven by raw material availability and environmental regulations. This paper will discuss the rationale behind developing coal tar/petroleum binder pitches including the future of coal tar pitch supplies, the potential environmental advantages of coal tar petroleum pitches, and performance evaluations of coal tar petroleum pitches. Also, procedures for identifying acceptable petroleum components of coal tar/petroleum pitch will be discussed.

2:55 PM
Increasing Pitch Yield by Thermal Soaking of Tar for Pitch Manufacture: *Olof Malmros*[1]; Stewart H. Alsop[2]; *Nigel R. Turner*[3]; [1]Tarconord A/S, Avernakke, Nyborg DK-5800 Denmark; [2]Bitmac Limited, Meridian House, Normanby Rd., Scunthorpe, North Lincolnshire DN158QX UK; [3]Bitmac Limited, Scunthorpe Works, Dawes Ln., Scunthorpe, North Lincolnshire DN15 6UR UK

Abstract Submission for TMS 2000 Carbon Session Programme Techniques to increase the production of premium binder are a priority because the aluminium industry demand for coal tar pitch is predicted to exceed supply within the next ten years. According to current practice, anode binder pitch produced receives a small amount of thermal soaking during the tar distillation process. One technique to increase pitch availability is to thermally soak the coal tar prior to its distillation into pitch. Pre-treated tar was thermally soaked in the laboratory under nitrogen at pressure then vacuum distilled to produce pitch. Typical laboratory tar thermal soak conditions of 385ºC for 6 hours under nitrogen at 5 bar pressure gave an increase in pitch yield of 4 to 6% absolute. Toluene insolubles, quinoline insolubles and coking value were all increased, without producing any optically visible mesophase. Bench scale anodes fabricated with pitches made from thermally soaked tar had similar physical and chemical properties to control anodes fabricated with standard non-thermally soaked precursors.

3:20 PM
Characterization of Optical Texture in Cokes by Image Analysis: *Stein Rørvik*[1]; Marianne Aanvik[2]; Harald A. Øye[2]; Morten Sørlie[3]; [1]SINTEF Applied Chemistry, Inorganic Chem., Trondheim N-7465 Norway; [2]Norwegian University of Science and Technology, Instit. of Chem., Trondheim N-7491 Norway; [3]Elkem ASA Research, Vågsbygd N-4675 Norway

A fully automatic method for image analysis of optical texture of cokes has been developed. The method outputs mosaic index, which is a measure of optical domain size; and fiber index, which is a measure of optical domain anisotropy. The method has been applied to cokes made of high purity precursors doped with different aluminium/sulphur compounds and carbonized under pressure in the laboratory. A large variation in optical texture is seen, depending of the added compound type (organic/inorganic), amount added, and the position in the reactor the sample is taken from. The results are compared to calcined industrial petroleum cokes.

3:45 PM Break

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Reactivity and Texture of Cokes Doped with Aluminum Compounds: *Marianne Aanvik*[1]; Morten Sorlie[2]; Harald A. Oye[1]; [1]Norwegian University of Science and Technology, Dept. of Chem. and Inorganic Chem., Trondheim N-7491 Norway; [2]Elkem ASA Research, P.O. Box 8040 Vagsbygd, Kristiansand N-4675 Norway

High purity coke precursors are doped with aluminumacetylacetonate, aluminumfluoride, cryolite or sodiumfluoride and carbonized under pressure. The Al_2O_3 (from the added aluminumacetylacetonate), Na_3AlF_6 and NaF catalyzed both the air and CO_2 gasification reactions. AlF_3 however, inhibited the reactions. Addition of 1 wt% sulfur to the aluminumacetylacetonate-cokes had no apparent effect on neither the CO_2 nor the air reactivity. The additions interfered with the growth and coalescence of the mesophase particles during the carbonization process. The coke reactivity depends on the catalyst concentration as well as the resulting coke texture, expressed in terms of a mosaic index.

4:20 PM
Factors Influencing the Carboxy Reactivity of Calcined Coke: *Roy Allan Cahill*[1]; Ralph E. Gehlbach[1]; G. Scott Tittle[1]; [1]Reynolds Metals Company, Carbon and Environmental Tech. Dept., Smelter Tech. Lab., 4276 Second St., Muscle Shoals, AL 35661 USA

Examination of more than 180 laboratory calcined samples from 17 different refineries have resulted in a number of correlations between green and calcined coke properties and their affect on CO2 reactivity. The observed differences found in the carboxy reactivity behavior between East Asian and South American refineries and North American and Middle East refineries was of particular interest. The carboxy reactivity was found to be directly proportional to calcination temperature for the East Asian and South American refineries and inversely proportional for the North American and Middle East refin-

eries examined. Parameters such as%S,%N evolved, impurities, etc. were examined in an attempt to explain this phenomenon. The results of this study are discussed.

Cast Shop Technology: Fluxing and Filtration

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Paul Crepeau, General Motors Corporation, GM Powertrain Group, Pontiac, MI 48340-2920 USA; James N. O'Donnell, Commonwealth Aluminum Corporation, Department of Engineering, Louisville, KY 40202-2823 USA

Wednesday PM Room: Mississippi
March 15, 2000 Location: Opryland Convention Center

Session Chair: David V. Neff, Metallics Systems Company LP, Solon, OH 44139-2717 USA

2:00 PM Introductory Remarks

2:05 PM
Gas Fluxing of Molten Aluminum, Part 2: Removal of Alkali Metals: *Geoffrey K. Sigworth*[1]; [1]GKS Engineering Services, 116 Derby St., Johnstown, PA 15905 USA

The aluminum industry is under continual pressure to improve metal quality, while at the same time reduce costs. It is also necessary to reduce undesirable emissions to the environment. The only way to do this is through continual process optimization. In this review a theoretical analysis is given for the removal of dissolved alkali metals by chlorine fluxing, and suggestions are made for ways to improve the process. Particular emphasis is placed on minimization of chlorine use.

2:30 PM
The Alcan Compact Trough Degasser (ACD): *Martin Taylor*[1]; Hugo Van Schoonevelt[2]; [1]STAS, 1846 Outarde, Chicoutimi, Quebec G7K1H1 Canada; [2]A.N.O. Southwire

The Alcan Compact Trough Degasser (ACD) has been operating in many plants continuously for more than five years in North America, Europe, Australia, South Africa and Asia, not only within Alcan installations but also in non-Alcan plants. The early experience was with can stock production (ingots), but more recent experience has been with billets, remelt ingots and continuous casting. This paper updates performance results, not only for degassing but paying particular attention to alkaline removal and inclusion removal in non-Alcan plants. Extensive testing by various companies, sampled from the more than 40 plants now using the ACD, has shown that up to 90% alkaline removal is possible and up to 85% inclusion removal has also been achieved. Conditions are described under which these removal rates are obtained using both LimCa and PodFa results for inclusion removal and Alscan for hydrogen removal. These conditions include the relative humidity in the cast house, the type of alloys degassed, the temperature of the molten alloys and the quantities of chlorine and argon gas.

2:55 PM
Removal of Alkali Metals From Aluminum: *Eddie M. Williams*[1]; Ron W. McCarthy[1]; Sander A. Levy[1]; Geoffrey K. Sigworth[2]; [1]Reynolds Metals Company, Corp. Tech. Ctr., 13203 N. Enon Church Rd., Chester, VA 23834 USA; [2]GKS Engineering Services, 116 Derby St., Johnstown, PA 15905 USA

The alkali metals Na, and to a lesser extent Li and Ca, are found as undesirable impurities in aluminum. These must be removed to extremely low levels for acceptable product quality. The traditional way to refine the metal is to flux with chlorine and an inert gas. This process is poorly understood, however, and excessive chlorine consumption and chloride emissions to the atmosphere may occur. This paper presents the results of an extensive experimental program conducted to characterize and optimize the process of fluxing with chlorine. One of the unique features of the program is the utilization of an on-line emissions monitor to control the fluxing process. A 25,000 pound melting furnace in the casting complex at the Reynolds Metals Company Corporate Research Center was used in these trials. The furnace is tilting with porous plugs in the bottom for fluxing. In-line gas fluxing was also done outside the furnace with an efficient spinning degasser. The experimental results obtained in this study, together with a theoretical analysis, have allowed Reynolds to construct a detailed model of the alkali metal removal process. This understanding allows one to significantly reduce chlorine consumption and chloride emissions during the metal refining process.

3:20 PM
Evaluation of a Probe to Detect Salts in Molten Aluminum Alloys: *Dawid D. Smith*[1]; Kenneth R. Butcher[1]; [1]Selee Corporation, Eng., 700 Shepherd St., Hendersonville, NC 28792 USA

The use of choloine in the degassing of molten aluminum usually results in the formation of molten salts that compromise the performance of downstream filters and results in sporadic releases of inclusions. This paper describes the development and evaluation of a real time salt probe that detects the presence of liquid salts in molten aluminum. The theory behind the probe is discussed as well as the evaluation of the probes in experimental and production environments.

3:45 PM Break

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Evaluation of a Filter Developed to Remove Liquid Salts from Molten Aluminum: *Kenneth R. Butcher*[1]; Dawid D. Smith[1]; Leonard Aubrey[1]; [1]Selee Corporation, R&D, 700 Shepherd St., Hendersonville, NC 28792 USA

The use of chlorine as a fluxing gas in the treatment of molten aluminum usually results in the formation of molten salts that can compromise the performance of downstream filters and are associated with salt/oxide inclusion agglomerates. The purpose of developing a salt filter was to reduce or eliminate these problems and to improve final melt quality. Salt filtration is achieved by the use of microporous media designed to selectively adsorb the molten salt. The theory behind the development of the salt filter is presented as well as the DOE sponsored performance evaluation of a unit at the Alcoa Technology Center.

4:15 PM
A New Approach for the Investigation of the Fluid Flow in Ceramic Foam Filters: *Bettina Hübschen*[1]; Joachim G. Krüger[1]; Neil J. Keegan[2]; Wolfgang Schneider[3]; [1]RWTH Aachen, Aachen Germany; [2]Foseco Aluminium, Tamworth UK; [3]VAW Aluminium AG, Bonn, Germany

The filtration efficiency of ceramic foam filters depends strongly on the fluid flow in the channels of the filter. To investigate this two new water models were used. The first one was a full scale filter box model. Tracer tests on the CFF were made to investigate the change of flow behaviour with flow rate and filter pore size. The transient point from laminar to turbulent flow could be determined by pressure drop measurements. The second water model used was a specially designed single channel model to simulate the flow in one channel of a CFF. By pulse input of a tracer, the flow behaviour could be determined quantitatively. It was found that flow velocity is a crucial parameter for filtration efficiency. Filtration volume decreases rapidly if filtration velocity increases thus making deposition of particles more unlikely.

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2D Transient Mathematical Model of Aluminum Filtration: *Duygu Kocaefe*[1]; Rung Tien Bui[1]; Peter Waite[2]; [1]University of Quebec, Dept. of Appl. Sci., 555 Boul. De l'Universite, Chicoutimi, Quebec, Canada; [2]Alcan International Limited, P.O. Box 1250, Jonquiere, Quebec, Canada

A two-dimensional mathematical model has been developed for representing the dynamic behavior of the filter bed. This model involves the solution of Navier-Stokes equations for the flow field and the inclusion mass fraction equations for the concentration field. All

the relevant physical phenomena taking place in the bed are taken into account. The inclusion deposition and re-entrainment are represented as a source term of the mass fraction equation. The model accounts for the change of bed porosity and bed particle size with inclusion deposition as a function of bed depth and time. The pressure drop is calculated from Ergun equation. The mechanisms involved in aluminum filtration are not well known. However, the model appears to predict the trends observed in the industry reasonably well. In this paper, the various applications of the model such as effect of bed segregation on the filter behavior will be presented.

Cyclic Deformation and Fatigue of Materials; A Symposium in Honor of Professor Campbell Laird: Environmental Fatigue

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials

Program Organizers: Zhirui Wang, University of Toronto, Department of Metals and Materials Science, Toronto, Ontario Canada; Charles McMahon, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA; Pedro D. Peralta, Arizona State University, Department of Mechanical and Aerospace Engineering, Tempe, AZ 85287-6106 USA; J. K. Shang, University of Illinois, Department of Materials Science and Engineering, Urbana, IL 61801 USA

Wednesday PM — Room: Canal A
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: P. K. Liaw, The University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; H. Yaguchi, Kobe Steel Limited, Met. Rsch. Labs., Eng. Div., Japan

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Interface Damage Mechanism during High Temperature Fatigue Test in SiC Fiber-Reinforced Ti-15-3 Matrix Composite: *Yoshihisa Tanaka*[1]; Yutaka Kagawa[2]; Y. -F. Liu[1]; Chitoshi Masuda[1]; [1]National Research Institute for Metals, Processing Matl. Div., 1-2-1, Sengen, Tsukuba-shi, Ibaraki-ken 305-0047 Japan; [2]The University of Tokyo, Instit. of Industrial Sci., 7-55-1, Roppongi, Minato-ku, Tokyo 106-8558 Japan

It is well-known that the progress of interface debonding and sliding in fiber-reinforced composites plays an important role in the composite damage evolution. The present study focused on the interface damage mechanism occurring during isothermal fatigue test of an unnotched in SiC (SCS-6) fiber-reinforced Ti-15-3 alloy matrix composite. The post-fatigue morphology of the fiber-matrix interface was observed and evaluated quantitatively using atomic force microscope. Abrasive wear due to frictional sliding was observed at near the fiber fracture end and the mean amplitude of asperity roughness at the weared interface was found to decrease with the increase of fatigue cycles. The wear behavior showed strongly dependence on the interface relative sliding length. The relation between the interface sliding length and number of fatigue cycles was assessed using mean roughness amplitude along the interface. The effect of interfacial wear behavior on load transfer mechanisms was discussed.

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Influence of Test Temperature on Cyclic Stress Response and Fatigue Characteristics of Aluminum Alloy 2524: *D. Kolar*[1]; T. S. Srivatsan[1]; P. Magnusen[2]; [1]The University of Akron, Dept. of Mech. Eng., Akron, OH 44325-3903 USA; [2]Aluminum Company of America, Alco Tech. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA

The effective design of structural components for use in the aerospace, automotive and related ground transportation industries demands satisfactory performance from the material under conditions of cyclic stress and strain amplitude control. In this connection, a study has been made to understand the influence of test temperature on high strain low-cycle fatigue and fracture behavior of aluminum alloy 2524, in the T351 microstructural condition. Test specimens of the alloy were cyclically deformed using fully-reversed tension-compression loading, under total strain-amplitude control, over a range of strain amplitudes giving less than 104 cycles to failure, and at three different test temperatures. In this presentation, the low-cycle fatigue properties and fracture characteristics of the alloy will be highlighted in light of competing and mutually interactive influences of cyclic plastic strain amplitude, concomitant response stress, intrinsic microstructural effects and dislocation-microstructure interactions during cyclic straining. The influence of test temperature on cyclic stress response, cyclic strain-strain characteristics, fatigue-life and fracture behavior will be rationalized.

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Fatigue Behavior and Thermography of Pressure Vessel Steels at 1,000 Hz and 20 Hz: *P. K. Liaw*[1]; H. Wang[2]; L. Jiang[1]; B. Yang[1]; J. Y. Huang[3]; R. C. Kuo[3]; J. G. Huang[4]; [1]The University of Tennessee, Dept. of Matls. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Institute of Nuclear Energy Research, P.O. Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan, Taiwan 325; [4]Taiwan Power Company, Taipei, Taiwan

Fatigue behavior was investigated on reactor pressure vessel (RPV) steels (SA533B1I2) at 1,000 Hz and 20 Hz. The microstructure of the steel is a tempered martensite. Using round-bar specimens, loads were applied at 1,000 Hz and 20 Hz to study the fatigue characteristics of RPV steels at different frequencies. A thermographic infrared detection system has been used to measure the temperature profiles of fatigued specimens at 1,000 Hz and 20 Hz. Four stages of temperature profiles were observed during fatigue testing: an initial increase of the specimen temperature, a saturation-temperature region, an abrupt increase of temperature, and a drop of temperature following specimen failure. A shorter life of the test specimens has been observed at 1,000 Hz and 20 Hz. At the same time, a higher saturation-temperature above 100°C can be reached at 1,000 Hz, depending on the applied maximum stress level, while at 20 Hz, it approaches only about 23 to 24°C. A much greater temperature generated at 1,000 Hz than 20 Hz can result in a shorter fatigue life at 1,000 Hz. The micro-cracking behavior and the damage mechanisms responsible for the fatigue life will be elucidated. Research supported by Taiwan Power Company, National Science Foundation (DMI-9724476 and EEC-9527527 with Dr. D. Durham and Ms. M. Poats as contract monitors, respectively), the U.S. Department of Energy Secretary for Energy Efficiency and Renewable Energy, and Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program under contract DE-AC05-96OR22464, managed by Lockheed Martin Energy Research Corporation.

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Thermal Activation of Fatigue Damage: *William J. Baxter*[1]; Donald R. Lesuer[2]; Chol K. Syn[2]; [1]GM, R & D Ctr., 30500 Mound Rd., Warren, MI 48090-9055 USA; [2]Lawrence Livermore National Laboratory, Livermore, CA USA

The effect of temperature on the fatigue of aluminum alloys results from a combination of thermally induced changes in the microstructure and the intrinsic temperature dependence of the fatigue process. These two effects are separated for the first time, and it is shown that the intrinsic fatigue process is thermally activated. Two distinct regimes are identified. For fatigue lives $<3x10^6$ cycles, the activation energy is 86kJ/mole in 339 aluminum/15% Kaowool composites and 120 kJ/mole in unreinforced 5086 aluminum, i.e., in the range reported for diffusion in aluminum. For fatigue lives $>3x10^6$ cycles, the activation energy is 240 kJ/mole. The magnitude of all three activation barriers decreases in direct proportion to the applied cyclic stress. These results are consistent with a dislocation model of jog formation

at low cyclic stresses and the diffusion assisted motion of jogs at high cyclic stresses. The activation volumes correspond to dislocation loop lengths of 10 to 30 nm.

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Influence of Mercury Environment on Fatigue Behavior of Spallation Neutron Source (SNS) Target Materials: James T. Broome[1]; J. P. Strizak[2]; P. K. Liaw[1]; D. Fielden[1]; L. Jiang[1]; B. Yang[1]; S. J. Pawel[2]; L. K. Mansur[2]; J. R. DiStefano[2]; G. T. Yahr[2]; K. Farrel[2]; [1]University of Tennessee, Dept. of Matls. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

The Spallation Neutron Source (SNS) is an accelerator-based instrument that provides pulsed beams of neutrons by bombarding a mercury target with intense beams of 1-GeV protons. The facility is being designed to fulfill the needs of the neutron scattering community in the U. S. well into the next century. Mercury has been selected as a possible SNS target, and type 316 LN stainless steel has been chosen as a possible target container material. Fatigue behavior of 316 stainless steel will be investigated in an air as well as mercury environment. The samples will be subjected to fatigue loading in the frequency range of 1 Hz to 1000 Hz. The 1000 Hz tests will be performed in a specially constructed, temperature-controlled, soundproof room. It will be determined whether or not the frequency and mercury environment have a great influence on the fatigue crack initiation and crack growth rate behavior of the cyclically-loaded stainless steel. The ability to run tests at 1000 Hz will greatly reduce testing time and allow for the development of pertinent fatigue results at 109 cycles, which takes about eleven and a half days. The possibility of liquid metal embrittlement (LME) will also be investigated by comparing the results in air and mercury environments. The samples will be one of two types. The first type, for uniaxial testing, is a rod with a gauge section. The second type is a disc, which is used to simulate the loading condition of target container materials. Calculations have been performed to determine the displacement necessary to achieve bending stresses to exceed the yield strength of the samples at 1000 Hz. The fatigue results will be discussed in light of the application of 316 stainless steel as a target container material in the SNS system. Research sponsored by the Division of Materials Sciences, Office of Basic Energy Sciences, U.S. Department of Energy, under Contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation. In addition, J. B. and P. K. L. are very grateful to the support of the Oak Ridge National Laboratory under the contract number, LMER 4500007186, to the University of Tennessee. We would also like to acknowledge the support of the National Science Foundation [DMI-9724476 and EEC-9527527 with Dr. D. Durham and Ms. M. Routs as program managers, respectively].

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Corrosion Fatigue Behavior of a Zr-Based Bulk Amorphous Metal: *V. Schroeder*[1]; R. O. Ritchie[1]; [1]University of California, Dept. of Matls. Sci. and Mineral Eng., Berkeley, CA 94720-1760 USA

Recently, a number of strongly glass-forming metallic alloys have been found; one commercial alloys is $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ (at.%). Our initial investigations of this Zr-based metal revealed that it has high tensile strength (~1.9 GPa), good toughness (K_{Ic} ~18-59 MPa$\sqrt{m}$), and fatigue-crack growth properties in room air that are comparable to ductile crystalline metals. Despite such promising mechanical properties, in an aerated 0.5 M NaCl solution, we find that fatigue-crack growth rates increase dramatically, by 2-3 orders of magnitude. In the present study, we examine this large increase in fatigue-crack growth rates with the specific goal of identifying the role of environment in the fatigue-crack growth process. To this end, fatigue testing has been performed in a number of environments, including de-aerated 0.5 M NaCl, 0.05 M NaCl, de-ionized water, and 0.5 M $NaClO_4$, under both open circuit and potential control; in addition, static load testing has been performed in an aerated 0.5 M NaCl solution. It is found that the effect of sodium chloride solution depends on an anodic process that is active under cyclic and static loads (stress-corrosion cracking), and depends on the concentration of the solution and the identity of anions in the solution.

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Fatigue Damage Evaluation in Aluminum Heat Transfer Tubes by Measuring Dislocation Cell Thickness: *H. Yaguchi*[1]; H. Mitani[1]; K. Nagano[2]; T. Fujii[3]; M. Kato[3]; [1]Kobe Steel Limited, Matls. Rsch. Labs.; [2]Kobe Steel Limited, Eng. Div.; [3]Tokyo Institute of Technology, Dept. of Innovative and Eng. Matls.

A method to evaluate fatigue damage prior to crack formation in aluminum heat transfer tubes which undergo cyclic thermal stresses has been developed. In the low cycle fatigue region where dislocation cells are formed, cell wall thickness measurement has been found to be a useful method to evaluate fatigue damage both in laboratory experiments and in practical devices. The validity of the cell wall thickness measurement method on fatigue damage evaluation will be discussed.

General Abstracts: Intermetallics II: Aluminides

Sponsored by: TMS

Program Organizers: Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Alton T. Tabereaux, Reynolds Metals Company, Smelter Technology Laboratory, Muscle Shoals, AL 35661-1258 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Wednesday PM Room: Johnson A/B
March 15, 2000 Location: Opryland Convention Center

Session Chair: Joseph W. Newkirk, University of Missouri-Rolla, Dept. of Met. Eng., Rolla, MO 65409-0340 USA

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Effect of W, Mo and V on Tensile and Creep Properties of Orthorhombic Ti_2AlNb-Based Alloys: *Masuo Hagiwara*[1]; Feng Tang[1]; Satoshi Emura[1]; [1]National Research Institute for Metals, The Third Rsch. Grp., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

The orthorhombic (O-phase) Ti_2AlNb-based alloys are potential high temperature materials for aircraft applications. Ti-22Al-27Nb is a two phase O+β alloy and is said to have the best balance of tensile, creep and fracture toughness properties. In order to increase the mechanical properties further, transition elements (W Mo and V) were added to this alloy by substituting them for a part of Nb in an amount depending on their beta stability. Six new alloys containing different amount of W, Mo or V were prepared, and their microstructures, tensile and creep properties were investigated. The test results showed that W is effective on increasing the high temperature tensile strength and creep resistance.

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The O(rthorhombic)-Phase Embrittlement in Nb-Ti-Al Alloys: *Sundar Amancherla*[1]; Richard Grylls[2]; Hamish L. Fraser[1]; [1]The Ohio State University, Dept. of Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]GE Aircraft Engines, 1 Neumann Way, MB5, Cincinnati, OH 45215 USA

Advanced jet engine design requires new structural materials with higher temperature capability, lower density, adequate toughness and producibility. A candidate system is the Nb-Ti-Al system which exhibits good high temperature properties as well as room temperature ductility in certain compositional ranges. However, at intermediate temperatures (<1000°C), some alloys in this system show a substantial loss of ductility and tend to be embrittled. Thus, recent research conducted on the tensile behavior [Hou, 1994] and discontinuous yield behavior [Perungulam, 1997] of some alloys in this system shows

strong evidence for a significant decrease in ductility at intermediate temperatures. This embrilltlement may be attributed to the O-phase which forms at intermediate temperatures in these alloys. The O-phase has a stoichiometry of Ti_2AlNb with the orthorhombic crystal structure. Tensile tests have been conducted on specimens which have been heat-treated at various intermediate temperatures to determine whether or not there is a functional dependence between embrittlement and the formation of the O-phase. Samples deformed and fractured have been characterized using SEM and TEM techniques, and the relationship between microstructure and properties will be discussed.

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Microstructure and Mechanical Properties of As-Cast and Aged Nb-15 at.% Al-10 at% Ti, -25 at.% Ti and -40 at.% Ti Alloys: Dah-Liang Guan[1]; *Charlie R. Brooks*[1]; Peter K. Liaw[1]; [1]University of Tennessee, Matls. Sci. and Eng., Knoxville, TN 37996 USA

Compression tests have been conducted at 900°C at a strain rate of 10^{-4} s^{-1} for Nb-15 at.% Al-10 at.% Ti, -25 at.% Ti and -40 at.% Ti alloys in the initial conditions and after aging. The 10Ti and 25Ti alloys were in the as-cast condition, and the 40Ti alloy was in the hot-rolled condition. Specimens were aged for 10 or 100 h at 600, 900 and 1100°C. The microstructures were characterized by optical and scanning electron microscopy. There was a marked increase in strength for all conditions as the Nb content increased. This is attributed to solid solution strengthening by the Nb, but the increased presence of the harder Nb_3Al δ phase also contributed. The 40Ti alloy remained a single phase (B2 structure) for all heat treatments, and there was no significant effect of aging on the strength. The 25Ti and 10Ti alloys showed increased strength with increased aging temperature and time, which correlated with increasing amounts of the δ phase.

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The Strain-Induced Paramagnetic to Ferromagnetic Transition in FeAl: *Ian Baker*[1]; Y. Yang[1]; D. Wu[1]; P. Martin[2]; [1]Dartmouth College, Thayer School of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA; [2]Oak Ridge National Laboratory, Metals and Ceramics Div., Oak Ridge, TN 37831 USA

Single crystals of Fe-40Al were cold rolled, and then heated at 10 K min^{-1} in a differential scanning calorimeter (DSC), whereupon three exothermic peaks were observed. The cold rolling induced a transition from paramagnetism to ferromagnetism. At room temperature, the ferromagnetism disappeared upon annealing above the lowest-temperature exothermic peak, but at temperatures below 225 K the annealed specimen still showed a larger magnetic susceptibility than the unrolled single crystal. Analysis of the possible contributions to the ferromagnetic behavior suggests that antiphase boundaries (APBs), principally in APB tubes, are the source. Tensile tests on single crystals at a variety of temperatures followed by annealing in the DSC have been used to confirm this suggestion. This research was supported by National Science Foundation grant DMR 9973977 and U.S. Department of Energy grant DE-FG02-87ER4311.

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In-Situ TiC Reinforced Fe-28at%Al Intermetallic Based Composites Manufactured by MA-Pulse Discharge Sintering Process: *Se Hyun Ko*[1]; Bong Gyu Park[1]; Yong Ho Park[1]; Hitoshi Hashimoto[1]; Toshihiko Abe[1]; [1]Tohoku National Industrial Research Institute, Matls. Sys. Div., 4-2-1, Nigatake, Miyagino-ku, Sendai, Miyagi-ken 983-8551 Japan

Titanium carbide reinforced iron aluminide composites were manufactured by in-situ reaction between titanium and carbon during mechanical alloying and pulse discharge sintering processes. A homogeneous distribution of TiC particles in the Fe_3Al matrix was achieved after sintering. As for raw materials 99wt% iron powder, 99.9wt% aluminum powder, 99.9wt% titanium powder and 99.9wt% graphite powder were used. The ratio of titanium to carbon was 51.6:48.4, which is the ratio in TiC formed during in-situ melting process. To examine the effect of mechanical alloying, the specimens with the same composition were sintered after mixing. All the alloys were evaluated metallographically, by SEM-EDX analysis and by XRD for phase identification. Supersaturated iron solid solution was obtained by mechanical alloying, resulting in the extension of solid solubility in iron. Supersaturated aluminum, titanium and carbon were precipitated to Fe_3Al and TiC in sintering. The formation procedure and mechanism of in-situ composites will be presented.

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Solid-State Joining Behavior of TiAl Based Alloys with Various Microstructures: *Yuehui He*[1]; Peter K. Liaw[1]; *Baiyun Huang*[2]; [1]The University of Tennessee, Matls. Sci. and Eng. Dept., Knoxville, TN 37928-2200 USA; [2]Central South University of Technology, Powder Metallu. Rsch. Instit., Changsha, Hunan 410083 PRC

The solid-state joining behavior of TiAl based alloys with various microstructures for the hot-press/diffusion and superplastic-deformation/diffusion joining processes has been investigated using Vacuum Hot-press Furnace and Thermal-imitation Machine. The mechanical properties of the joined component materials at room temperature were measured. Test results show that the original microstructure significantly affects the joining property of TiAl based alloys. When the as-cast TiAl based alloy with coarse full lamellar microstructure was used as a couple components, the recrystallization would take place on the joining boundary for two solid-state joining processes. The fine duplex microstructure forms on the joining boundary. Remaining holes exist in the joining boundary due to the difficulty of plastic deformation on the joining surfaces. The fracture failure of the joining component of coarse full lamellar materials trends to occur in the joining boundary under the tensile load so that the joining component exhibits the lowest tensile properties at room temperature.

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The Effect of Ternary Additions on Solid State Transformations in Undercooled and Rapidly Quenched Gamma Titanium Aluminides: Timothy Montgomery Miller[1]; James Wittig[1]; William H. Hofmeister[1]; [1]Vanderbilt University, Matls. Sci. and Eng., 24 th and Highland, Nashville, TN 37212 USA

Microstructural analysis of undercooled and rapidly solidified $Ti_{50at\%}Al_{48at\%}X_{2at\%}$ (X = Cr, Nb, Mo) reveals different solid state phase transformation kinetics for each ternary alloy. Samples (0.47g ± 0.04g) are electromagnetically levitated, induction melted, undercooled below the melting point by flowing UHP helium over the liquid drop, and rapidly solidified by twin anvil splat quenching. The rapidly quenched microstructures were analyzed using standard optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Deeper undercooling results in a faster solidification rate, a thicker sample, and subsequent slower solid state cooling. For all alloys, rapid solidification produces an equiaxed hexagonal alpha structure, which transforms in a massive fashion to the tetragonal gamma phase. The lamellar two-phase microstructure is completely suppressed. Although the massive transformation is observed in all alloys for all undercoolings, the amount of massive transformation product is dependent upon the degree of undercooling and the type of ternary atom.

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Solidification Structures of Ti-Al-Mo Alloys: Paula R. Alonso[1]; *Eduardo E. Vicente*[1]; Gerardo H. Rubiolo[1]; [1]Comisión Nacional de Energía Atómica, Dept. de Materiales, Av. del Libertador 8250, Buenos Aires 1429 Argentina

The high-temperature Ti_2AlMo intermetallic compound (B2 structure) has been of recent interest as a component of refractory-based superalloys. However, its range of existence, both in temperature and composition, is not well established. In the present work the region of the ternary phase diagram close to the Ti_2AlMo stoichiometric composition was investigated. The alloys were prepared in a non-consumable electrode arc furnace. Their solidification structures were examined by optical and scanning electron microscopy, and analyzed by X-ray diffraction and electron-probe microanalysis. The results show that the B2 ordered phase is formed in the Ti-Al-Mo system.

General Recycling of Materials: Topics Related to Ferrous and Heavy Metals Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee

Program Organizers: Guy Fredrickson, Reynolds Metals Company, Smelter Technology Laboratory, Muscle Shoals, AL 35661 USA; Ilaria Accorsi, Daimler Chrysler, Toledo, OH 43606 USA

Wednesday PM — Room: Canal C
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: Gerrit H. Nijhof, Nijhof Consultancy, 2102 KN Heemstede, The Netherlands; Xiangwen Wang, Reynolds Metals Company, Smelter Tech. Lab., Muscle Shoals, AL 35661 USA

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The Use of Life Cycle Assessment (LCA) for the Environmental Evaluation of the Recycling of Galvanized Steel: *Christina Viklund-White*[1]; [1]Mefos, New Tech. Dept., P.O. Box 812, Luleå SE-97125 Sweden

The potential environmental impacts of the disposal of zinc used for galvanizing steel have, by the use of Life Cycle Assessment (LCA), been compared to recycling. A number of hypothetical recycling routes were composed involving three different EAF dust treatment processes, Waelz kiln, DC-furnace, and Ezinex, as well as scrap dezincing. The study shows that recycling of zinc used for galvanizing steel clearly has environmental benefits in that it saves zinc resources. However, zinc recovery does not necessarily decrease the potential impact on global warming and acidification. The magnitude of these two impact categories is tightly correlated with the amount and type of primary energy consumed in a process. The high electricity consumption in the dezincing process resulted in that this route has the highest impact on Global Warming Potential as well as Acidification Potential. The major part of the energy required for the production of primary zinc from primary as well as from secondary sources is consumed in the reduction of ZnO to Zn. The consequence is that the theoretically possible savings in primary energy by recycling zinc-containing materials is relatively small. The impact categories land use and waste generation are not considered in this study, but most likely the evaluation of impacts such would further increase the potential environmental impact of the landfill alternative. The results also show that the location of an electricity-intensive process highly affects the potential environmental impact. Comparing process and material alternatives in LCA studies where branch average data is used is therefore considerably more intricate than when LCA is used within a company.

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Use of Rotary Hearth Furnaces for the Recovery and Recycling of Iron Units from Steel Mill Wastes: An Idea Whose Time Has Come, or Will Never Come?: *Larry M. Southwick*[1]; [1]L.M. Southwick & Associates, 992 Marion Ave., Ste. 306, Cincinnati, OH 45229 USA

There is mounting interest in recovering and recycling iron units from steel mill wastes. Materials being utilized as feeds include minimill electric arc furnace dust (which is an EPA-classified hazardous waste), various dusts and sludges generated by the blast furnaces, BOF's and other units in integrated mills, as well as iron ore fines produced during the shipment of iron ore or taconite pellets. The interest is driven by (i) the increasing need for quality iron units, (ii) the potential decreasing scrap supply and the desire to find suitable alternatives, and (iii) current and potential future regulations relating to these wastes. At present, the most popular system to produce quality iron units from these wastes is a rotary hearth furnace (RHF). To date four plants have been built, two for minimill dusts, one for iron ore fines and one for a mix of fines and integrated waste oxides. None have been particularly successful to date. This paper reviews and critiques those operations, analyses other pro posed systems, looks back at previous efforts to utilize RHF's in similar service, and evaluates the long term potential for successful performance of these units.

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Decreasing Acid, Ammonia and Manganese Usage of Electrolytic Manganese Production by Recycling MOR Fume: *Arash M. Kasaaian*[1]; [1]Eramet Marietta Inc., P.O. Box 299, Marietta, OH 45750 USA

The current manganese source for production of electrolytic manganese at Marietta plant is a special slag from high carbon ferromanganese furnace operation. A new process has been developed which uses fume from refining of high carbon ferromanganese. The fume was very hard to recycle into the submerged arc furnaces. In the new process the fume has been used advantageously. The use of the fume increases the recovery of manganese and reduces the use of sulfuric acid and ammonia. The new process is based on leaching the fume with SO_2 under strict control of oxidation reduction potential, removing Fe by oxidation and removal of Si from the process solution.

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Characterization of Electric and Electronic Scrap: *Menad Nourreddine*[1]; Bo Björkman[1]; [1]Luleå University of Technology, Dept. of Process Metallu., Luleå SE-971 Sweden

Electric and electronic scraps are heterogeneous mixtures with mainly copper, aluminium, and iron attached to or mixed with various types of plastics and ceramics. Typically, small amounts of precious metals are incorporated in these mixtures. Today, electric and electronic scraps constitute an environmentally problematic fraction in waste disposal. Their physico-chemical characteristics have been investigated through chemical, x-ray diffraction, infrared spectroscopy, scanning electron microscopy and thermal analyses.

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Recycling of Manganese from Industrial Residues by an Electrochemical Leaching: *Philippe Henry*[1]; André Van Lierde[2]; [1]Meura Technologies, RD, Voie Minckelers 1, Louvain-la-Neuve 1348 Belgium; [2]Cath. University of Louvain-la-Neuve, Place Ste Barbe 2, Louvain-la-Neuve 1348 Belgium

A novel electrochemical cell has been developed for the recovery of MnO_2 from pyrolusite ore leaching residues loaded with Fe, Al, Ni, Co and Si impurities. This new process can be considered as an alternative to the traditional methods: SO_2, H_2O_2 or bioleaching. It has been observed to proceed fairly rapidly and could prove to be a more economically viable process, as well as being more environmentally acceptable. Electrochemical characterisation and kinetic studies have shown that MnO_2 is effectively leached by electrogenerated ferrous ions. The chemical reactions are: Cathode (inox): 2 Fe_{3+} + 2 e_- 2 Fe_{2+}, Bulk: MnO_2 + 2 Fe_{2+} + 4 H_+Mn_{2+} + 2 Fe_{3+} + 2H_2O, Anode (Pb-Ag): H_2O ½ O_2 + 2H_+ + 2 e_-, Global: MnO_2 + 2 H_{+Fe}= Mn_{2+}+H_2O + ½ O_2 The catholyte (MnO_2 pulp) circulates upward in the expanded mesh cathode separated from the sulphuric acid solution anolyte by a diaphragm which shows high mechanical resistance, low porosity, low potential drop, and reasonable cost. Laboratory and mini-pilot tests has led to the construction of an operating pilot plant (100 kg residue/hr) able to leach more than 95% of Mn, Ni, Co at 1000-1500 A/m_2, 50°C with high faradic yield (> 95%) and low power consumption. (3.2 kWh/kg Mn). New applications like the treatment of spent batteries, polluted soils and spent catalysts are now investigated at a laboratory and mini-pilot scale.

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New Technique for Recovery of Titanium Component from Blast Furnace Slag: *Yuhai Li*[1]; Taiping Lou[1]; Zhitong Sui[1]; [1]Northeastern University, School of Matls. and Metallu., P.O. Box 119, Shenyang 110006 PRC

More than 3 million tons of blast furnace slag containing about 25% TiO_2 are produced by smelting V-Ti bearing magnetite every year at

the Panzhihua Iron and Steel Company works. However, because of the dispersed distribution of the Ti component in various mineral phases, the very fine grain size (< 10μm), and the effects of complex interfacial combinations, it is difficult to recover the Ti component from the slag by traditional separation techniques. As a result, the slag has not been effectively utilized. It is necessary to find a way to recover the Ti component from the slag. The separated Ti enriched part can be used for producing TiO_2 pigment, and smelting Ti-Fe alloys. The process-minerology and SEM observation (with EDX qualitative analysis) show that several Ti-containing mineral phases occur in the solidified slag. Perovskite is the phase containing the highest concentration of titanium, but this phase accounts for less than half the total titanium content of the slag. In order to separate the perovskite phase from the slag by mineral dressing methods, it is first desirable to fully grow and coarsen the phase. The present work is a study of the effects of additives and heat-treatment on the precipitation and growth behavior of the perovskite phase in the slag.

5:05 PM

Metal Values Recovery from NiMH Batteries: *Carla Lupi*[1]; Daniela Pilone[1]; Giuseppe Cannavale[2]; Alessandro Pescetelli[3]; [1]University of Roma "La Sapienza", Dept. ICMMPM, Via Eudossiana 18, Roma 00184 Italy; [2]Mo.Smo.De. S.a.s., Crotone, Italy; [3]Texeco Eng., Roma, Italy

NiMH sealed cells (portable cells) are today widely used in all consumer applications replacing primary alkaline batteries: wireless mobile communication, portable computers and camcorders, are the largest application segments. The Italian market of NiMH cells is steadily growing, following the impressive penetration rate of the cellular phones. The organization of a national collecting system and correct recycling process are the key factors to prevent environmental impact associated to these wastes, while the metal values recovery can improve the feasibility of the recycling process. The University of Rome, Texeco Engineering S.r.l., and MO.SMO.DE. S.a.s. have developed a combination of mechanical and hydrometallurgical processing to recover Nickel, and Cobalt salable products and rare earths intermediate products. A new plant located in South Italy, designed after this technology, is able to recycle waste Ni-MH batteries collected in the Italian territory, together with other industrial and portable batteries based on different chemistries. The process is able to treat both individual cells and plastic power packs and includes as basic steps an original crushing and elutriation treatment to separate plastic, metallics, and active mass components that are subsequently treated by a hydrometallurgical process to recover Ni, Co, and RE. The hydrometallurgical main steps are: acidic leaching, R.E separation, multistage precipitation of Ni, Co and Fe and final solvent extraction on dissolved Ni and Co salts. For each operation the operative condition have been determined.

5:30 PM

Hydrometallurgical Routes for Recycling of Used Alkaline Batteries: *Cleusa Cristina Bueno Martha de Souza*[1]; Jorge Alberto Soares Tenorio[1]; [1]University of Sao Paulo, Dept. of Metallu. and Matls. Eng., Av. Prof. Mello Moraes, 2463, Sao Paulo-SP 05508-900 Brazil

Currently in Brazil, the final disposal of spent batteries includes sanitary landfills (for batteries from domestic sources) and hazardous waste industrial landfills (for batteries from industrial sources). The environmental effects caused by the improper disposal of spent batteries have been discussed in the literature for years. Some techniques have been proposed for recycling spent batteries which also have the economic advantage of recovering metals such as Cd, Hg, Pb, and Zn. The processing of spent batteries by hydrometallurgical techniques is an efficient method for recovering such metals. This paper discusses the characterization of spent alkalline batteries and the experimental results of leaching tests using sulfuric acid as the leachate. After batteries dismantling by mineral processing techniques, the material produced was characterized by XRD and AA spectrophotometry to determine the phases present. The batch laboratory experiments were conducted to determine the appropriate leaching conditions for the recovery of zinc.

5:55 PM

Hydrometallurgical Recycling of Nickel-Cadmium Batteries: Iliija B. Ilic[1]; *Srecko R. Stopic*[1]; [1]University of Belgrade, Faculty of Tech. and Metallu., Karnegijeva 4, P.O. Box 5303, Belgrade 11000 Yugoslavia

The recycling of Ni-Cd batteries is a big problem in automobile industry in the world for a long time. Generally, these spent batteries are recycled in a pyrometallurgical process, whereby the shred cells are blended with other nickel, chrome and iron bearing wastes and smelted in an electric furnace, to produce nickel alloys pigs, used as feedstock by specialty steel producers. The cadmium portion is converted to pellets in a retorting unit, for reuse, primarily in the battery industry. In this work the waste dust obtained in production of Ni-Cd batteries contained in percentage value: Ni-29.258; Cd-6.848; Co-1.743; Fe-0.0154, Zn-0.0085, moisture-20.00 and remainder. Recycling of Ni-Cd batteries was performed with ammonia-ammonium carbonate solution below 333 K, and with sulfuric acid in temperature range from 298 to 373 K. Experimental conditions of leaching and deposition of nickel and cadmium were investigated by hydrometallurgical methods. Relatively spherical particles of nickel and cadmium have just been obtained by leaching of the waste dust. Special attention was paid to the separation of nickel from cadmium. The main goal of this study was to safely and properly collect and recycle whole Ni-Cd battery waste reusing all component materials.

High Resolution Electron Microscopy in Materials Science: Other Applications and Structures

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

Program Organizers: Diane E. Albert, Los Alamos National Laboratory, MST-6, The Metallurgy Group, Los Alamos, NM 87545 USA; Martin Allen Crimp, Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824-1226 USA; John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA

Wednesday PM — Room: Canal D
March 15, 2000 — Location: Opryland Convention Center

Session Chair: John E. Smugeresky, Sandia National Laboratory, Livermore, CA 94550 USA

2:00 PM

HREM of Unusual Incommensurate, Modulated and Disordered Structures in Various Metal Disilicides: *Terence E. Mitchell*[1]; Amit Misra[1]; [1]Los Alamos National Laboratory, Ctr. for Matls. Sci., MS-K765, Los Alamos, NM 87545 USA

Most of the refractory metal disilicides are metallic and have $C11_b$, C40 or C54 structures which are formed by various stacking sequences of hexagonal layers. The Group VII disilicides are unusual in that they are semi-conductors, they are silicon-deficient, and they have a stoichiometry close to $MSi_{1.75}$ (M = Mn, Tc, Re). In addition, $MnSi_{2-x}$ has been found to exhibit incommensurate structures and Nowotny "chimney-ladder" structures based on the C54 $TiSi_2$ unit cell. Recently we have found that melt-processed $ReSi_{2-x}$ exhibits similar incommensurate structures except that these are based on the tetragonal $C11_b$ $MoSi_2$ structure. The incommensurate periodicity gives rise to orthorhombic and monoclinic distortions. Annealing at 1250°C leads to the formation of a commensurate structure with a monoclinic unit cell four times the size of the $C11_b$ unit cell. On the other hand, $ReSi_{2-x}$ films formed by reactive deposition on Si substrates at 650°C are epitaxial and have the C11b structure; however, they contain a very high density of (001) stacking faults which are apparently formed by the collapse of vacant Si planes, leading to one-dimensional disorder of the $C11_b$ structure. By comparison, $(Mo,Re)Si_{2-x}$ films, formed in the same way and also epitaxial, have a highly regular modulated

structure (possibly spinodal) with sinusoidal variations of the Mo/Re ratio normal to the substrate. These phenomena have been studied by electron diffraction, high resolution electron microscopy and EDS techniques. The phenomena are tied strongly to the difficulty of accommodating the high density of structural Si vacancies, leading to a series of metastable "frustrated" structures.

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Characterization of Nanostructured Materials by HREM: *Harriet Kung*[1]; [1]Los Alamos National Laboratory, Matls. Sci. and Tech. Div., MS G755, Los Alamos, NM 87545 USA

Nanocrystalline materials have been attracting rapidly increasing interest in the last decade mainly due to the potential gain in a wide range of engineering applications. One characteristic feature of nanostructured materials is the high volume fraction of surfaces, grain boundaries and interfaces. The high surface/interface to volume ratio has contributed to interesting physical properties in the areas of catalysts, magnetics, optics, and structural applications. The structural constraint may also stabilize unusual phases with unique properties. HREM is an excellent tool in the characterization of nanostructured materials due to its superb spatial resolution. In this presentation, I will review several HREM studies on the characterization of the defect structure, grain boundary structure, unusual phases, and structural stability of metallic powders/compacts and multilayers. Specifically, the role of defects in affecting the mechanical properties of nanostructured materials will be discussed.

3:00 PM Break

3:30 PM

HRTEM Analysis of GMR Spin Valve Multilayers: Hong Geng[1]; *John W. Heckman*[1]; William P. Pratt[2]; Jack Bass[2]; Martin A. Crimp[1]; [1]Michigan State University, Matls. Sci. and Mech., East Lansing, MI 48824 USA; [2]Michigan State University, Dept. of Phys. and Astro., East Lansing, MI 48824 USA

The Giant Magnetoresistance (GMR) effect in multilayer spin valves (SV) is very sensitive to the film structure. Therefore, it is important to characterize the structure of the SV multilayers to correlate this with their magnetic properties. In this study, GMR SV structures, of the form [Nb//Cu/FeMn/Permalloy(Py)/Cu/Py//Nb] and [Nb//Ag/Py/Ag/Py/FeMn//Nb], grown on Si (001) substrates, were characterized using conventional and high-resolution transmission electron microscopies (CTEM and HRTEM). CTEM revealed that the layers are generally polycrystalline with columnar/epitaxial growth through the layers. HRTEM images revealed that the growth of the Nb contacts and SV layers occurred on close-packed planes ({110} for BCC and {111} for FCC). HRTEM analysis also revealed that non-equilibrium structures exist in certain regions of some of the SV layers. Computer simulations and comparisons of the proposed non-equilibrium structures, within the imaging and resolution limits of the microscope used, support these findings.

3:50 PM

HRTEM Study of Epitaxial Magnetic Multilayers and Spin-Valves Grown by Dc Sputtering: *Hong Geng*[1]; Reza Loloee[2]; William P. Pratt[2]; Martin A. Crimp[1]; [1]Michigan State University, Dept. of Matls. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824-1226 USA; [2]Michigan State University, Dept. of Phys. and Astro., 2B Physics Bldg., East Lansing, MI 48824 USA

The study of giant magnetoresistance (GMR) in magnetic multilayers and spin-valves has shown that the electrical transport in these materials is structurally dependent at the micro and atomic level, with film growth direction and interfacial structure playing important roles. In this study, (Cu/Co) magnetic multilayers and (Cu/Py/Cu/Py/FeMn) spin-valves have been epitaxially grown using dc magnetron sputtering on (1-10) Nb buffer layers that were deposited on (11-20) Al_2O_3 substrates, where Py=permalloy (NiFe). Cross-section conventional and high-resolution transmission electron microscopy (CTEM and HRTEM) studies have been performed to investigate microstructural features of the multilayers, such as degree of epitaxy, interfacial structure, layer quality, layer orientations and defects. Fast Fourier transform (FFT) image analysis has been employed to assist interpretation of the HRTEM images. The results will be compared with those from similar polycrystalline magnetic multilayers and spin valves grown by dc sputtering. This work was supported in part by the MSU CFMR, and by the US NSF under grants MRSEC DMR 94-00417 and 98-09688.

4:10 PM

In-Situ TEM Studies of Abnormal Grain Growth in Nano-crystalline Ag: *Rand Dannenberg*[1]; E. Stach[2]; J. R. Groza[3]; B. J. Dresser[1]; [1]BOC Coating Technology, Fairfield, CA 94533 USA; [2]National Center for Electron Microscopy, Lawrence Berkeley Lab., Berkeley, CA USA; [3]University of California-Davis, Dept. of Chem. Eng. and Matls. Sci., Davis, CA USA

80 nm thick Ag films were DC sputter deposited onto back-etched amorphous silicon nitride membranes. Specimens were annealed in a heating stage in an in-situ TEM for various temperatures and hold times. The grain size distribution of the as-deposited films is bi-modal, with large abnormal grains with 100 nm diameters, embedded in a matrix of smaller grains of 15 nm diameters. Coarsening begins at temperatures of approximately 100°C, and quickly reaches a plateau. The growth process restarts only after sufficient temperature increases, and plateaus at each succeeding temperature. Using a variation of the Mullins-Von Neumann law, the activation energy for the abnormal growth is found to be 0.23 eV consistent with surface diffusion. Grain growth appears to stop above temperatures of 350°C, eventually leading to triple junction pore formation at 350°C and de-wetting of the film from the substrate at 600°C.

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Hydrogen-Induced Phase Transformations in Titanium Aluminides: *Marc De Graef*[1]; Bryan Molloseau[1]; [1]Carnegie Mellon University, Matls. Sci. and Eng., Roberts Eng. Hall 130, 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

We report on the structure determination by means of high resolution transmission electron microscopy and neutron diffraction of a new ternary hydride in a cast Ti-48Al-2Cr-2Nb duplex alloy, hydrogen charged at 800°C and 13.8 MPa. The hydride has a structure belonging to the Cmm2 space group and is pseudo-tetragonal. The hydride grows from the α_2 phase and completely replaces the latter. HRTEM was used in combination with electron and neutron diffraction to determine the metal atom positions. The Cmm2 hydride can be considered to be the end member of a series of hydrides which differ from each other in terms of defect densities. We will also report on the analysis of three different planar defects in the related tetragonal θ hydride.

High Temperature Processes for Waste Treatment & Minimization: I

Sponsored by: Extraction & Processing Division, Waste Treatment & Minimization Committee

Program Organizers: Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA; Patrick R. Taylor, University of Idaho, Department of Metals & Mining Engineering, Moscow, ID 83843-3024 USA

Wednesday PM — Room: Jackson A/B
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: Brajendra Mishra, Colorado School of Mines, Metallu. & Matls. Eng., Golden, CO 80401 USA; Patrick R. Taylor, University of Idaho, Metallu. & Min., Moscow, ID 83843 USA

2:00 PM

Nitrogen Oxides of Formation during the Combustion of the Blast Furnace and Coke Gases and Their Mixtures: *Leonid P. Gres*[1]; *Michael I. Ivanov*[1]; *Alexey N. Lozhko*[1]; [1]State Metallurgical Academy of Ukraine, Therm. Eng. Dept., 4 Gagarin Prosp, Dniepropetrovsk 320635UA Ukraine

Industrial heat generating facilities and automobiles, in which various types of fuels are combusted comprise the main sources of deleterious emissions (NOx, SOx, CO, and C_20H_{12}) into the environment. The available literature contains adequate data on the mechanisms of formation of nitrogen and carbon oxides and their concentration, when heat generating fuels are burned, such as fuel oil, powdered coal, natural gas. But as far as metallurgical fuels are concerned, such as blast furnace gas and coke gas and their mixtures, their is lack of such data and very often the available data are contradictory. Studies have been carried out to determine the concentrations of deleterious emissions in the off-gases of blast furnace stoves, as well as type and concentration of the nitrogen-containing blast furnace gas components. Calculations have shown that if all the combined nitrogen present in me blast furnace gas in form of gaseous compounds and dissolved in the condensate is fully transformed into NOx, then the portion of the "fuel" nitrogen oxides my reach 10-68%. In order to compare the results of the studies, samples of water from the coke quenching tower and of the coke gas condensate were also selected. Thus, during the combustion of the blast furnace and coke gases or their mixture-"fuel" NOx are formed which can make a substantial contribution to the "thermal" nitrogen oxides.

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Titanium Behavior and Applications in Waste Treatment Processes: J. S. Grauman[1]; *Stephen P. Fox*[1]; [1]TIMET, Henderson Tech. Lab., P.O. Box 2128, Henderson, NV 89009 USA

Titanium has provided nearly twenty years of service in wet air oxidation (WAO) waste treatment facilities. In addition, titanium has been used for more than ten years as lining material in flue gas desulfurization (FGD) systems of coal fired power plants. The drive to use titanium in these type of systems has been the unusually corrosive environments that can be encountered when chemically treating a waste product. Highly oxidizing, acidic solutions must sometimes be employed (or are the result) of waste stream processing. Titanium and its alloys are uniquely suited to withstand many of the aggressive environments often seen in waste processing systems. This excellent corrosion behavior has more recently been utilized in the new generation processes known as super critical waste oxidation (SCWO). Titanium has been identified as one of the few materials capable of surviving the rigors of this process, designed to treat toxic organic chemicals producing simple non-toxic chemicals such as water and CO_2. This paper will review the current and possible future applications for titanium in waste treatment systems, as well as the unique corrosion behavior of titanium that allows its use in these very demanding processes.

3:00 PM

Direct Smelting of Zinc Smelter Wastes in the Electric Arc Furnace: *W. K. O'Connor*[1]; D. C. Dahlin[1]; P. C. Turner[1]; [1]Albany Research Center, Off. of Fossil Ene., 1450 Queen Ave. S.W., Albany, OR 97321 USA

An evaluation of the zinc smelter wastes from a former smelter site in Spelter, West Virginia was conducted at the U.S. Department of Energy (DOE) Albany Research Center (ARC), in Albany, Oregon. This evaluation included a characterization study and direct smelting tests conducted in a bench-scale electric arc furnace (EAF). In contrast to other thermal treatment technologies considered for these wastes (i.e., fluidized bed combustion), direct smelting in the EAF can process 100% of the wastes without prior beneficiation, and utilizes solid oxidant additions (in this case iron ore) rather than air to oxidize the carbon. This solid oxidant addition is advantageous because it results in the production of several value-added furnace products. These products include cast iron resulting from the reduction of the iron oxides, baghouse dust containing virtually all of the zinc from the residues, and a clean (nonhazardous) slag product suitable for recycle as concrete aggregate or construction fill. Offgas from the smelting furnace also holds potential as fuel gas for cogeneration. The direct smelting tests conducted thus far were successful at producing the desired furnace products, exhibiting the potential for complete recycle of the waste pile. A cast iron product of comparable quality to conventional cast irons was produced, at nearly 80% iron recovery to the metal. The slag product was determined to be nonhazardous, based on the EPA TCLP. Lead and zinc partitioning to the dust product was greater than 99%. The material balance over the furnace indicates that for every ton of feed processed, 540 lb. of cast iron, 600 lb. of slag, and 150 lb. of dust would be produced. Energy consumption was roughly 1.10 kW×h/lb of feed material. Prior experience suggests that these figures measured in the bench-scale furnace would translate to 0.40 to 0.50 kW×h/lb at the industrial-scale.

3:30 PM Break

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Electron Beam Processing for Waste Treatment: *Vadim J. Jabotinski*[1]; Francis H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., Moscow, ID 83844-3026 USA

Electron beam processing offers great technical and economic capabilities for the high temperature treatment of hazardous materials. A uniquely high power efficiency of the electron beam sources of energy (80-90% of the power from the electric power line is converted to useable energy) suggests that this technique will be a major waste treatment technology in the not far future. This paper will consider advanced electron beam concepts for solid, liquid, and gaseous waste treatment such as stabilization of radioactive solid and liquid hazardous streams. Fundamental aspects and applications including an economic potential of the electron beam waste treatment will be discussed.

4:15 PM

Kinetics of Non-Isothermal Precipitation Process of Perovskite Phase in CaO-TiO_2-SiO_2-Al_2O_3-MgO System: *Yuhai Li*[1]; Taiping Lou[1]; Yuhu Xia[1]; Zhitong Sui[1]; [1]Northeastern University, Metallu. Dept., Shenyang, Liaoning 110006 PRC

Kinetics of non-isothermal precipitate process and crystal growth of perovskite ($CaO.TiO_2$) phase in CaO-TiO_2-SiO_2-Al_2O_3-MgO system were studied. The experimental results show that the relative volume fraction can be described by the equation given by K. Matusita et al. and the experiential expression of average crystal radius was obtained. The particle coarsening in non-isothermal process has important effects on the crystal growth of perovskite phase.

4:45 PM

Kinetics of Coarsening of Diffusion-Controlled Precipitate Phase in Non-Equilibrium Systems: *Taiping Lou*[1]; Yuhai Li[1]; Zhitong Sui[1]; [1]Northeastern University, Schl. of Matls. and Metallu., P.O. Box 119, Wenhua Rd., Shenyang, Liaoning 110006 China

The coarsening process of precipitates in non-equilibrium systems were studied, and then a physical model of coarsening was proposed. The coarsening process of CaO-TiO_2 in MgO-TiO_2-Al_2O_3-SiO_2-CaO-CaF system was investigated. It is shown that the coarsening process by the model predicting is agreed with the experimental results.

Honorary Symposium for Professor Oleg D. Sherby: Superplasticity A

Sponsored by: Structural Materials Division, Materials Processing and Manufacturing Division, Structural Materials Committee, Shaping and Forming Committee

Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; Chol K. Syn, Lawrence Livermore National Laboratory, Manufacturing & Materials Engineering Division, Livermore, CA 94550 USA

Wednesday PM — Room: Bayou E
March 15, 2000 — Location: Opryland Convention Center

Session Chair: Woo-Jin Kim, Hong-Ik University, Matls. Sci. and Metallu., Seoul, Korea

2:00 PM Invited

Mechanical Behavior of Materials: *Andrew Crowson*[1]; [1]U.S. Army Research Office, Phys. Sci. Direct., P.O. Box 12211, Research Triangle Park, NC 27709 USA

The Army continues to develop new materials that can extend systems capabilities beyond the physical limits of conventional materials. A major focus at the Army Research Office (ARO) in achieving such capabilities is on understanding the fundamental relationships between the structure of materials and their mechanical properties as influenced by composition, processing, environment, stress state, and plasticity, and toughening mechanisms for preventing or retarding fracture; especially at large strains (to 1000%) and high strain rates (to 10^6). The emphasis is on developing new knowledge of the fundamental deformation processes in materials including load transfer, fatigue, creep, transformation toughening, superplasticity, and shear localization. In addition, new processing approaches and/or procedures, inclusive of biomimetics and hierarchical materials, are also being investigated to optimize and improve the mechanical behavior and reliability of materials. This paper will describe programs in this area with special reference to contributions that have been made by Professor Oleg D. Sherby under ARO support.

2:20 PM Invited

Superplasticity in the Iron Aluminide $Fe_3Al(Cr)$: *Gorge Frommeyer*[1]; C. Derder[1]; J. A. Jimenez[2]; [1]Max-Planck Institut fur Eisenforschung GmbH, Max-Planck-Str. 1, Dusseldorf D-40237 Germany; [2]Centro National de Investigaciones Metalurgicas, Madrid, Spain

Superplasticity in an Fe_3Al based intermetallic alloy with 3 at. percent chromium in solid solution have been investigated in the strain-rate range from 10^{-5} to 10^{-2} s^{-1} at test temperatures between 700 and 900°C. The overall composition of the iron aluminide was $Fe_{69}Al_{28}Cr_3$ with small amounts of titanium and carbon. In the thermomechanically processed condition the material possessed a coarse microstructure with an average grain size of 55 ± 10 microns. Superplasticity occurred at strain-rate exponents of $0.33 \leq m \leq 0.42$ and at strain-rates of the order of 10^{-4} s^{-1}. Maximum elongations to failure of 300% and more were achieved. From thermal activation analysis of superplastic flow an activation energy of 185 ± 10 $kJmol^{-1}$ was derived. This value is comparable with activation energies of superplastic flow in $Fe_3Al(Ti)$ alloys. However, in unalloyed Fe_3Al the activation energy is higher of about 240 $kJmol^{-1}$. Optical microscopy and TEM studies showed subgrain formation. Subgrains of the order of 0,3 to 0,4 microns in size revealed, and grain refinement to about 30 microns occurred. Superplasticity deformation in this iron aluminide is characterized by viscous dislocation glide, controlled by solute drag, in the ordered B2 lattice. After a certain amount of superplastic deformation the samples showed subgrain formation in the strained gauge sections. From this it is concluded, that dynamic recrystallization has an important contribution to the deformation mechanism of superplastic flow in this material.

2:40 PM Invited

Tensile Ductility Behavior of Superplastic Ceramics: *Woo-Jin Kim*[1]; [1]Hong-Ik University, Metallu. and Matl. Sci., 72-1 Sangsu-dong Mapo-ku, Seoul 121-791 Korea

The tensile elongation of fine-grained ceramics is shown to increase as a strong function of decreasing flow stress, even though the values of strain-rate-sensitivity exponent remains high. This trend in tensile elongation is explained based on a "fracture mechanics model". The tensile ductility dependence on grain size was also investigated for many fine-grained ceramics either under a constant strain rate or stress conditions, and could be well explained quantitatively by the fracture mechanics model. The difference in the tensile ductility behavior of superplastic ceramics and metallic alloys can be related to their different failure mechanisms. The superplastic ceramics deform without necking and fail by intergranular cracks growing perpendicular to the applied tensile axis. In contrast, superplastic metallic alloys commonly fail by intergranular and trangranular mechanisms with associated void formation in the neck regions.

3:00 PM Invited

Effects of Microstructural Scale on High Temperature Plasticity of Dispersion Strengthened Materials and Composites: *Rajiv S. Mishra*[1]; [1]University of Missouri-Rolla, Dept. of Metallu. Eng., 218 McNutt Hall, Rolla, MO 65401 USA

Three microstructural features are important for materials with second phase particles: matrix grain size, second phase particle size and interparticle spacing. These microstructural features influence the creep deformation and superplasticity. The effects of microstructural scale on various creep and superplasticity mechanisms are discussed with the help of microstructure-based deformation mechanism maps. Some possibilities of transition in deformation mechanisms with microstructural scale are presented. These transitions are particularly important for dispersion strengthened materials and composites with ultrafine microstructure.

3:20 PM Break

3:30 PM Invited

On the Creep and Superplastic Behavior of the ODS Nickel-Based Superalloy PM 3030: *Martin C. Heilmaier*[1]; Michel Nganbe[1]; Frank E.H. Müller[2]; [1]IFW Dresden, P.O. Box 270016, Dresden D-01171 Germany; [2]Plansee GmbH, Siebenbürgerstrasse 23, Lechbruck D-86983 Germany

Since the review of Lin and Sherby in 1980 [1] the creep properties novel oxide dispersion strengthened (ODS) superalloys produced by mechanical alloying (MA) techniques have recently regained renewed attention from the scientific community and industry. Our study is focused on PM 3030, a nickel-based superalloy recently developed by Plansee GmbH Lechbruck (Germany). Due to its high content of Al, PM 3030 is strengthened by a high volume fraction of coarse ordered γ' particles of cuboidal shape. Additionally, the production by means of mechanical alloying enables the incorporation of low volume fractions of incoherent spherical Yttria dispersoids. While the latter are of the order of 20 nm in diameter, the γ' particles lie in the range of 0.5 to 1 μm. The high temperature deformation properties of three differently processed semifinished products have been investigated using compressive and tensile tests under constant true strain rate: a) an as-hipped fine-grained material with a grain size of about 1 μm (heat 1), b) a subsequently annealed variant to produce coarser equiaxed grains of around 15 μm in diameter (heat 2) and a hot extruded and fully recrystallized material with coarse elongated grains (heat 3). The latter possesses a grain aspect ratio of roughly 100. Similar to the early work by Gregory, Gibeling and Nix [2] on MA 6000, heat 1 exhibits a potential for superplastic flow at high strain rates accompanied by low necessary flow stresses. Obviously, grain coarsening is effectively suppressed by a "duplex grain structure" consisting of γ and γ' phase. The larger grains in heat 2 lead to a dramatic increase in creep strength and in stress exponent. While heat 3 shows further superiority in creep resistance to heat 2 at temperatures above 800 °C, the reverse behavior is observed below that limit. We will discuss the observed differences in the high temperature deformation behavior in terms of actual

microstructurally based creep concepts. [1] J. Lin, O.D. Sherby, Res Mech. 2 (1980), 251. [2] J.K. Gregory, J.C. Gibeling, W.D. Nix, Metall. Trans. 16A (1985), 777.

3:50 PM Invited
Micromechanics-Based Constitutive Relations of Superplastic Materials: *Namas Chandra*[1]; [1]Florida State University, Mech. Eng., 2525 Pottsdamner Rd., Tallahasse, FL 32310 USA

Abstract text not available

4:10 PM Invited
Hyperplasticity: Enabling Complex Sheet Components with Fast Deformation: *Glenn S. Daehn*[1]; Vincent J. Vohnout[1]; Hemant Panshikar[1]; Subrangshu Datta[1]; Keith Crane[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Oleg Sherby has never been afraid of taking original even anti-conventional approaches to existing problems or issues, and he shows great respect for solutions pioneered in the "forgotten past". These are a couple of the most important lessons the presenting author learned from Oleg. These lessons were put to use in developing a research program in high velocity sheet metal forming. Here we will show that high velocity forming, an extension of "forgotten" explosive forming, can have great relevance in treating contemporary issues in metal forming. With high velocity forming, formability is improved, wrinkling is suppressed and precise, complex parts can be formed. After introducing the fundamentals, examples and strategies for using these techniques to enable the fabrication of components of current interest will be presented.

4:30 PM Invited
Superplasticity at Ultrahigh Strain Rates and Elevated Temperatures—Can it Occur?: *Robert D. Caligiuri*[1]; Lawrence E. Eiselstein[1]; Charles G. Schmidt[2]; [1]Exponent Failure Analysis Associates, P.O. Box 3015, 149 Commonwealth Dr., Menlo Park, CA 94025 USA; [2]Hewlett Packard Corporation

A preliminary model for the occurrence of superplastic phenomena at ultrahigh strain rates and elevated temperatures is presented. This model is based on the increase in transition strain rate from superplastic to non-superplastic behavior with temperature. At 1200°C, the transition strain rate is predicted to be about 10^2 s^{-1} for a material with a grain size of 1 mm. There should be insufficient time at this strain rate for significant grain growth to occur, assuming the material reaches temperature rapidly, as can happen under explosive loading conditions. Results on experiments on ARMCO iron and fine grained ultrahigh carbon steel miniature tensile specimens using a split Hopkinson Bar are in qualitative agreement with the predictions of the model.

4:50 PM Invited
Creep of (La,Sr) MnO_3; A Fuel Cell Cathode: *Jeff B. Wolfenstine*[1]; Russ Cook[2]; Ken Goretta[2]; Jules Routbort[2]; [1]U. S. Army Research Laboratory, AMSRL-SE-DC, 2800 Powder Mill Rd., Adelphi, MD 20783-1197 USA; [2]Argonne National Laboratory, Matls. Sci. Div., Argonne, IL 60439-4838 USA

The deformation of fine-grained (<10 mm) La,Sr) MnO_3 with relative densities between 85-90% was investigated over the temperature range 1150-1300°C as a function of applied stress, oxygen activity and Sr content. The fine grain size, brief creep transients, stress exponent close to unity, absence of deformation induced dislocations and lack of grain shape change suggested that the deformation was controlled by grain boundary sliding accommodated by diffusion. A comparison of the activation energy for creep with existing diffusion and creep data for perovskite oxides revealed that grain boundary sliding was accommodated by lattice diffusion. The effect of oxygen activity on the deformation rate suggested that the rate-controlling defect is cation vacancies at low oxygen partial pressures and oxygen vacancies at high oxygen partial pressures. These results are in excellent agreement with a point defect model that incorporates cation non-stoichiometry.

Liquid Metal Atomization: Fundamentals and Practice: Other Methods

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee
Program Organizers: Khershed P. Cooper, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5343 USA; Frank Biancaniello, NIST, Gaithersburg, MD 20899-8556 USA; Stephen D. Ridder, NIST, Gaithersburg, MD 20899-8556 USA

Wednesday PM Room: Bayou B
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Frank Biancaniello, National Institute of Standards & Technology, Gaithersburg, MD 20899-8556 USA; James C. Foley, Ames Laboratory, Ames, IA 50011-3020 USA

2:00 PM Invited
Atomization of Melts Using the Impulse Atomization Technique: *Hani Henein*[1]; [1]University of Alberta, Dept. of Chem. and Matls. Eng., Adv. Matls. and Process. Lab., 536 Chem.-Matls. Eng. Bldg., Edmonton, AB T6G2G6 Canada

The Impulse Atomization Process (IAP) is a single fluid atomization technique that is capable of producing droplets of a desired size and a narrow size distribution with a predictable cooling rate. The process has been successfully employed to produce a wide range of metal droplets including Pb-Sn alloys, aluminum alloys, copper alloys, low carbon steel and tool steel. Atomization characteristics determined from load cell measurements, video imaging and particle size analysis will be discussed as a function of process characteristics. It is shown that atomization occurs by Rayleigh instability and that only primary atomization of the stream is in effect. The rate of cooling of a moving molten droplet has been modeled and experimentally validated using this atomization technique. It will be shown that in atomization, the droplet size is an important variable contributing to the high magnitude of the heat transfer coefficient which was determined to be around 2000 to 5000 watts/Km^2. By contrast, the droplet Nusselt number ranged from only almost 2 to 10 indicating that conduction of heat from the droplet to the gas is an important mechanism by which the droplet looses heat. These atomization and heat flow characteristics clearly demonstrate a number of unique features of this technique as well as its flexibility to meet different processing requirements for production and research.

2:30 PM Invited
Centrifugal Atomisation of Alloys: *Panayiotis Tsakiropoulos*[1]; Huiping Li[1]; [1]University of Surrey, Schl. of Mech. and Matls. Eng., Guildford, Surrey GU25XH England

The centrifugal atomisation (CA) process is used for the production of powders of a variety of alloys, which include Al and Ti based alloys, superalloys and steels. The most widely used variant of CA is the rotating electrode process (REP). A basic requirement of REP is that the alloy to be atomised is available in bar form. This restriction means that REP is often not suitable for the production of powders of developmental alloys, which are not available in bar form. The CA variant, which is based on the rotating disk principle, is suitable for the production of powders of ferrous and non-ferrous alloys including reactive and refractory metal alloys, and developmental alloys. Near net shape processing via spray casting on a cylindrical substrate is also possible. The paper will describe the basic phenomena of interaction of the melt with the rotating disk and the break up/atomisation of the melt at the edge of the rotating disk. Experimental results for ferrous and non-ferrous alloys will be compared with the predictions of a recently developed model which studies the formation of a thin film of

melt on the rotating disk and the break up of the melt at the edge of the rotating disk for different atomisation regimes.

2:55 PM Invited

Recent Advances in Highly Controlled Molten Metal Droplet Formation from Capillary Stream Break-up: *Melissa E. Orme*[1]; [1]University of California, Mech. and Aero. Eng., Irvine, CA 92697-3975 USA

The science of capillary stream break-up into droplets has recently attracted significant industrial and academic interest for applications requiring uniform metal particle production. Exploitation of the high droplet production rates intrinsic to the process and the unparalleled uniformity of droplet sizes and speeds attained with proper applied forcing to the capillary stream make many new applications related to the net-form manufacture of structural components and electronic packages feasible. Recent research results on the uniform production of aluminum and aluminum alloy droplet streams for the application of net-form manufacturing will be presented. Issues affecting the droplet stream stability such as oxidation, corrosion by molten aluminum and other chemical reactions at elevated temperatures are presented. Additionally, new research on the uniform production of solder droplets for the application to electronic package manufacturing is also presented. Issues common to all molten metals such as the basic phenomenon of capillary stream formation and break-up into droplets, acoustic excitation issues relevant to apparatus design, electrostatic charging and deflection for high speed "printing" or particle "sorting" applications, and novel forcing disturbances for more flexible droplet production are discussed.

3:20 PM Invited

The Effect of Oxygen Concentration on the Break-Up Behavior of Laminar, Liquid Metal Jets: *Pyongwon Yim*[1]; *Jung-Hoon Chun*[2]; Nannaji Saka[2]; Juan Carlos Rocha[3]; [1]Samsung Information Systems America, Hdd R&D Ctr., 75 W. Plumeria Dr., San Jose, CA 95134 USA; [2]Massachusetts Institute of Technology, Lab. for Manu. and Product., 77 Massachusetts Ave., Room 35-233, Cambridge, MA 02139 USA; [3]Applied Materials, Santa Clara, CA USA

It is well known that as a laminar, liquid jet issues from a small orifice, the surface tension force of the liquid mediates the cylindrical jet to break up into a train of spherical droplets. In 1878 Lord Rayleigh advanced a linear analysis of the break-up phenomenon of free laminar jets. He further demonstrated that uniform droplets can be produced when the jet is subjected to a periodic oscillation of wavelength greater than the circumference of the jet. In recent decades, wavelengths of three-and-a-half to seven times the jet diameter have been used to produce uniform droplets of various liquids such as water and ink. The break-up length of a liquid jet depends on the jet diameter, the physical properties of the liquid, and the amplitude and frequency of oscillation applied to the jet. Prediction of the break-up length of a reactive molten metal jet, however, is more difficult when it is sprayed into an oxygen-laden atmosphere. Metal-oxygen gases may form oxides or other tenacious films on the jet surface. In some cases such surface films stabilize the jet, which results in the production of fibers instead of droplets. Thus, determination of the critical concentration of oxygen below which the molten metal jet can be broken into a train of uniform droplets is of paramount importance to the successful applications of the break-up phenomenon of molten metal jets. This paper reviews the break-up behavior of laminar, liquid jets and investigates the role of oxidation in the break-up behavior of liquid tin jets to establish the maximum concentration of oxygen below which jet instability can be promoted by experimentally varying the oxygen concentration from 5 to 200,000 ppm. It was observed that a 100 μm diameter tin jet does not break into droplets when the oxygen concentration levels are above 1600-1800 ppm.

3:45 PM Break

4:00 PM Invited

Rapidly Spinning Cup Atomization: Correlation of Particle Characteristics with the Melt/Quench Liquid Impact: *Stephen J. Mashl*[1]; Khershed P. Cooper[2]; [1]Bodycote IMT Inc., Rsch. and Dev., 155 River St., Andover, MA 01810 USA; [2]Naval Research Laboratory, Matls. Sci. and Tech., 4555 Overlook Ave. S.W., Code 6324, Washington, DC 20375 USA

The rapidly spinning cup (RSC) atomization process has shown promise for the production of fine spherical metal powders. Unfortunately, atomization conditions which promote fine spheres typically also produce other, less desirable particle morphologies. A recent study has employed high speed photography to identify RSC atomization mechanisms over a range of conditions. This photographic examination demonstrated that the character of the collision between the melt jet and the quench liquid could vary significantly with changes in both melt and quench liquid velocity. In this study, information gathered using high speed photography is combined with a review of particle morphology and particle size distribution of the resultant powders. Particle characteristics are compared to the nature of the melt/quench collision and the probable evolution of various particle shapes is discussed.

4:25 PM Invited

A Study of the Effect of Liquid Metal Atomization Media on Particle Size and Morphology: *Khershed P. Cooper*[1]; Cynthia M. Chambers[1]; [1]Naval Research Laboratory, Code 6324, 4555 Overlook Ave. S.W., Washington, DC 20375-5343 USA

The rapidly spinning cup is a convenient tool to investigate the effect of atomizing liquids on the characteristics of atomized metal powder. The nature of the spinning cup apparatus is such that the atomizing liquid media can be easily changed. Oils and other hydrocarbons of varying physical properties were selected as atomizing fluids. While densities and most other properties are similar for these liquids, viscosities vary by a couple of orders of magnitude. Viscosity plays a major role in fostering shear forces which are responsible for melt break-up. Experiments with Al-Cu eutectic alloy demonstrated a significant effect of atomizing media on mean particle size and particle morphology. There was no correlation between mean size and viscosity suggesting that other properties such as heat capacity may be playing a role. Similar experiments were performed with Bi-Mn eutectic alloy. Bi-Mn is denser than Al-Cu and expands upon solidification. The results of these studies will be discussed in terms of liquid metal break-up mechanisms and solidification behavior.

4:50 PM Invited

Using Multiple Regression Analysis to Gain Insight into the Physical Mechanisms in Spinning Cup Atomization: *Charles I. Whitman*[1]; Khershed P. Cooper[2]; [1]Industrial Problem Solving, 910 Flintlock Rd., Southport, CT 06490 USA; [2]Naval Research Laboratory, Code 6324, 4555 Overlook Ave. S.W., Washington, DC 20375-5343 USA

By using Statistical Modeling one can often gain insight into the fundamental physical processes going on in a process, and suggest process improvements. Here, some 54 experiments with molten tin performed at the Naval Research Laboratory were analyzed by Multiple Regression to develop a mathematical model containing some nine terms. The model, which contained an unusual three factor interaction, was used to explain this interaction as an effect resulting from the use of a smaller orifice in some of the experiments. This model also explained the presence of small amounts of "encapsulated" particles in the powder, and led to a design change in a larger atomizer to control such particles. Further, also explained was the role of turbulence in coarsening the average particle size of powder produced in the process. The effect of changing the melt to eutectic Al-Cu was also explored.

5:15 PM Invited

Modeling and Experiments on Using a New Pulsed Power Technique to Produce Fine Metallic Powders: *F. Douglas Witherspoon*[1]; Russell Kincaid[1]; Arul Mozhi[2]; [1]UTRON Inc., 8506 Wellington Rd., Ste. 200, Manassas, VA 20109 USA; [2]National Materials Advisory Board, Nat. Acad. of Sci., 2001 Wisconsin Ave. N.W., Washington, DC 20007 USA

This paper presents results of an ongoing Small Business Innovation Research project. This project is developing a new approach to producing inert gas atomized metal powders of size 10's of nm to 20 μm. This technique replaces the gas stream used in conventional atomization with a pulsed plasma jet to generate a much higher (three orders of magnitude) momentum flux atomizing medium. This new technique has the potential to decrease the cost of production. In

Phase I, fine spherical copper powders in the size range 0.2 to 8.0 μm, and steel powders of size 0.5 to 6.0 μm, were successfully produced. Calculations predict that experimental conditions achievable in Phase II will result in metal powders in the 10's of nm (theoretically down to 3 nm). In the ongoing Phase II, UTRON is building an engineering prototype to demonstrate the production of 2 to 3 kg batches of fine iron-based powders for characterization. An induction furnace is used to generate the melt stream which is then atomized in a confined geometry nozzle region. The atomized powders are collected in an inert atmosphere. Experimental and modeling results on production of fine iron-based powders from this ongoing project will be presented.

Magnesium Technology 2000: Physical and Mechanical Properties

Sponsored by: Light Metals Division, Reactive Metals Committee, International Magnesium Association
Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA

Wednesday PM — Room: Bayou C
March 15, 2000 — Location: Opryland Convention Center

Session Chair: Mihriban O. Pekguleryuz, Noranda Technology Center, Pointe Claire (Montreal), Quebec, Canada

2:00 PM
Wear Resistance Property and Microstructure of Magnesium AZ91 Composite: *Jamaliah Idris*[1]; J. C. Tan[1]; [1]Universiti Teknologi Malasia, Faculty of Mech. Eng., Johor, Skudaim 81310 Malaysia

The wear resistance of magnesium AZ91 composite produced by powder metallurgy technique and reinforced with 0,5,10,15, and 20 vol.% SiC were being investigated. Pin-on-disk dry sliding wear tests were carried out to study the wear performance and wear mechanisms. The magnesium matrix composites were used as pins while the counterface consisted of mild steel disks. After the wear tests, worn surfaces of pins and the wear debris were investigated by using scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX). The wear resistance performance of magnesium AZ91 composites was found to increase with increasing sliding distance and the wear rate was greatly reduced after the wear-in phenomenon. During the wear-in stage, abrasion was found to be predominant. Oxidative wear took place when the applied normal load exceeded critical load limit.

2:25 PM
Elements of the Fatigue Process in Magnesium Die Casting Alloys: *Terje Kr. Aune*[1]; Darryl L. Albright[1]; Oddvin Orjasaeter[2]; Odd K. Nerdahl[2]; [1]Norsk Hydro, Rsch. Ctr., Porsgrunn N-3901 Norway; [2]SINTEF

Magnesium alloy die casting provide opportunities for achieving high performance, low weight, cost efficient, and fully recyclable solutions to complex engineering design challenges. Today, alloys based upon the addition of aluminum, manganese and zinc to the base metal form the basis for structural components designed to successfully withstand the forces of fatigue. Although as cast surfaces represent the most common condition for service, the roles of vibratory polishing and mirror polishing in performance were also investigated. Additionally, the variables of mean stress and frequency were included in the test program. Variations in alloy chemistry and the resulting phase distribution led to relatively small differences in the measured fatigue properties. While vibratory polishing also led to only minor property differences, the incorporation of mirror polishing had a significant effect on the fatigue performance. The result led to the establishment of design curves, along with a proposed model for fatigue damage accumulation.

2:50 PM
Fracture Toughness of Magnesium Alloy AM60B: *S. K. Iskander*[1]; R. K. Nanstad[1]; S. Viswanathan[1]; R. L. Swain[1]; J. F. Wallace[2]; [1]Oak Ridge National Laboratory, Bldg. 4508, MS 6083, Rm. 135, Oak Ridge, TN 37831-6083 USA; [2]Case Western Reserve University, Cleveland, OH USA

The fracture toughness of Magnesium Alloy AM60B was measured, using of 5-mm thick compact tension specimens. Characterization of the fracture toughness behavior included tests in two different orientations and two temperatures. Results indicated stable tearing behavior, and testing was discontinued when crack extension exceeded the clip gage capacity. Values of the Tearing Modulus and a "K form JQ" were measured. These two parameters did not reveal any effect of specimen orientation on fracture toughness. Subsize Charpy specimens were prepared in the same two orientations as those in which compact specimens were tested, as well as in other orientations in which compact tension specimens could not be prepared. Charpy impact testing also confirmed that the fracture toughness is generally independent of orientation effects. Force vs. displacement traces from Charpy testing also confirmed the stable tearing behavior observed in fracture toughness testing. Fracture surfaces on some of the tested compact tension specimens showed some porosity, which did not influence the fracture toughness values measured because of the direction of crack-propagation is not influenced by the orientation of the porosity, but could have influenced the fracture toughness in other orientations in which the flaws could have negative effects. Scanning Electron Microscopy of fracture surfaces, Metallography of surfaces parallel to the different orientations, and macro graphs of fracture surfaces including one with porosity and/or lamination were also performed to document the results.

3:15 PM
Deformation-Induced Texture as an Alloy/Process Optimization Tool: *S. R. Agnew*[1]; [1]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Understanding which deformation mechanisms are active is often a necessary component of high performance alloy development, particularly for non-cubic and/or ordered intermetallic alloys where strength and ductility are strong functions of the operative deformation modes. Deformation-induced texture data coupled with appropriate polycrystal plasticity simulation techniques can provide rapid insight into these issues. The preferred orientation (or texture) which results from deformation is a fingerprint of the imposed deformation geometry and the active deformation mechanisms that sustained the plasticity. Although the ductility of traditional wrought magnesium alloys is moderate, their low temperature formability is quite limited. This fact makes magnesium an ideal candidate to demonstrate the advantage of a texture-based approach because the limited formability is largely due to the limited number of active deformation modes (slip, twinning, etc.). Hexagonal close packed solid solution alloys containing lithium have been particularly potent for demonstrating the potential of this approach. The addition of lithium is known to enhance the ductility of magnesium and texture analysis has confirmed that lithium promotes dislocation slip on the prism planes as well as the basal plane.

3:40 PM Break

3:50 PM
Superplasticity of Magnesium Alloys: U. Draugelates[1]; A. Schram[1]; *C. C. Kedenburg*[1]; [1]Institute fur Schwelstechnik und Trennende Fertigunsvefahren, Tech. Universitat Clausthal, Agricolastrasse 2, Clausthal D-38678 Germany

Through the use of the superplastic behaviour of magnesium materials the productivity of the processing of semifinished products to complex formed finished products can be drastically raised compared to conventional conversion procedures. Especially the production of highly terminal measurements of components by the means of the superplasticity forming of metals and their alloys is an inexpensive production procedure compared to the alternative extensive remove material process or join process. The increasing demand of cheap

producable products capable of withstanding high stress and due to the restraint to light weight products and the wanted material and energy savings especially in the fields aeronautics and astronautics, the high speed railway systems, automobile and equipment construction, but also news and data processing technology has brought about the growing interest in superplastic forming. One reason for this procedure is the special suitability of manufacturing highly thin-walled structural components for the light weight design. The presentation emphasizes the following subjects: In the introductory part of the presentation presuppositions for the superplastic behavior of magnesium-alloys are discussed. Methods to quantify the superplastic behavior and the importance of the m-values derived thereof are explained in the following. Since a major precondition for superplastic forming is a very fine grain structure, various possibilities of grain-fining-procedures of magnesium-alloys are described with the help of parameter-lists and pictures of grain-structures. Finally, the results of the investigations are graphically displayed and an outlook to closer aims of the research program is given.

4:15 PM

Fatigue Behaviour of AZ91D Magnesium Alloy and its Composite Reinforced with SiC: *A. Bag*[1]; W. Zhou[1]; D. Taplin[1]; E. S. Dwarakadasa[2]; [1]Nanyang Technological University, Schl. of Mech. & Product. Eng., Nanyang Ave. 639798 Singapore; [2]Indian Institute of Science, Dept. of Metallu., Bangalore 560012 India

Increasing demand for lighter components and light weight design has led to magnesium alloys being considered as possible engineering materials in the last few years. However, low fatigue strength has been an important factor in the limited use of magnesium alloys in more highly stressed designs. In particular magnesium matrix composites are attracting a lot of attention because the addition of a reinforcing phase, such as ceramic particles or fibres may produce a remarkable improvement in the property profile. Reinforcement with ceramic particles can lead to an increase in strength, Young's modulus and hardness, particularly at room temperature whereas the coefficient of thermal expansion is reduced. However, addition of SiC particles to AZ91D matrix may result in lower fatigue resistance. Therefore the present investigation examines the fatigue properties of AZ91-10%SiC composite in different heated treated conditions and compares them with the monolithic AZ91 alloy. Fatigue crack growth study was conducted by using half-compact tension specimens with the load ratio of R= 0.1. Crack growth tests supplemented by optical and scanning electron fractography have been used to assess the role of magnesium matrix, which has HCP crystal structure and ceramic particle interactions on the crack initiation, propagation and near threshold behaviour and finally to compared with the monolithic magnesium alloy. The experimental results show that appropriate heat treatment can improve the crack growth behaviour of AZ91 alloy and its composite.

Materials Issues in Microelectronics: Optical, Electrical, and Thermal: Low Alpha Pb and Applications

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Yellapu V. Murty, Carpenter Technology Corporation, Research and Development, Reading, PA 19612-4662 USA; Prasad Godavarti, Motorola, Austin, TX USA; Sung-Ho Jin, Bell Laboratories, Lucent Technologies, Murray Hill, NJ 07974 USA; Sung Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598 USA; John Macwilliams, US Competitors, LLC., Newark, DE 19711 USA; Mark McCormack, Fujitsu Computer Packing Technologies, San Jose, CA 95134 USA; Martin Weiser, AlliedSignal Electronic Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Wednesday PM — Room: Lincoln C
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: Sung K. Kang, IBM, Yorktown Heights, NY 10598 USA; Prasad Godavarti, Motorola, Austin, TX 78721 USA

2:00 PM Opening Comments

2:05 PM

Low Alpha Solder Electrolyte Used for Flip Chip Bump Formation: *Masayoshi Kohinata*[1]; Kiyotaka Tsuji[2]; Keigo Obata[3]; [1]Mitsubishi Materials Corporation, 12-6, Technopark Sanda, Hyogo 669-1339 Japan; [2]Ishihara Chemical Company Limited, 5-26, Nishi-Yanagihara, Hyogo-Ku, Kobe 652-0806 Japan; [3]Daiwa Fine Chemicals Company Limited, 1-17, 2-Chome Shimosawa-Dori, Hyogo-Ku, Kobe 652-0047 Japan

We have developed a low alpha solder electrolyte used for flip chip bump formation. It is generally known that the alpha particle count from the deposit immediately after plating are low, but increase with time. Using our electrolyte, the alpha particle count from the deposit can be very low and stable. The characteristics of the bump plated for 63% tin and for 5% tin solder electrolytes are given, including the uniformity of the bump height and the bump compositions over the wafer. The starting materials and electrolyte are fully developed, so our electrolyte is already in mass production. We also describe an evaluation method for the electrolyte that controls parameters influencing bump characteristics.

2:30 PM

Low-Alpha Lead for Solder Bumping Production: *Mark W. Roberson*[1]; Phil A. Deane[1]; Salvatore Bonafede[1]; Alan Huffman[1]; Sundeep Nangalia[1]; [1]MCNC, Elect. Tech. Div., 3021 Cornwallis Rd., Research Triangle Park, NC 27709 USA

Soft-errors caused by lead in solder-bumping have been a concern for many years. The problem is of special concern for high-density interconnection applications requiring solder to be placed directly over active circuitry. In that situation, alpha particles emitted by radioactive lead cause soft-errors with no possibility of shielding circuitry. For optimal cost-effectiveness, though, not all solder bumped wafers require low-alpha lead. MCNC has developed a solder bumping facility with both a research branch at MCNC and a full-scale production facility at its spin-off, Unitive Electronics Inc. We present results here of our work in incorporating low-alpha lead as part of our solder bumping process. We describe the amount of cross-contamination measured when alternating plating baths of regular lead and low-alpha

lead. We also present geometric correction factors for solder bumps measured with alpha detection counters.

2:55 PM

A Low Alpha Eutectic Type 6 Solder Paste for Ultra Fine Pitch Chip Bumping: *Mike Grosse*[1]; Jim Grundy[1]; Tom Herrington[1]; [1]Johnson Matthey Electronics, 10080 Willow Creek Rd., San Diego, CA 92131 USA

The rapid growth in chip level interconnection density continues to present new challenges to the suppliers of semiconductor packaging materials. Further increases in device speed and functionality with corresponding decreases in device geometry has placed tighter constraints on packaging materials and their related processes; solder paste being one of those critical materials. With the advent of bump arrays numbering in the thousands of bumps/print at ever decreasing pitch dimensions, higher and higher first pass yields will be required of the solder paste and process used for flip chip bumping. In order to achieve the higher print resolutions demanded by the flip chip process, it was necessary to optimize both the powder process and flux system. Process optimization revealed that by instituting additional controls, a type 6 powder as specified by IPC in standard J-STD-006 could be produced with reasonable final yields. Various designed experiments were conducted to identify the critical process variables and conditions that influence particle shape, surface morphology, particle size distribution and oxide content. Furthermore, as powder particle size is reduced to enhance print definition, the surface area of the powder and consequently the amount of surface oxide increases and requires careful monitoring to insure lot to lot consistency. The reduction in on-chip feature size has led to a closer proximity of logic elements and the solder bump interconnects. Industry concerns have grown over the phenomena of radioactive decay induced soft errors generated by trace isotope impurities found in lead containing solders. In order to address this reliability concern, it was necessary to obtain a lead supply with an extremely low concentration level of the isotope 210Pb. Post powder processing alpha emission flux testing is performed to determine the alpha emission rate and to gain insight into the decay characteristics of the material.

3:20 PM Break

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Phase Equilibria of Ag-Sn-Cu Ternary System: *Yee-wen Yen*[1]; Sinn-wen Chen[1]; [1]National Tsing-Hua University, Dept. of Chem. Eng., #101 Sec. 2 Kuang-Fuh Rd., Hsin-Chu, Taiwan 300

Ag-Sn alloys are a prominent group of lead-free solders. Cu is the most popular substrate, and the phase equilibria of Ag-Sn-Cu ternary system is thus of importance in electronic industry. Various Ag-Sn-Cu alloys are examined and the isothermal sections at 240°C and 450°C have been experimentally determined. At 240°C, the e1-Cu3Sn phase is in equilibrium with Ag, z-Ag4Sn, and e2-Ag3Sn phases. The existence of d-Cu4Sn phase at 450°C does not change much of its phase equilibria, and e1 phase is still in equilibrium with Ag, z and e2 phases at 450°C, which indicates that the e1 phase is a very stable phase. Based on two experimentally determined isothermal sections and the knowledge of the thermodynamic models of its three binary constituent systems, thermodynamic models of the Ag-Sn-Cu have been assessed in this study. The calculated results and the experimental determination of phase equilibria are in good agreement.

4:00 PM

3-D Simulations of Electromigration and Stress Evolution in Interconnections: *Zhineng Fan*[1]; Sven Rzepka[1]; Matt Korhonen[1]; Che-Yu Li[1]; [1]Cornell University, Dept. of Matls. Sci. and Eng., Bard Hall, Rm. 356, Ithaca, NY 14853 USA

As IC devices scale down, the cross section of interconnect lines on the chip are reduced and the current density increases. This puts electromigration in a particular position to interconnection reliability. While the electromigration and its related phenomena are rather complex, analytical simulation methods only give the results under very simplified conditions. In most cases, those models are one-dimensional and apply the hydrostatic stress as the chemical driving force. Numerical simulation, especially finite element analysis (FEA), is much more powerful to solve the complex problems. In this paper, we demonstrate a 3-D electromigration model that was developed based on a commercial FEA code. The model is capable to include Coble creep and Nabarro-Herring creep by applying normal stress as the chemical driving force. The interconnect segment consisting a polycrystalline section with bamboo structure connected to it at both sides was surveyed. The evolution of the atomic flow, normal stress and hydrostatic stress on the grain boundary are presented in this paper. It is shown that by using hydrostatic stress as the chemical driving force will overestimate the backflow of the stress-driven diffusion.

4:25 PM

Characterization of Electroplated Bismuth-Tin Alloys for Electrically Conducting Adhesive Materials: *Sung K. Kang*[1]; Stephen L. Buchwalter[1]; Cornelia K. Tsang[2]; [1]IBM, Rsch. Dept., T.J. Watson Rsch. Ctr., P.O. Box 218, Yorktown Heights, NY 10598 USA; [2]MIT, Matls. Sci. & Eng., Cambridge, MA 02139 USA

Electrically conducting adhesives are promising alternatives for lead (Pb)-containing solders in microelectronic applications. However, most common silver-filled epoxy materials have various limitations to meet the requirements of the solder joints yet. To overcome these limitations, several new formulations have been developed recently. Among them, a new high conductivity Pb-free conducting adhesive developed for low temperature applications has been previously reported. This conducting adhesive contains a conducing copper filler powder coated with a low melting point metal or alloy, such as Sn or BiSn. The low melting point layer serves as a joining material among the filler particles as well as to the substrate. In this paper, characterization of electroplated BiSn alloys on a Cu substrate is reported for their microstructure, electrical properties, oxidation behavior and others. The experimental results have provided a better understanding of the joining mechanism of the newly-developed Pb-free conductive adhesive.

4:50 PM

An Evaluation of Automated Ball Indentation for Measuring the Tensile Properties of Solder Alloys: *Michael L. Santella*[1]; Tsung-Yu Pan[2]; Frank W. Gayle[3]; [1]Oak Ridge National Laboratory, Met. & Cer. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6096 USA; [2]Ford Motor Company, Ford Rsch. Lab., 20000 Rotunda Dr., MD 3135, SRL, P.O. Box 2053, Dearborn, MI 48121-2053 USA; [3]National Institute of Standards and Technology, Rm. 223-B164, Gaithersburg, MD 20899 USA

The automated ball indentation (ABI) testing technique is not well developed for alloys other than steels, but its apparent ability to measure mechanical properties in small volumes of material makes it of interest for testing solder joints. The technique was evaluated by first preparing 1.5-mm-thick tensile specimens of bulk a Sn-3.5Ag wt% alloy and testing them at room temperature. ABI tests were then done on the shoulder regions of the tensile specimens. The overall agreement of the data sets was good, with the ABI data indicating a slightly lower yield strength and slightly higher flow stresses and work hardening rate compared to the tensile test data. Subsequently, small FR4 test boards were obtained that contained arrays of solder bumps made with the same alloy. The solder bumps had nominal dimensions of 2.5 mm long x 0.8 mm wide x 0.30-0.50 mm thick. ABI test results from the solder bumps agreed well with those from the tensile specimen shoulders. Details of the data comparisons will be presented and discussed in terms of relevant alloy characteristics and microstructures. The results indicate the reasonable possibility of making valid tensile property measurements on actual solder joints.

Materials Processing in the Computer Age III: Computer Aided Instruction

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizers: Vaughan Voller, University of Minnesota, Saint Anthony Falls Laboratory, Minneapolis, MN 55414-2196 USA; Hani Henein, University of Alberta, Edmonton, AB T6G 2G6 Canada; Sulekh Jain, Ge Aircraft Engineering, MId M-89, Cincinatti, OH 45215 USA

Wednesday PM Room: Lincoln A
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Brian G. Thomas, University of Illinois, Mech. Eng. Dept., Urbana, IL 61801 USA; Garry W. Warren, University of Alabama, Metallu. & Matls. Eng., Tuscaloosa, AL 35487 USA

2:00 PM
Introducing Materials to 6th, 7th and 8th Grade Science-A First Step: *Garry W. Warren*[1]; [1]University of Alabama, Metallu. & Matls. Eng., P.O. Box 870202, Tuscaloosa, AL 35487 USA

Computer Aided Instructional (CAI) software with a materials component have been developed for 6th to 8th grades science classes. The software is a result of the educational outreach activities of the NSF sponsored MRSEC at the University of Alabama's Center for Materials for Information Technology. A primary emphasis of the MRSEC's educational outreach activities involves collaboration with a very innovative 6th to 8th grade Integrated Science (IS) curriculum based in the Center for Communication and Educational Technology at the University of Alabama. Three of the authors are teachers in this program and participated in producing three software packages dealing with (1) Simple Machines (levers), (2) The Periodic Table and (3) The Scientific Method. In many cases these software packages probably represent the students first exposure to these topics, therefore a primary objective behind their development was to present the information in a fashion that would retain student interest, convey important information accurately, and above all complement and reinforce other classroom activities. Emphasis was placed on making the software truly interactive, requiring the student to participate by answering questions, selecting options and by incorporating moving objects, sounds and rewards for correct answers. Wherever possible interesting materials and applications thereof are incorporated. The financial support of the NSF Research Experiences for Teachers program is gratefully acknowledged.

2:20 PM
Use of the Web in Materials Teaching and Research: *Vaughan R. Voller*[1]; [1]University of Minnesota, Saint Anthony Falls Lab., Mississippi River at 3rd Ave. S.E., Minneapolis, MN 55414 USA

Among the recent innovations of the computer age the World Wide Web has and will continue to have a significant impact in teaching, education and research dissemination. This paper explores some examples of use of the Web and associated resources for teaching and research in the materials processing field. Specific examples will include: An outline of authoring tools for preparing engineering learning modules. HTML for interactive Web Quizzes Preparation of simple GIF animations. Running Interactive Web programs in scripting languages (e.g., JAVA-Script). For the most part the examples will be taken directly from the experiences of the author. The central purpose will be to show the accessibility and ease of use of available tools and demonstrate how a small amount of effort can lead to a significant value added to education and research endeavors. The key messages are (1)—taking note of other computer innovations of the last 50 years (e.g., programming)—to be fully effective individual educators and researchers need to take control of the tools and not rely on others to do it for them, and (2) the need to recognize, in an educational setting in particular, that although the infrastructure of a web page is important the critical component is the hard content. The work is supported by a TEL grant from Academic & Distributed Computing Services, University of Minnesota and by the NSF under Grant NSF/EEC-9711743.

2:40 PM
Computer Aided Instruction in Materials Science and Engineering Education: *Hani Henein*[1]; [1]University of Alberta, Adv. Matls. and Process. Lab., 536 Chem.-Matls. Eng. Bldg., Edmonton, Alberta T6G2G6 Canada

The easy accessibility and power of the PC as well as the education of our engineering students in the use of these tools, are compelling reasons for developing computer aided instructional (CAI) tools. There is a wide range of areas in the engineering curriculum where students must learn concepts involving the application of judgment for problem solving. Typically, throughout North America, the instructor of a course will provide theoretical guidelines to students on how to apply the required judgment. All too often, these points are lost on the student as it is deemed too complicated and too abstract. It is often too time consuming to get students to the point where they can practice this judgment. A number of computer aided packages have therefore been developed for use in several courses in the materials science and engineering curriculum that encompass both calculational and instructional capabilities. This talk will present an overview of some of these packages on the solution of a non-linear equation, the instruction of binary diffusion couples, an introduction of crystallography and an introduction to heat transfer. The context in which these CAI Tutorials were developed and are being used will be discussed. The use of the tutorials has important implications on the expectations from an engineering education of both instructor and student.

3:00 PM Break

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Open Discussion and Software Demonstration
The Role of Computer Based Training in Materials Processing

3:50 PM Closing Remarks

Process Synthesis and Modeling for the Production & Processing of Titanium & Its Alloys: Session IV

Sponsored by: Materials Processing and Manufacturing Division, Structural Materials Division, Titanium Committee, Shaping and Forming Committee
Program Organizers: James A. Hall, Oremet-Wah Chang, Albany, OR 97321 USA; F. H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026 USA; Isaac Weiss, Johnson Matthey, USA; Kuang Oscar Yu, RMI Corporation, R&D, Niles, OH 44446-0269 USA

Wednesday PM Room: Knoxville B
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Kuang Oscar Yu, RMI Corporation, R & D, Niles, OH 44446-0269 USA; James Hall, Oremet-Wah Chang, Albany, OR 97321 USA

2:00 PM
Solar: A Numerical Software to Optimize Vacuum Arc Remelting Operations: *A. Jardy D. Ablitzer*[1]; [1]Laboratoire de Science et Genie des Materiaux Metalliques, Ecole des Mines, Parc de Saurupt, Nancy, Cedex F-54042 France

A numerical model of the VAR process has been developed at the Ecole des Mines in Nancy and applied to simulate remelting operations realized in particular at Compagnie Europeenne du Zirconium CEZUS. The transient-state model named SOLAR (which stands for SOLidification during Arc Remelting) was continuously improved, by successively accounting for heat transfer, magnetohydrodynamic effects, and solute transfer (i.e. macrosegregation) during the melting, then while solidification of the final liquid pool proceeds. An important feature of the model is its ability to take into account any evolution of the melting rate and the stirring sequence during the melting, and to allow to understand the link between such operating parameters and the intensity of macrosegregation in the ingot. SOLAR has been used with some success to simulate pilot-plant and full-scale remeltings of steels, titanium and zirconium alloys. Recently, a significant improvement has allowed to simulate a triple VAR melt, using the computed composition of each ingot after cooling as the electrode composition for the next melting operation. SOLAR has enabled, for example, to determine the influence of the stirring sequence of preliminary melts on the intensity of macrosegregation in the final third ingot. Moreover, the potential interest of using a so-called "compensated" electrode, which means an initial electrode where the alloying element repartition varies along the length, can be quantitatively predicted before performing any actual triple vacuum arc remelting operation. Results of the model are presented and discussed.

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Ti-6V-Al under Electromagnetic Stirring and Melt Current Lorentz Force in Industrial Scale VAR: Parameter Sensitivity in Simulations: *L. A. Bertram*[1]; F. Spadafora[2]; S. N. Kempka[3]; R. L. Williamson[3]; R. S. Minisandram[4]; [1]Sandia National Laboratories, 7011 E. Ave., Livermore, CA 94551-0969 USA; [2]RMI Titanium Company; [3]Sandia National Laboratories, MS 0603, P.O. Box 5800, Alburquerque, NM 87185-0603 USA; [4]Allvac, Allegheny Teledyne Co., 2020 Ashcraft Ave., P.O. Box 5030, Monroe, NC 28111-5030 USA

In experiments conducted to establish, among other things, appropriate thermal and electrical boundary conditions for the numerical simulation of the VAR of 36" Ti-6-4 ingots, the following were observed: the ingot is over half liquid at power-off from full power (i.e., no "hot-topping" power cut-back was used, as it would have been for a production ingot); the "steering" of the VAR arc by the stirring magnetic fields is clearly evident; magnetic probe data show non-zero time averaged fields and non-uniform stirring. In seeking to simulate the experimentally established solid/liquid boundaries for the above, considerable care is necessary in choosing those parameters of the process which are not established by independent measurement. Among these are model turbulence intensity, coefficient of thermal expansion, fraction of arc current entering pool surface. If solenoidal windings develop full strength stirring magnetic induction Bs when the furnace arc is operating, the simulated flows can change from cases dominated by flow down the pool sidewall to cases dominated by flows down the ingot axis (as required by experiment) for quite plausible parameter values. These results suggest that a judicious mix of experiment and simulation is crucial to making realistic predictions about thermal conditions in such VAR cases. Axisymmetric '2-1/2D' simulations run to date using boundary conditions of the same type as reported for superalloy melting indicate large liquid volumes, but have not yet reproduced details of the pool shape.

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Comparison of Induction Slag Melted and Commercial Ti-6Al-4V Alloys: *Alan D. Hartman*[1]; Karol K. Schrems[2]; Gordon R. Holcomb[2]; Edward R. Argetsinger[1]; Jeffrey S. Hansen[1]; Jack I. Paige[1]; Paul C. Turner[1]; [1]Albany Research Center-DoE, Therm. Treat. Tech., 1450 Queen Ave. S.W., Albany, OR 97321 USA; [2]Albany Research Center-DoE, Matls. Conserv. Div., 1450 Queen Ave. SW, Albany, OR 97321 USA

The Albany Research Center of the U.S. Department of Energy has been investigating a means to form useful wrought products by direct and continuous casting of titanium bars using cold-wall induction melting rather than current batch practices such as vacuum arc remelting. Continuous ingots produced by cold-wall induction melting, utilizing a bottomless water-cooled copper crucible, without slag (CaF2) additions had minor defects in the surface such as "hot tears". Slag additions as low as 0.5 weight percent were used to improve the surface finish. Therefore, a slag melted experimental alloy ingot was compared to a commercial alloy Ti-6Al-4V ingot in the areas of physical, chemical, mechanical, and corrosion attributes to address the question, "Are any detrimental effects caused by slag addition?".

3:15 PM Break

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Study and Modeling of the Electron Beam Melting Process: *J. P. Bellot*[1]; H. Duval[1]; S. Besse[2]; [1]Laboratoire de Science et Genie des Materiaux Metalliques, Ecole des Mines, Parc de Saurupt, Nancy, Cedex 54042 France; [2]SNECMA, Matls. and Process. Dept., Centre de Villaroche, Moissy-Cramayel 77550 France

Since 1991 we have undertaken a complete study of the Electron Beam Melting process, with a particular emphasis on the cold hearth melting of the titanium alloys. Resulting from this research work, three comprehensive numerical models have been set up concerning the molecular gas dynamics of the vapor phase, the thermo-hydrodynamic and solute behavior of the alloy in the cold hearth and dissolution kinetics of the low density inclusions. The paper describes the main results obtained which are focused on: the volatilization losses of the alloying elements and the reduction of these losses by addition of an inert gas, the thermo-hydrodynamic behavior of the liquid pool taken into account the Marangoni and thermal natural convections, the history of the low density inclusions in the hearth (such as hard-alpha) in terms of trajectory and dissolution, and the effects of the beam scanning frequency on the transient thermal behavior of the surface of the liquid pool.

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Mathematical Modeling of the Electron Beam Cold Hearth Refining of Titanium Alloys: *Shesh K. Srivatsa*[1]; [1]GE Aircraft Engines, Matls. and Process. Eng. Dept., One Neumann Way, Mail Drop M87, Cincinnati, OH 45215-1988 USA

Electron Beam Cold Hearth Refining (EBCHR) is used to refine titanium alloys for critical aircraft engine rotating parts. In this paper, a computational model is presented for predicting the fluid flow, heat transfer, phase change, and the motion of inclusions in an EBM hearth. The model uses a control volume method for the discretization and solution of the momentum, continuity and energy equations to predict the flow and temperature fields within the hearth. The model accounts for all the important physical phenomena influencing the process, including temperature-dependent Marangoni and buoyancy effects and radiation heat transfer. A Lagrangian tracking approach is used to predict the inclusion trajectories and their change in size due to dissolution. The model has been applied to analyze an EBM hearth operating at different power conditions. The predicted surface temperatures and the pool shape compare well with the experimental measurements. The model can be used to develop insights into the underlying physical phenomena occurring in the EBCHR process, analyze the effect of process parameters on inclusion removal effectiveness, and ultimately achieve better design and control of the overall process.

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A Spectroscopic and Electrochemical Study of Titanium Electrorefining: *Luis Ortiz*[1]; Donald R. Sadoway[1]; [1]Massachusetts Institute of Technology, Dept. Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 8-109, Cambridge, MA 02139-4307 USA

Sputtering targets for the microelectronics industry are made by electrorefining metallurgical grade titanium in a molten chloride electrolyte. The feedstock is titanium sponge produced by the Kroll or Hunter process. The kinetics of metal deposition are being investigated by spectroscopic (visible and Raman) and electrochemical (voltammetric and impedance) techniques. Sponsorship of the research from The ALTA Group, JM Electronics, is gratefully acknowledged.

Rare Earths and Actinides; Science Technology and Applications IV: Rare Earths II: Processing

Sponsored by: Light Metals Division, Reactive Metals Committee

Program Organizers: Renato G. Bautista, University of Nevada-Reno, Department of Chemical and Metal Engineering, Reno, NV 89557-0136 USA; Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA

Wednesday PM — Room: Lincoln E
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: Danesh Chandra, University of Nevada, Metall. and Matls. Eng., Reno, NV 89557 USA; Seshadri Seetharaman, Royal Institute of Technology, Dept. of Metallu., Stockholm SE-100 44 Sweden

2:00 PM

The Preparation of Gd//5(Si//x/Ge//1-x/)//4/Magnetic Refrigerant Materials from Commercial Gadolinium Metal: *Karl A. Gschneidner*[1]; Alexandria O. Pecharsky[1]; Vitalij K. Pecharsky[1]; [1]Iowa State University, Ames Lab., 255 Spedding, Ames, IA 50011-3020 USA

About three years ago we discovered the giant magnetocaloric effect materials Gd//5/(Si//x/Ge//1-x/)//4/, where x≤0.5. These materials have great promise as useful magnetic refrigerant alloys for a wide range of applications-building air conditioning, supermarket chillers, frozen food processing plants, automotive climate control, etc. To date most of the samples have been prepared by arc-melting using high purity (99.8 at.%) Ames Laboratory Gd metal. The sample sizes are of the order of 50g or less. If commercialization is to be realized, one will need to use inexpensive commercial grade Gd (95 to 98 at.% pure with major impurities of O and C) and a melting process which can be used to prepare large quantities (1kg or larger) of the Gd//5/(Si//x/Ge//1-x/)//4/alloys. We found that carbon impurities are detrimental by preventing the formation of the monoclinic phase and thus destroy the giant magnetocaloric effect in Gd//5/(Si//x/Ge//1-x/)//4/for x≤0.5. This paper will discuss the development of processes to overcome these and other difficulties. Kilogram quantities of the material which exhibits the giant magnetocaloric effect have been prepared. The resultant magnetocaloric effect is slightly less than that obtained by arc-melting high purity Gd metal with Si and Ge. Supported by US DOE, Office of Basic Energy Sciences, Div. of Mats. Scis., under Contract No. W-7405-ENG-82.

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Thermal Aging Studies of $LaNi_{4.25}Al_{0.75}$ Hydrides and Tritides: *D. Chandra*[1]; W. N. Cathey[1]; D. Clare[1]; H. Mandalia[1]; J. R. Wermer[2]; J. S. Holder[3]; W. C. Mosley[3]; [1]University of Nevada, Metallu. and Matls. Eng. Div., Mackay School of Mines, Mail Stop 388, Reno, NV 89557 USA; [2]Los Alamos National Laboratory, Mail Stop C 348, Los Alamos, NM 87545 USA; [3]Westinghouse Savannah River Company, Savannah River Tech. Ctr., Aiken, SC 29808 USA

The $LaNi_5$ type hydrides are important for applications such as hydrogen/tritium storage systems and others. In this study, thermal aging of $LaNi_{4.25}Al_{0.75}$-hydride was performed in the range of 473 to 583K, and $LaNi_{4.25}Al_{0.75}$-tritide at room temperature. The hydrogen aging experiments performed at 473K, showed a rapid initial decrease in hydrogen pressure followed by gradual decreases in pressure; the initial hydrogen pressure was 1930 kPa. Isotherms taken at 389K, after hydrogen thermal aging at 473K, showed that the desorption pressure of the isotherm decreased by approximately 45 kPa. Vacuum annealing of $LaNi_{4.25}Al_{0.75}$ showed that the plateau pressure decreases were not due to metallurgical heat treatment but because of hydrogen interactions. Similar decreases in hydrogen pressures were obtained from studies on thermally aged $LaNi_{5-x}M_x$ with Sn (x=0.24) and Mn (x=0.4) substitution. Thermal aging at 563K and 583K also showed similar trends as that of 473K aging, except that desorption isotherms developed slopes at lower H/M values; the absorption isotherms were unaffected. Room temperature aging of $LaNi_{4.25}Al_{0.75}$-tritide for 6.8 years showed that the desorption isotherms developed a steep slope. During tritium aging, helium-3 formed due to radiolytic decay and remained trapped in the metal lattice. This resulted in non-uniform strain in the lattice which broadened the X-ray diffraction Bragg peaks. The thermal aging results will be discussed.

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Hydrogen Amorphization of $GdFe_2$ Laves Phase Hydride: *Dhanesh Chandra*[1]; Ricardo B. Schwarz[2]; [1]University of Nevada, Metallu. and Matls. Eng. Div., Mackay School of Mines, Mail Stop 388, Reno, NV 89557 USA; [2]Los Alamos National Laboratory, Matl. Sci. and Tech. Div., Mail Stop G755, Los Alamos, NM 87545 USA

We have investigated the effect of hydrogen interaction with $GdFe_2$ Laves phase alloy. Crystalline hydrides are formed at relatively low temperatures and pressures, and the reaction is reversible. A $GdFe_2H_{4.8}$ is formed at room temperature, which is reversible, with a H/M ratio of 1.6. However, at this temperature desorption of all the hydrogen from the hydride is difficult because of slow kinetics and, for pressures below 10^3 Pa, the desorption isotherm deviates from the absorption isotherm. An amorphous $GdFe_2H_x$ phase forms at intermediate temperatures and pressures. The absorption isotherm for this hydriding reaction is quite unusual in that, as the crystal-to-amorphous transformation is accompanied by either a gain or a loss of hydrogen. Absorption isotherms taken below 475K showed that there is an abrupt decrease in the hydrogen capacity of the alloy during the crystalline-to-amorphous hydride phase transformation. Whereas the absorption isotherms taken above 475K showed that there is an abrupt increase in the hydrogen capacity during amorphization. At temperatures above 525 K, hydrogen absorption causes to the disproportionation of the $GdFe_2$ crystal into a two-phase mixture of GdH_2 and bcc α-Fe. The formation of the crystalline and amorphous GdFe2Hx phases, phase stability regions, disproportionation of the hydride will be discussed.

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Metallothermic Processing of Nd from NdF3: *R. S. Chiou*[1]; R. G. Reddy[2]; [1]The University of Alabama, Chem. Eng., Tuscaloosa, AL 35487 USA; [2]The University of Alabama, Metallu. and Matls. Eng., Tuscaloosa, AL 35487 USA

Production of neodymium metal by the reduction of neodymium fluoride with sodium as a reductant in presence of NaCl-NaF flux was studied. Process calculations were carried out using Gibbs energy minimization method. The effect of process parameters such as temperature, pressure, salt and feed materials composition on the yield and impurity content of the products were analyzed. The yield of Nd metal increased with an increase in temperature and the fluxing agents and so did the impurity content of the metal. An excellent agreement was obtained between the experimental and the calculated data. The results were also compared with other metallothermic processes for the production of neodymium.

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Kinetics Studies of Nitridation of Fe17-Nd2 Alloys: V. V. Hong[2]; R. E. Aune[1]; *Seshadri Seetharaman*[1]; [1]Royal Institute of Technology, Dept. of Metallu., SE-100 44, Stockholm, Sweden; [2]The Vietnam Center for Science and Technology, Instit. of Matls. Sci., Dept. of Rare Earth Met., Hoang Quoc Viet-Cau giay, Hanoi, Vietnam

The magnetic properties of Fe-Nd alloys are significantly improved by the introduction of interstitial elements like boron and nitrogen. In the processing of these property optimized magnetic alloys, it is important to have access to the kinetic information regarding the nitrogen uptake by these alloys at the process temperatures. The present work was undertaken to study the kinetics of nitridation of Fe17-Nd2 magnetic alloys in the temperature range 1173-1473 K. Thin plates of the high purity alloy (10 mm diam, initial weight between 207 and 463 mg) were used. The increase in mass of the sample was followed using a SETARAM, TAG 92 unit, as a function of time at 1173, 1273, 1373

and 1473K and nitrogen partial pressures of 100%, 75%, 50%, and 25%(vol%). Nitrogen gas was purified so that the partial pressure of oxygen was < 10 -23 bar. The incubation period was a function of temperature, followed by a rapid nitridation period, and a slow down in the reaction rate due to the formation of product layer. Oxygen impurity have a strong influence on the reaction kinetics. The activation energy for the rate controlling step during the initial stages of nitridation was evaluated. The reaction mechanism and the effect of oxygen impurity on the reaction rate are discussed.

5:00 PM
Gaseous Fluorination of Metal Hydrides: *M. L. Anderson*[1]; I. E. Anderson[1]; J. C. Foley[1]; [1]Iowa State University, Ames Lab., Metallu. and Cer., Ames, IA 50011 USA

Corrosion of the metal hydride electrode in nickel-metal hydride (Ni/MH) rechargeable batteries is a common cause of failure. The primary cause of the corrosion is the interaction with the caustic potassium hydroxide (KOH) electrolyte solution. A method has been developed to react the lanthanum contained within the metal hydride material with nitrogen trifluoride gas (NF3), creating a lanthanum fluoride (LaF3) layer at the surface of the powders. Development of a consistent passivation layer would enable manufacturing of longer lasting Ni/MH batteries. The method used to create this coating and results pertaining to the effect the coating has on the ability of the material to perform as a battery electrode will be described. The Materials Science Division of DOE/BES provided funds for this research under contract W-7405-Eng-82.

Research and Development Efforts on Metal Matrix Composites: Processing of MMCs

Sponsored by: Joint ASM-MSCTS/TMS-SMD Composites Committee; Young Leaders Committee
Program Organizers: John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; Warren H. Hunt, Aluminum Consultants Group Inc., Murrysville, PA 15668 USA

Wednesday PM Room: Bayou A
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Warren H. Hunt, Aluminum Consultants Group, Murrysville, PA 15668 USA; James C. Foley, Ames National Laboratory, Ames, IA 50011-3020 USA

2:00 PM Invited
Status, Issues and Opportunities in Processing of Metal Matrix Composites: *Glenn S. Daehn*[1]; [1]The Ohio State University, Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

If one starts by considering the optimization of metal matrix composite properties, a list of technical issues that must be dealt with through processing can quickly emerge. Concerns include: uniform dispersion of reinforcement, scale of reinforcement and microstructure, thermodynamic compatibility, mismatch in thermal expansion to name a few. In some way these issues can work either with or against the composite and process designer and must be considered together when designing a composite and its manufacturing process. This presentation will consider the limiting cases of how one might create a composite (solid-consolidation, reaction processing, solidification of slurries, and direct deposition). From this an attempt will be made to point out under-exploited opportunities for reducing cost and improving performance in metal matrix composites.

2:30 PM
In-Situ Nondestructive Evaluation Method for Characterizing Sintering of Metal Matrix Composites: *James C. Foley*[1]; David K. Rehbein[1]; [1]Ames Laboratory, Metallu. and Cera. Pgm., 122 Metals Dev., Ames, IA 50011 USA

Renewed interest in the area of metal matrix composites has spurred the development of technologies to enable the production of low-cost metal matrix composites. One such technology that is being developed at the Department of Energy's Ames Laboratory is an in-situ nondestructive evaluation method to characterize sintering. The new method consists of a high temperature EMAT to measure the amplitude and velocity of an ultrasonic tone burst traveling through a sample during sintering. Samples of Al-4 wt% Cu + 10%, 20% and 30% SiC respectively were examined with the new method. In addition, the samples were examined with standard metallography, density measurement, thermal analysis and mechanical testing techniques. Comparison of obtained results demonstrates that density and the extent of sintering can now be directly observed as a function of time with the new technique. Support from an Ames Laboratory directed research and development grant and DOE-BES-DMS under contract no. W-7405-Eng-82 is gratefully acknowledged.

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Thermodynamics of In-Situ Reaction between TiO2 and Pure Al: *Iulian Gheorghe*[1]; Henry J. Rack[1]; [1]Clemson University, Cer. & Matls. Eng. Dept., 204 Olin Hall, Clemson, SC 29634 USA

The reaction between the matrix and reinforcement is normally considered deleterious in the fabrication of MMC's. Indeed major efforts have been made to avoid matrix-reinforcement reaction Al-SiC, Al-Graphite, etc. However it is possible to fabricate thermodynamically stable systems by recognizing the inevitability of these reactions and fabricating composites that allow the reactions to progress to completion. This presentation summarizes the thermodynamic design basis for the in-situ formation of Al2O3 reinforced (Ti3Al, TiAl, Al3Ti). Thermodynamic calculations show that the reduction of TiO2 to Al2O3 and the formation of titanium aluminum intermetallic compounds Ti3Al, TiAl, Al3Ti is possible within a large temperature range. Isothermal Ti-Al-O cross sections at 800°C and 1100°C indicate that either Ti3Al + Al2O3, TiAl + Al2O3, and Al3Ti + Al2O3 two-phase equilibrium, or Ti3Al + Al2O3 + alpha-Ti, Ti3Al + TiAl + Al2O3, and Al + Al3Ti + Al2O3 three-phase equilibrium is possible. Furthermore the calculated Al-TiO2 pseudo-binary phase diagram was calculated and is showing the stability of Al2O3 + Ti3Al, Al2O3+ TiAl, Al2O3+ Al3Ti, Al2O3+ Ti3Al + TiAl, and Al2O3+ TiAl + Al3Ti phase regions up to very high temperatures creating in the same time the base for the design of quantitative experiments. This work is supported by the Office of Naval Research and coordinated by Dr. S. Fishman, under contract No. 96PR07712-00.

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High Strain Rate Superplasticity of In-Situ ZrAl3 Fiber Reinforced Aluminum Composites: *Tsunemichi Imai*[1]; Jianfu Mao[2]; Lin Geng[2]; Sumito Kojima[3]; [1]National Industrial Research Institute of Nagoya, 1-1 Hirate-cho, Nagoya 462-8150 Japan; [2]Harbin Institute of Technology, Harbin 150001 PRC; [3]Nagoya Municipal Industrial Research Institute, 3-4-41 Rokuban-cho, Atsuta-ku, Nagoya, Japan

Nano ZrO2 particles used as reinforcement were mixed with pure aluminum powder (1N90 and sintered at 823K under the pressure of 285MPa for 20 minutes. This produced ZrAl3 fiber reinforced 1N90 pure aluminum composite with fiber diameter less than 1mm by reaction of ZrO2 with aluminum. The volume fractions of ZrO2 selected were 0.05, 0.10 and 0.15. Sintered ZrAl3/1N90 Al composites were extruded with the extrusion ratio of 44:1 at 823K. The extruded ZrAl3/1N90 composite(Vf=0.05) exhibited an m value more than 0.3 and a total elongation of about 150% at the strain rate of 0.1/sec at 923K. Hot rolling the composite after extrusion increases the total elongation to about 200% at 0.1/sec and at 913K. TEM micro-characterization clarifies that the fine fiber should be ZrAl3 and grain size of ZrAl3/1N90 composite is 2~3mm.

3:30 PM Break

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Interface Design Strategies for High Temperature Composite Systems: *Joon Sik Park*[1]; John H. Perepezko[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., 1509 Univ. Ave., Madison, WI 53706 USA

A clear understanding of the basic factors controlling reproducible composite processing is critical for the high temperature application. Since the usual composite materials represent ternary or higher order systems, model ternary systems including potential reinforced materials such as B4C and SiC phases have been examined in order to provide effective strategies for the composite materials design. One attractive approach to obtain the stable phase combination (i.e. compatibility) is to produce the desired phase combination by using controlled interface reaction to achieve in-situ synthesis. For example, if the reaction between matrix and reinforcement materials does not yield a preferred phase combination, the diffusion path can be biased to produce the desired phase combination. An effective approach to control the reaction products and diffusion pathway by adding an extra component layer as a kinetic bias has been developed and investigated based upon flux calculations and a semi-empirical database. The growth kinetics of the product phases and the effect of the biasing layer during interdiffusion reaction will be presented for Ni/SiC and TiSi2/TiAl systems. The influence of the kinetic bias includes not only a control over the phase selection and sequencing, but also a control over the microstructural morphology that develops during interdiffusional reactions. An analysis of the diffusional interactions in terms of component chemical potential variations provides useful general guidance for reaction path control and the limiting kinetics. With an interface design strategy based upon a diffusion path directed by kinetic biasing to include thermodynamically stable phase combinations, the resulting in-situ synthesis offers a self-healing of internal damage to yield robust composite performance. The support of ONR (N00014-92-J-1554) is gratefully acknowledged.

4:00 PM

Reactive Infiltration Processing and Compression Creep of NiAl and NiAl Composites: *T. A. Venkatesh*[1]; David C. Dunand[2]; [1]MIT, Matls. Sci. and Eng., Rm. 8-139, 77 Massachusetts Ave., Cambridge, MA 02139 USA; [2]Northwestern University, Matls. Sci. and Eng., 2225 N Campus Dr., MLSB 1123, Evanston, IL 60208 USA

Reactive infiltration processing of bulk and composite NiAl was investigated with powder and wire preforms of nickel. Inhomogeneous microstructures were often obtained with powder preforms because their high surface-to-volume ratio, low permeability, and irregular infiltration paths lead to simultaneous infiltration and reaction. Homogenous NiAl could be obtained with nickel-wire preforms which had a lower surface-to-volume ratio, higher permeability, and regular infiltration paths, because infiltration was completed before the onset of reaction. Composites with continuous tungsten (W) and sapphire fibers were also successfully fabricated by reactive infiltration, while composites with molybdenum particulates and short-fibers showed significant dissolution in NiAl. The high-temperature uni-axial compression creep behavior of uni-directionally reinforced continuous fiber composite materials was investigated using NiAl-W as a model system for the case where both the NiAl matrix and the tungsten fiber underwent plastic deformation by creep. The creep behavior of the constituents NiAl and W and NiAl composites reinforced with 5-20 volume% W was characterized at 1025°C and 715°C. At 1025°C, the NiAl-W composites exhibited three stage creep behavior with distinct primary, secondary, and tertiary creep, where the composite creep-rate decreased monotonically, remained constant, and increased rapidly, respectively. At 715°C, the NiAl-W composites exhibited insignificant primary and tertiary creep but significant secondary creep. Microstructurally, primary and secondary creep were characterized by pure uni-axial compression of tungsten fibers while brooming, bulging, buckling, and kinking were four fiber deformation modes that contributed to tertiary creep. The observed secondary creep behavior correlated well with the rule-of-mixtures isostrain model developed for composites where both phases undergo creep deformation while new models were developed for the composite primary and tertiary creep and were correlated reasonably well with the experimental results.

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Excimer Laser Shock Processing on Metal Matrix Composites: *James G. Cross*[1]; Jong-Kook Park[1]; Kali Mukherjee[1]; [1]Michigan State University, Matls. Sci. & Mech., High Energy Laser Process. Lab., East Lansing, MI 48824 USA

Particulate reinforced metal matrix composites (PRMMC's) have received strong interest due to the potential advantages over monolithic metal alloys in numerous engineering applications. However, the PRMMC's retain high levels of residual stresses, due to a mismatch of coefficient of thermal expansion (CTE), between a ceramic reinforcement and a metal matrix. The residual stresses result in surface crack initiation, which limits structural application of metal matrix composites. Laser shock processing (LSP) is a unique way to locally alter the residual stress profile on a metal matrix composite. By using a very short laser pulse with high energy density, LSP can generate surface plasma that induces high-pressure stress wave propagation. The residual stresses can be relieved on the surface by incident shock waves, which are generated by the confined expansion of the high-pressure plasma. A KrF excimer laser is used for the pulsed laser irradiation, on a stir-melt processed alumina/6061 aluminum composite. An x-ray diffractometer is used for measurements of the residual stresses.

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Friction-Stir Welding of Metal-Matrix Composites: Aluminum 6061-20% Al_2O_3 and Aluminum A339-10% SiC: *Lawrence E. Murr*[1]; Ying Li[1]; Elizabeth A. Trillo[1]; [1]The University of Texas, Metallu. and Matls. Eng., 500 W. University Ave., El Paso, TX 79968-0520 USA

Friction-stir welding (FSW) involves deformation-induced dynamic recrystallization to facilitate solid-state, superplastic flow. In the joining of aluminum alloys such as 6061, heat affected areas cause reductions in residual strength and hardness of nearly 50%, and strategies to reduce this behavior have been only partially successful. However, when Al_2O_3 particles are added to aluminum alloy 6061, the initial yield stress and hardness are increased; nominally by 33% at 20% particle additions. When aluminum alloy 6061 + 20% Al_2O_3 MMC is friction-stir welded, there is a reduction in hardness just outside the weld zone of 58%. However, in contrast to 6061 Al, the minimum residual hardness is reduced by only 9%. Similar results are obtained for the FSW of aluminum alloy A339 (11% Si, 1% Cu, Mg, and Ni, 0.5% Fe, balance Al) + 10% SiC; and this MMC welded to the Al 6061 + 20% Al_2O_3. In each case the hard particles are homogeneously stirred into the weld zone, which has been dynamically recrystallized. Examination and comparison of the microstructures associated with FSW in these systems not only provides some examples of the technological potential for FSW in joining complex and dissimilar MMC systems, but also the opportunity to examine particulate flow phenomena in solid-state, extreme deformation processing. This is especially notable in the examination of intercalated flow in the Al-6061-20% Al_2O_3/Al-A339-10% SiC system which is facilitated by differential etching in optical metallography. Implications for friction processing such as friction extrusion reforming of previously formed MMC are also presented. Research supported in part by a NASA Cooperative Agreement (NCC8-137) and by a General Services Administration grant.

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Distribution of Reinforcing Particles in MMCs Produced by Spray Atomization and Deposition: *Qingzhou Xu*[1]; Enrique J. Lavernia[1]; [1]University of California, Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697 USA

Aluminum-based MMCs are synthesized by spray atomization and deposition combined with a co-injection method or a stirring method. The present work attempts to provide insight into the distribution of reinforcing particle in the different processing approaches. For the co-injection method, it is easy to incorporate SiC particles into the spray of metallic droplets, but the aggregation of SiC particles often is very severe as a result of the non-uniform spatial distribution of particles which come from several isolated injectors. For the stirring method, it is difficult to add SiC particles into aluminum liquid due to the effect of surface tension. However, the particle distribution is very uniform in the matrix since MMCs are generated through the accumulation of individual droplets containing SiC particles. Additionally, the

interactions between SiC particles and the moving solid-liquid solidification interface as well as their influence on the final distribution of reinforcing particles are analyzed theoretically.

Surface Engineering in Materials Science I: Coating/Films Properties Evaluation (PE)-II

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee
Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816 USA; Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA; John Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA

Wednesday PM Room: Canal B
March 15, 2000 Location: Opryland Convention Center

Session Chairs: John J. Moore, Colorado School of Mines, Adv. Coat. and Surf. Eng. Lab., Golden, CO 80401-1887 USA; John D. Demaree, Army Research Laboratory, Weapons & Matls. Rsrch. Directorate, Aberdeen Proving Ground, MD 21005-5069 USA

2:00 PM
Prediction and Evaluation of Mechanical Properties of Two Laser Surface Processed Low Carbon Steels: *Mary Helen McCay*[1]; Narendra B. Dahotre[1]; John A. Hopkins[1]; T. Dwayne McCay[1]; [1]University of Tennessee Space Institute, Ctr. for Laser App. MS 24, B. H. Goethert Pkwy., Tullahoma, TN 37388 USA

Two low carbon steels were coated with compositional mixes of chromium and chromium/nickel powders and laser surface processed at four different energy levels to investigate the ability to predict, and therefore control, the metallurgical properties. Metallurgical analysis consisted of metallographic observations, hardness tests, x-ray diffraction, SEM and microprobe compositional determination and wear tests. Analytical calculations using processing parameters and compositional mix parameters were employed to predict melt depths and final alloy composition. Nickel and chromium equivalents were then calculated and the phases evaluated based upon the Shaeffler diagram (for lower compositions) and the Fe, Ni and Cr phase diagrams (for higher compositions). This provided the basis for estimations of hardness and wear. Results show the possibility of predicting laser surface processed alloy layer phases and properties.

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Tribological Properties of Diamond Coatings and Their Application to a Machine Element: *Ryo Nawata*[1]; Hitoshi Tokura[1]; [1]Tokyo Institute of Technology, Dept. of Mech. Eng., 204 Ishikawadai Kenkyuzikkento, 2-12-1 O-okayama Meguro-ku, Tokyo 152-8552 Japan

Almost twenty years have past since the synthesis of diamond coatings by chemical vapor deposition (CVD) was completed. Diamond coatings have many excellent properties, such as high hardness, a low friction coefficient and low wear rate. Nevertheless, they have not many uses, except for cutting tools, because polishing diamond coatings consumes much time and money. From a point of view described above, if as-deposited diamond coatings can be used without polishing, they will be applied to many machine elements. Firstly, we examined tribological properties of diamond coatings as the basic research. Diamond coatings were deposited on cemented carbide pins and disks. Diamond coated pins were rubbed against diamond coated disk and stainless steel and titanium disks in different environments. When as-deposited diamond coatings were rubbed against as-deposited diamond coatings, we confirmed that the friction coefficient decreased rapidly. Some studies on this phenomenon have been reported and several mechanisms have been proposed. This phenomenon suggests that as-deposited diamond coatings can be used without polishing. From observation of the surface by scanning electron microscopy (SEM), the change of surface morphology caused this phenomenon. Secondly, making use of this phenomenon, we applied diamond coatings to a machine element such as bearings.

2:40 PM
Study of Corrosion Behavior of Laser Glazed and TiC Laser Coated H13 Die Steel: *Daiel Pirzada*[1]; E. G. Baburaj[1]; M. R. Govindaraju[2]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]Karta Technology Inc., 1892 Grandstand, San Antonio, TX 78238 USA

There is a growing interest in application of intermetallic and ceramic coatings and surface treatments to extend the die-casting die life. These coatings reduce the molten metal corrosion and erosion to the die. Due to its excellent corrosion and highly abrasion resistant nature titanium carbide is a suitable candidate for coating die-casting dies. Amongst the different surface treatments and coating technologies being presently used, laser surface engineering is very attractive due to its flexibility, depth of penetration, high solidification rates (103-108 K s-1), and production of metastable phases. This paper presents the results of a study involving isothermal corrosion of laser surface treated and TiC laser coated H13 steel under accelerated corrosion conditions. The effects of grain size of the TiC coating and different laser surface treatment parameters on corrosion behavior of H13 die steel in liquid aluminum alloy A390 have been investigated. A significant improvement in corrosion resistance was achieved for H13 steel coated with TiC. The finer grain size and laser shot peening contribute significantly towards improving the corrosion behavior of steel in molten aluminum. Based on metallographic studies and energy-dispersive spectrometry (EDS) the effectiveness of the coatings along with the possible reason for their behavior are presented. The potential applications for laser surface treatment and TiC coatings to address specific industrial problems are also discussed.

3:00 PM
Effect of Deposition Temperature on the Physico-Chemical Behavior of Ti-Al-N Thin Films: *S. Seal*[1]; A. Kale[1]; V. Desai[1]; D. Jimenez[1]; K. Sundaram[2]; N. Dahotre[3]; [1]University of Central Florida, AMPAC & MMAE, Eng. 381, Orlando, FL 32816 USA; [2]University of Central Florida, Elect. and Comp. Eng. Dept., Orlando, FL 32816 USA; [3]University of Tennessee Space Institute, Ctr. for Laser App., Dept. of Matls. Sci. & Eng., MS-24, B. H. Goethert Pkwy., Tullahoma, TN 37388 USA

Nitride based hard coatings are of prime interest in today's cutting tool technology. Ternary (Ti,Al)N with a 1:1 Ti:Al ratio seems to be a promising alternative candidate to the widely used titanium nitride. The major concern of using TiN in high-temperature applications is due to the fact, that it oxidizes rapidly at temperatures above 500°C. In contrast, (Ti,Al)N coatings are characterized not only by high microhardness and dense microstructure, but also by their greater thermal stability. In this study, (Ti,Al)N coatings were deposited onto 316SS substrates under ambient and liquid nitrogen temperatures by dc-magnetron sputtering. Both structure and morphology are greatly affected by the deposition temperatures. The as deposited films were oxidized in a vertical fused-silica tube furnace in pure O_2 atmosphere at 850°C for 3.5 and 7.5 hours. Because of their outstanding properties with respect to hardness, wear resistance, oxidation resistance and corrosion resistance, it seems to be desirable to study the crystal structure, mechanical and surface chemical properties of (Ti,Al)N thin films in detail by using SEM, XRD, AES and XPS.

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Corrosion Resistance of Ion Nitrided AISI 304 and 316L Stainless Steels: *R. Vallerio*[1]; M. Landis[1]; R. Hidalgo[1]; K. Marchev[2]; B. C. Giessen[1]; [1]Northeastern University, Barnett Instit. and Dept. of Chem., Boston, MA USA; [2]Saint Gobain Industrial Ceramics Inc., Norton Diamond Film, Northboro, MA USA

As the technology of ion nitriding stainless steels progresses, it becomes imperative to examine the effect of nitriding on the corrosion resistance of the surface of the treated steel. This work will give some insight into the relationship of the conditions of the nitriding treatment to the corrosion resistance of the surface layer through their effect on its structure. By varying the nitriding conditions, single or multi-phase nitride layers can be obtained, leading to dramatic changes in the corrosion resistance. As reported earlier, a textured tetragonal martensitic phase (mphase) can be produced by nitriding under mild conditions. The corrosion resistance of m phase was compared and found to be superior to that of a mixture of phases (ε and γ) present after higher temperature treatments.

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Wear Behavior of Cr3C2-NiCr Detonation Spray Coating: *Jun Wang*[1]; Sun Baode Li[1]; Yaohe Zhou[1]; [1]Shanghai Jiao Tong University, School of Matls. Sci. & Eng., Shanghai, China

Coatings can be applied to surfaces to improve the surface characteristics over those of the bulk properties and are widely used in tribological applications either to reduce wear and/or to modify friction during contact. One of the foremost coating methods for combating wear is thermal spraying, however, despite its widespread industrial use, little is known about the basic friction behavior and the mechanisms by which such coatings wear. Thus, most thermal spray wear coating applications and developments are based on empiricism. In order to prolong the conticaster roll's life, Cr3C2-NiCr detonation spray coating has been processed on the roll surface in the steelmaking plant of Bao ShanSteel Company. The wear behavior of the coating was studied systematically in this paper. The abrasive and dry frictional wear testing were performed on a pin-on-disk tester. Experiment results show that the wear resistance of the coated samples without the risk of seizure are much better than those of the uncoated at room and elevated temperature with any load and sliding velocity. And the coating wear mechanisms under variation test condition were discussed.

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Development and Testing of Corrosion-Resistant Properties of CVD Mullite Coatings for Silicon Nitride: *Svetlana M. Zemskova*[1]; James A. Haynes[1]; Matthew K. Ferber[1]; Kevin M. Cooley[1]; David P. Stinton[1]; [1]Oak Ridge National Laboratory, Met. and Cer., P.O. Box 2008, 1 Bethel Valley Rd., Oak Ridge, TN 37831-6063 USA

Recently it has been demonstrated that thin (3-5mm) chemical vapor deposited (CVD) mullite 3Al2O3x2SiO2 provides excellent oxidation protection for Si3N4 and SiC in high-pressure steam. However, CVD mullite microstructure and composition may substantially influence the coating properties. It was found that the Al:Si ratio in CVD coatings with "mullite-like" structures can be varied from 1:1 up to 9:1 depending on the deposition conditions. The present investigation is aimed at development of CVD parameters for fabrication of dense, uniform, crystalline mullite coatings with controlled Al:Si ratios. The mechanical properties of as-coated Si3N4 specimens were tested by four-point bending and compared with the properties of non-coated material. The effect of mullite Al:Si ratio on coating microstructure, mechanical properties and oxidation resistance was evaluated.

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Influence of Boron Ion Implantation on the Mechanical Properties of TiN Coatings Deposited by Cathodic Arc Evaporation: *Yao-Can Zhu*[1]; Y. Matusmoto[1]; K. Fujita[1]; N. Iwamoto[1]; N. Nagasaka[2]; T. Kataoka[3]; [1]Ion Engineering Research Institute Corporation, 2-8-1, Tsuda-yamate, Hirakata, Osaka 573-0128 Japan; [2]Ebara Research Company Limited, 2-1,Honfuzisawa 4-chome, Fujisawa 251-8502 Japan; [3]Ebara Corporation, 20-1 Nakasode, Sodegaura, Chiba-ken 299-0296 Japan

TiN coatings were deposited on SUS420J2 steel substrates by cathodic arc evaporation. Boron ions were implanted into TiN coatings at 75keV with a dose range of 1.0 ~ 8.0x1017 ions/cm_2. The composition and crystallographic structure of as-deposited and implanted TiN coating were characterized by X-ray photoelectron spectroscopy and Glancing-angle X-ray diffraction. A nanoindentor was employed to measure the hardness of TiN coatings. Ball-on-disc wear tests were carried out to evaluate wear resistance of as-deposited and implanted TiN coatings. The results of wear tests showed that boron ion implantation resulted in a significant improvement of the wear resistance of TiN coatings.

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Performance Comparison Between Various Carbide & Metal Co-Sputtered Coatings: *F. M. Kustas*[1]; B. Mishra[2]; J. Zhou[2]; [1]Engineered Coatings, P.O. Box 4702, Parker, CO 80134-4702 USA; [2]Colorado School of Mines, Dept. of Metallu. & Matls. Eng., Golden, CO 80401 USA

Carbide coatings have excellent potential for wear applications if their toughness, wear resistance, and durability can be increased. Addition of metals to ceramics is a well know method to increase toughness, as demonstrated by traditional cermet (ceramic/metal) bulk materials technology. Using a similar approach, several different carbide & metal cermet systems were fabricated in coating-form using bias-assisted unbalanced magnetron co-sputtering from a single target. Cermet systems that were fabricated for subsequent evaluation included boron carbide (B4C) & molybdenum (Mo), titanium carbide (TiC) & tungsten (W), TiC/titanium diboride (TiB2) & W, TiC/chromium carbide (Cr3C2) & metals, and silicon carbide (SiC) & metals. Coating elemental composition (by XPS) and structure (by XRD) were measured, while scratch adhesion, microhardness, and wear tests were performed to enable performance comparisons between the different cermet systems. In general, unique performances were measured for different cermet systems. For example, the TiC&W system exhibits excellent wear resistance (non-measurable wear against WC-Co at an initial stress of 1.3 GPa (192 ksi), whereas the B4C & Mo system has extremely high hardness (>5000 HKN). Reasons for the different performances of the cermet systems are proposed and candidate applications for these unique cermet coatings are discussed. Work partially supported by a Department of Energy grant under the Entrepreneur's Technical Assistance (ETAP) Program.

Ultrafine Grained Materials: Mechanical Behavior and Strengthening Mechanisms: II

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee, Shaping and Forming Committee

Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; S. L. Semiatin, Wright Laboratory, Materials Directorate, Dayton, OH 45440 USA; C. Suryanarayana, Colorado School of Mines, Department of Metal and Materials Engineering, Golden, CO 80401 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Wednesday PM — Room: Polk A/B
March 15, 2000 — Location: Opryland Convention Center

Session Chair: Ruslan Z. Valiev, Institute of Physics of Advanced Materials, Ufa 450000 Russia

2:00 PM Invited

Creep Inhibition of Ceramic/Ceramic Nanocomposites: *Tatsuki Ohji*[1]; [1]National Industrial Research Institute of Nagoya, Superplastic Nanoscience Lab., Hirate-cho, Kita-ku, Nagoya 462-8510 Japan

The dispersion of nanometer-sized silicon carbide particles into alumina or silicon nitride matrix results in significant improvements in creep resistance. The creep rates of the nanocomposites are about several orders of magnitude lower than those of the monolithic ceramics. In this paper much attention is paid to the interfaces between the intergranular nanoparticles and the matrix, and its role in creep inhibition. It will be shown that the intergranular nanoparticles are rigidly bonded to the matrix by several approaches. The rigid bonding of the interfaces causes the inhibition of grain boundary sliding, leading the remarkably improved creep resistance. The importance of change in grain boundary chemistry by doping the nanoparticles is also emphasized. In addition, below specific stresses, the creep rates are remarkably decayed, suggesting the presence's of the threshold stresses below which creep stops. The estimated threshold stress ranges from ten to several ten MPa, depending on the volume fraction of the nanoparticles. These stresses agree with those predicted from the Ashby's model, where motion of grain boundary dislocations responsible for vacancy nucleation and annihilation is considered to be pinned by hard particles.

2:25 PM Invited

Deformation Model During Equal-Channel Angular Pressing: *Patrick B. Berbon*[1]; [1]Rockwell Science Center, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA

In the last few years, the equal-channel angular (ECA) pressing technique has allowed remarkable achievements in the production of submicrocrystalline (SMC) materials with superb superplastic properties, particularly at low temperatures and high strain rates. Although some of the observed mechanical properties can be explained by the small grain size resulting from the process, other results are more puzzling. It appears they are caused by the specific mode of deformation occurring in ECA processing. In this paper, we are first reviewing the specificity of this metal working technique, and we are then proposing a possible deformation mechanism to explain the observed microstructures, their thermal stability, and the superb superplastic properties.

2:50 PM Invited

Microstructure and Mechanical Properties of Aluminum 5083 Processed by Equal Channel Angular Extrusion: John W. Sinclair[1]; *K. T. Hartwig*[1]; R. E. Goforth[1]; [1]Texas A&M University, Mech. Eng., 800 Swan Place, Worland, WY 82401 USA

Commercial grade Al-5083 was processed in bulk form by equal channel angular extrusion to a submicron sized microstructure with boundaries ranging in size from 0.3 to 0.50 μm and the room temperature strength and superplastic performance at 510°C and 350°C investigated. Processing involved hot working the material at 300°C followed by warm working at 200°C and 170°C to a cumulative processing strain of ~ 13.8. Inclusion of an intermediate solution-heat treatment at 530°C following a processing strain of ~ 9.2 was found to enhance superplasticity. Particle sizes greater than 0.56 μm were found to have an adverse effect on superplastic performance. Microtexture evolution indicated a progressive increase in misorientation angle of the boundaries as deformation strain increased. Conventional superplasticity was achieved at 510°C with elongation to failure over 400% and a strain-rate sensitivity index of 0.35. Marginal superplasticity was achieved at 350°C with elongation to failure over 250% and a strain-rate sensitivity index of 0.23. The measured particle volume fraction of 0.02 was insufficient to adequately stabilize the submicron-size microstructure against grain growth at both temperatures.

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Creep Behavior of Carbon Doped Nanocrystalline Nickel: *W. M. Yin*[1]; *S. H. Whang*[1]; R. Mirshams[2]; C. H. Xiao[2]; [1]Polytechnic University, Dept. of Mech. Eng., Six Metrotech Ctr., Brooklyn, NY 11201 USA; [2]Southern University and A&M College, Dept. of Mech. Eng., P.O. Box 9987, Baton Rouge, LA 70813 USA

Nanostructured nickel processed by pulse plating exhibits an excellent combination of full density, uniform grain size and ultrahigh tensile strength. Nevertheless, it shows room temperature creep under high stress conditions. It appears that the room temperature creep might be associated with high grain boundary diffusion. From this prospective, it is interesting to investigate effect of a particular solute element on the creep of this material, in that the solute plays a significant role in the grain boundary characteristics. In this presentation, we will report tensile properties and creep behavior of nanostructured nickel doped with 250ppm carbon. The experimental results showed that Young's modulus has been improved significantly by adding 250ppm carbon while the tensile ductility and yield strength decreased at room temperature in contrast with the solute hardening in the polycrystalline nickel. The tensile creep tests showed enhanced creep strain rate at room temperature and 373K under the stress ranging from 200MPa to 800MPa compared with that of pure nanocrystalline nickel. Further creep tests will be performed at low loads 50-100MPa and higher temperatures. The conventional TEM has been employed to investigate microstructures of the crept specimens. Effect of carbon on creep mechanisms at various conditions will be discussed based on mechanical testing results and microstructure characterization.

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Superplastically Sinter-Forged Si_3N_4 and Si_3N_4-SiC Ceramics: *Naoki Kondo*[1]; Yoshikazu Suzuki[1]; Tatsuki Ohji[1]; [1]National Industrial Research Institute of Nagoya, Superplastic Nanoscience Lab., Hiratecho, Kita-ku, Nagoya 462-8510 Japan

The microstructures and mechanical properties of Si_3N_4 and Si_3N_4-SiC, produced by a superplastic sinter-forging technique from submicron Si_3N_4 or Si-C powders, were investigated. Both the obtained materials exhibited highly anisotropic microstructures, where rod-shaped Si_3N_4 grains tended to be aligned perpendicularly to the forging direction. In addition, for the Si_3N_4-SiC material, β-silicon carbide grains with micrometer-size and nanometer-size were found at the grain boundaries and within the Si_3N_4 grains, respectively. Very high bending strength as well as high fracture toughness were achieved when a stress was applied perpendicularly to the pressing direction.

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Superplastic Microstructure of Modified AA-5083 Aluminum Alloy Processed by Equal Channel Angular Extrusion: *Darrell R. Herling*[1]; Mark T. Smith[1]; [1]Pacific Northwest National Laboratory, Matls. Process., 902 Battelle Blvd., Mail Stop: P8-35, Richland, WA 99352 USA

Current processing methods that are used to develop fine-grained superplastic microstructures in aluminum alloys involve extensive hot and cold deformation steps, usually in the form of hot and cold rolling. This approach has distinct limitations that can have a significant influence on the cost and quality of superplastic forming (SPF)-grades of aluminum sheet. First, the extensive cold rolling required for SPF aluminum sheet typically results in substantial edge cracking and overall yield losses. The second limitation is that the high levels of hot and cold work necessary to achieve the desired microstructure requires starting with very large ingot size, while the final product is usually limited to thin gage sheet. In addition, through conventional rolling thermal-mechanical-processing (TMP) schedules, the microstructure is typically limited to 5-10 micrometer grain size. An improvement in SPF performance can be achieved with the development of smaller-grain equiax microstructure ~1 micrometer. The Equal Channel Angular Extrusion (ECAE) process offers several potential advantages in the processing of SPF-grade aluminum alloys. The ability of the ECAE process to achieve high levels of work through localized shearing can develop a well defined subgrain structure and provide a mechanism for distributing the eutectic constituent particles and dispersoids that play a critical role in the recrystallization process and resulting thermally stable fine-grain size. In addition, with ECAE there is the unique ability to achieve these desirable microstructures in bulk form, without reducing the dimensions of the starting material, as is the case in conventional processing of SPF materials. The objective of this work was to process, via ECAE, a 5000-series aluminum alloy in bulk form to produce a fine-grain (~1 micrometer), thermally stable SPF microstructure. Previous work performed at Pacific Northwest National Laboratory on modified 5000-series alloys identified several compositional features that assist in developing a fine, thermally stable microstructure required for SPF. These modification, which include an increase in Mn level and the addition of Zr, develop fine dispersoids that assist in grain refinement and control excessive grain growth at SPF temperatures. Mechanical

tensile testing was conducted to evaluate the SPF properties of the ECAE processed materials.

4:25 PM

An Evaluation of the Applicability of Theoretical Models for Elevated Temperature Plasticity to Ultrafine Grained Materials: *Rajiv S. Mishra*[1]; [1]University of Missouri, Dept. of Metallu. Eng., 218 McNutt Hall, Rolla, MO 65409-0340 USA

In the last ten years synthesis of ultrafine grained materials, including nanocrystalline, has opened up the possibility of extending our knowledge of grain size dependent phenomenon to a much finer microstructural scale. Superplasticity and creep deformation are known to be grain size dependent and the mechanistic understanding in the microcrystalline range is fairly good. The emerging experimental data on elevated temperature plasticity in the ultrafine grain size range allows us to evaluate the applicability of these mechanisms in this new domain. The present analysis shows that the kinetics of grain boundary related deformation processes are significantly slower than the theoretical model predictions. Some thoughts on the reason for these discrepancies are presented.

4:45 PM Discussion Break