Daily Personal Schedule - Thursday - March 16

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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12:00 pm				
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12th International Symposium on Experimental Methods for Microgravity Materials Science: Session 5

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

Thursday AM — Room: Memphis A
March 16, 2000 — Location: Opryland Convention Center

Session Chair: Mike B. Robinson, MSFC/NASA, Science Directorate 47, Huntsville, AL 35812 USA

8:30 AM

Numerical Calculation of the Drag Force Acting on an Insoluble Particle Moving in Front of a Solidifying Interface: *A. V. Catalina*[1]; D. M. Stefanescu[2]; S. Sen[1]; [1]NASA Marshall Space Flight Center, USRA/SD 47, Huntsville, AL 35182 USA; [2]The University of Alabama, Tuscaloosa, AL 35487 USA

The distribution of insoluble particles in a metal casting depends primarily on the interaction of the particles with the solid/liquid interface (SLI) during the solidification process. Microgravity experiments have been performed on board the LMS and USMP-4 missions to study the fundamentals of this interaction. Whether a particle will be engulfed or pushed by SLI is essentially determined by the balance of the forces acting on the particle. An important component of this force balance is the drag force generated by the particle motion in front of the SLI. Previously developed mathematical models for particle/SLI interaction made use of steady-state solutions of this force provided by the lubrication theory. However, our numerical model based on the SLI tracking approach shows that not only the steady-state approach is inappropriate to model the interaction process, but also that even at steady-state the theoretical solution underestimates the drag force. It was found that regression analysis of steady-state numerical solutions for cylindrical particles moving normal to a flat SLI gives a relationship of the form $F_d^{num}=\sqrt{3}\Pi\eta V_p(R_p/d)^{10\gamma/3}$. This is compared to the theoretical solution: $F_d^{theor} = 3\sqrt{2}\Pi\eta V_p(R_p/d)^{3/2}$ where F_d is the drag force, η is the dynamic viscosity of the fluid, V_p is the particle velocity, R_p is the particle radius, γ is Euler's constant ($\gamma\cong0.577$), and d is the width of the gap between the particle and the SLI. The influence of the interface shape on the value of F_d will be discussed. It will be shown that the classical theory to calculate the drag force on a particle can be used only within certain limits. The proposed numerical model was validated against the classical theory within these limits and the microgravity experimental results.

8:50 AM

Numerical Study of Directional Solidification under Microgravity Conditions: *M. El Ganaoui*[1]; [1]Universite d'Aix Marseille, IRPHE-UMR, 6594 CNRS, IMT 38 Joliot Curie, Marseille 13451 France

The numerical modeling in the growth of semiconductor crystals and metal alloys plays an important role in the development of the understanding of the interplay between transport processes such as convection and diffusion. The problems arising: in full-scale computer simulation of crystal growth have stimulated the designing of new efficient and accurate numerical methods for solution of the comples solidification models. In these studying: both theoretical and applied objects are pursued. The selection of numerical algorithm plays an important role in computer simulation of crystal growth. To provide reliable results at a reasonable cost numerical algorithm should appropriate to the specific physical case or technological process that is considered. Two kinds of formulation could be used: the first one utilized independent conservation equations for each phase and ampoule with appropriate boundary conditions at the phase interface. Such methods are referred as multiple domain solutions. The second one consists in continuum formulation in all physical domains which eliminates the need of separating the phase conservation equations. Finite volume monodomain approach associated to homogeneous formulation is used for studying the thermodiffusion Stefan model corresponding to experimental solutions. The approach describes the solidification of a pure material with unsteady melt interacting with the interface and binary alloy with phase transition temperature depending on the composition of the liquid phase.

9:10 AM

Reduction of Microgravity Dendritic Growth Data: *A. O. Lupulescu*[1]; M. E. Glicksman[1]; J. C. LaCombe[1]; M. B. Koss[1]; J. E. Frei[1]; [1]Rennsselaer Polytechnic Institute, Dept. of Matls. Sci. and Eng., 110 8th St., C11 Rm. 4219, Troy, NY 12180-3522 USA

The IDGE consisted of 180 experiments on dendritic growth in succinonitrile (SCN) and 116 experiments on pivalic acid (PVA). Several discoveries were made during each microgravity space flight concerning the behavior of these model dendrites. IDGE film and telemetry data provide benchmark tip velocity and radii versus supercooling for critically testing dendritic transport theory and the interfacial physics of diffusion-limited growth of PVA. To reduce these microgravity data, we developed an image processing system comprised of a monochrome camera, Image-Pro software and custom macros. The macros provide digital tomography and implement the required shape regressions as a series of polynomials for varying regions of the interface near the dendrite tip. The resultant radii are currently being examined as a function of the sampling region. The PVA tip shape data will be added to the data base for SCN tip shapes, allowing quantitative testing of three-dimensional phase-field predictions.

9:30 AM

Theoretical Analysis of Fluid Flow Patterns Near the Solidification Front in Hypermonotectics during Directional Solidification: *T. T. Phillips*[1]; J. B. Andrews[1]; [1]University of Alabama, Dept. of Matls. and Mech. Eng., DEC 254, 1150 10th Ave. S., Birmingham, AL 35294 USA

The intent of this study is to develop a theoretical model that will allow the prediction of the fluid flow patterns that form near the interface during directional solidification of hypermonotectic alloys. Convection driven fluid flows are anticipated in these alloys due to the low-density solute boundary layer that develops. This analysis is part of a larger project to determine how fluid flow in the liquid adjacent to the interface can affect the stability of the solidification front. Experimental verification of the model will be obtained by directionally solidifying samples of the transparent metal analogue system, succinonitrile-glycerol, in a temperature gradient stage microscope. Experimental observation of fluid flow in the liquid phase near the interface will be facilitated by the use of tracer particles.

9:50 AM Break

10:10 AM

Turbulent Magnetically Driven Flows in Levitated Droplets: Suping Song[1]; [1]Washington State University, Schl. of Mech. Eng., Pullman, WA 99164 USA

This paper presents a numerical presentation of turbulent flow phenomena in magnetically levitated droplets. A finite element model with a variety of engineering turbulence models has been developed and applied to study the turbulence phenomena in TEMPUS systems. With the developed model, the turbulence in a magnetically levitated

droplet has been studied. The applied turbulence models include low Reynolds number turbulence flow models, standard k-e model, k-w model, and the k-e Renormalization group model. While the model predictions differ for standard test problems, these models seem to be able to predict the fluid flow level with the accuracy range associated with these models. The results further show that the fluid flow pattern and the magnitude of the velocities and also the temperature distribution within the droplet may be approximated reasonably well by assuming an effective constant molecular viscosity. Further studies are being carried to further assess the droplet oscillation under turbulence conditions.

10:30 AM

Insert Concepts for the Material Science Research Rack 1 (MSRR-1) of the Material Science Research Facility (MSRF) on the International Space Station: *Myscha R. Crouch*[1]; William E. Carswell[2]; Jeff Farmer[3]; Fred Rose[4]; Paul H. Tidwell[5]; [1]National Aeronautics and Space Adminstration, SD42, George C. Marshall Space Flight Ctr., Marshall Space Flight Center, AL 35812 USA; [2]University of Alabama in Huntsville, SD47, Marshall Space Flight Center, AL 35812 USA; [3]National Aeronautics and Space Administration, Marshall Space Flight Ctr., Thermal Design Grp., ED25, Marshall Space Flight Center, AL 36812 USA; [4]Pace and Waite, Inc., SD42, Marshall Space Flight Center, AL 35812 USA; [5]Micro Craft, Inc., 620 Discovery Dr., Huntsville, AL 35806 USA

The Material Science Research Rack 1 (MSRR-1) of the Material Science Research Facility (MSRF) contains an Experiment Module (EM) being developed collaboratively by NASA and the European Space Agency (ESA). This NASA/ESA EM will accommodate several different removable and replaceable Module Inserts (MIs) which are installed on orbit. NASA's planned inserts include the Quench Module Insert (QMI) and the Diffusion Module Insert (DMI). The QMI is a high-gradient Bridgman-type vacuum furnace with quench capabilities used for experiments on directional solidification of metal alloys. The DMI is a vacuum Bridgman-Stockbarger-type furnace for experiments on Fickian and Soret diffusion in liquids. This paper discusses specific design features and performance capabilities of each insert. The paper also presents current prototype QMI hardware analysis and testing activities and selected results.

10:50 AM

Development of the Materials Science Research Facility and Experiment Apparatus for the International Space Station: *David Allan Schaefer*[1]; Sharon Denise Cobb[1]; Frank R. Szofran[1]; [1]National Aeronautics and Space Administration, SD44, Huntsville, AL 35812 USA

The Materials Science Research Facility (MSRF) is a modular facility designed to accommodate the current and evolving cadre of peer-reviewed materials science investigations selected to conduct research in the microgravity environment of the International Space Station (ISS). The MSRF concept consists of three Materials Science Research Racks (MSRR-1, MSRR-2, and MSRR-3) which will be developed for phased deployment into the United States Laboratory Module beginning on the third Utilization Flight (UF-3). The facility will house the materials processing apparatus and common subsystems required for operating each device, and will use the ISS Active Rack Isolation System (ARIS). Each MSRR is an autonomous rack and will be comprised of on-orbit replaceable Experiment Modules, Module Inserts, investigation unique apparatus, and/or multi-user generic processing apparatus. The MSRF will be the primary apparatus for satisfying near-term and long-range materials science discipline goals and objectives with each MSRR supporting a wide range of materials science themes in the NASA research program.

11:10 AM

Reduction of Sample Rotation in Electrostatic Levitation: *R. W. Hyers*[1]; W. L. Johnson[2]; L. Savage[1]; J. R. Rogers[1]; [1]NASA Marshall Space Flight Center, Huntsville, AL USA; [2]California Institute of Technology, Pasadena, CA USA

In many containerless processing systems, control of sample rotation is an important issue. Sample rotation is even more important for microgravity containerless processing systems, where the centrifugal acceleration can approach 1g for even a small rotation rate. Prior work on rotation control by Rhim [1] focused on driving the sample rotation at a controlled rate for droplet dynamics experiments and measurement of electrical conductivity. His technique allows controlled, fast rotation, but for many microgravity experiments the goal is zero rotation. To minimize sample rotation, two approaches are apparent: first, to identify and balance or eliminate the driving forces for undesired sample rotation, or second, implement a feedback-based rotation control loop in parallel with the position control loop. In this work, we have taken the first approach. To minimize sample rotation, the simplest approach is to identify and balance or eliminate the driving forces for undesired sample rotation. Our experiments show that the dominant driving force for rotation of machined Zr spheres in the MSFC ESL is photon pressure from the heating laser. Experimental results showing the correlation between heating power and torque are compared to theoretical predictions, and a strategy for minimizing the torque due to photon pressure is presented.

Alumina and Bauxite: Alumina Industry Trends, Products, Environment

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

Thursday AM — Room: Jefferson B
March 16, 2000 — Location: Opryland Convention Center

Session Chair: Joe Anjier, Queensland Alumina Limited, Gladstone, Queensland 4680 Australia

8:30 AM

Russia's Aluminum Industry After August 1998 Crisis: Status and Prospects: *Boris I. Arlyuk*[1]; [1]Alumconsult Limited, St. Petersburg Russia

The fortunes of the Western World's alumina and aluminium markets are today closely linked to developments affecting these industries in Russia, through imports of 2Mt/a Western alumina into Russia and aluminium exports to the West of 2.5Mt/a. This paper provides an analysis of the direct cash costs of alumina and aluminium production for all Russian refineries and smelters before and after August 1998's economic crisis. As a result of the four-fold devaluation in the rouble direct cash costs of alumina production in Russia fell to between $90 and $130/t while smelting costs were cut to between $800 and $1100/t. There is a discussion of a forecasting methodology for alumina and aluminium prices using market inputs. An analysis of recent and current market price trends and dynamics, metal stock movements, traded volumes and fundamental market balances is sufficient to develop short and medium-term price forecasts for aluminium and alumina. However, forecasting models based on the usual statistical methods for analysing these market parameters do not give good results in view of the lack of timely and accurate data for some of the necessary inputs and the numerous empirical coefficients that must be utilised. Therefore, a model has been developed which reduces the aims and behaviour of market participants, producers, consumers and traders, to mathematical functions. It is clear that market fundamentals, the balance between supply and demand, stock levels held both in terminal market warehouses and by producers and consumers, and price trends provide a good basis for developing longer-term price forecasts. In the short term, however, speculative factors play a significant role, seen especially in the activity of major market participants. The accuracy of the price model has been evaluated. For short-term forecasts (2 days to one week) the error is around 30% of cases forecasted prices direction while for medium-term forecasts (one month to one quarter) show an error of 20%. Error of long-term price forecast depends on the accuracy of forecasted market balance. Prospects for existing plants and potential new projects are reviewed in the light of forecast

world market prices for alumina and aluminium. This forms the background to a near-term outlook for Russian alumina import requirements and availability of aluminium metal for export to the West.

8:55 AM

Bauxite Mine Reclamation Throughout the World: *Patrick R. Atkins*[1]; [1]Alcoa, 201 Isabella St., Pittsburgh, PA 15212 USA

The International Primary Aluminium Institute (IPAI) conducted a worldwide survey in 1991 to gather information on the technologies, monitoring practices and resources used throughout the industry to rehabilitate bauxite mines. The survey also gathered data on the rate and timing of the rehabilitation activities. Responses were received from over 65% of the world's producers by tonnage. A report, "Bauxite Mine Rehabilitation Survey," was published in 1992. A second worldwide survey has been completed and data will be used to develop an updated IPAI report on the status of bauxite mine rehabilitation throughout the world. The 1998 survey contains responses from over 70% of the world's producers by tonnage. This paper will compare results from the first mine reclamation survey with the information received in 1998 to highlight areas of significant progress and areas where technology transfer can provide assistance to the bauxite industry and perhaps other mining enterprises.

9:20 AM

A Study of the Effect of Hydrate on Properties of Metallurgical Grade Aluminas: *Miguel Llavona*[1]; [1]University of Oviedo, Dept. of Matls. Sci., Reinero Garcia s/n, Mieres 33600 Spain

In industrial aluminas, certain segmentation is produced due to the difference in the size, form and density of the particles. The fine particles contain more residual hydrate, alpha alumina and their microporosity is higher; however, the specific area is smaller. If the moisture of the aluminas is determines to 110°C, the MOI to 300°C and the LOI to 1200°C, according to the standard tests, and the alumina contains hydrate, the values of the MOI and LOI will be seen altered and they will not have meaning. The loss of weight to 500°C has been related with the content in hydrate in the industrial aluminas. It is important to determine the moisture of the aluminas to 110°C, the content in hydrate to 500°C-MOI-and the LOI to 1200°C.

9:45 AM

Aluminum Hydroxide with High Thermal Stability and Flame Resistance: *Qingwei Wang*[1]; [1]Zhengzhou Light Metals Research Institute, Shangjie District, ZhengZhou, Henan 450041 PRC

The surface of common aluminum hydroxide powder was modified by multilayer wrappings. The cooperating flame resisting group was grafted into the surface active agent. The affinity of the aluminum hydroxide for polymner, the flame resistance and the thermal stability are improved markedly. Thus it can have application widely in plastics and rubber with high temperature processing.

10:10 AM Break

10:30 AM

Improvement in Reactivity of Bayer Process Alumina Powders: Akira Sakamoto[1]; Susumu Shibusawa[1]; *Eiji Kanbara*[1]; [1]Showa Denko K.K., Yokohama Works, 8 Ebisu-cho, Kanagawa-ku, Yokohama, Kanagawa 221 Japan

Due to its excellent properties (resistance to heat/corrosion/abrasion, electrical insulation and thermal conductivity), alumina has been used as material for such sintered bodies as IC substrates, cutting tools and abrasion-resistant parts. Alumina for such applications should have small alpha-ultimate crystal size and be sinterable at low temperatures, and should result in sintered bodies with excellent mechanical strength, hardness and abrasion resistance. So far, thermal decomposition of high-purity aluminum salt has been a general way of producing ultra fine alpha-alumina particles, which method involves a disadvantage of high production cost. Established, through application and development of Bayer reactive alumina powder technology, is a method of producing alpha-alumina with fine particle size and purity of four nines.

10:55 AM

Removal of Nickel from Aqueous Solution Using Activated Red Mud: *J. Pradhan*[1]; S. N. Das[1]; R. S. Thakur[1]; [1]Regional Research Laboratory, Bhubaneswar 751013 India

Red mud, a colosal solid waster from bauxite processing, was treated by simple dissolution in hydrochloric acid and re-precipitation by ammonia to form activated red mud (ARM). This material was used to remove nickel (II) from aqueous solution. Various paramenters like pH, contact time, temperature, red mud to metal ion ratio, were determined. More than 60% removal of nickel (II) was achieved for an initial concentration of 10 ppm. Removal of metal ion increases by raising pH of the medium up to 6.5 but thereafter drops due to precipitation of metal hydroxides. Increase in rate of absorption was observed with rise in temperature. The loading capacity of ARM increased with higher Ni (II) removal at 10 ppm initial concentration was observed for an ARM concentration of 20g/L. The experimental data agreed well with Freundlich and Langmuir adsorption isotherms. Adsorption studies were extended to removal of heavy toxic metal ions from aqueous industrial effluents of electroplating and chromite mining industries.

11:20 AM

Characteristics of Red Mud Generated at NALCO Refinery, Damanjodi, India: *B. K. Mohapatra*[1]; [1]Regional Research Laboratory, Bhubaneswar 751013 India

Red mud generated at the NALCO Alumina Refinery in Damanjodi, India, has been characterized with respect to particle size, settling properties, mineralogy, mineral chemistry, and the reason for loss of some alumina value in the rejects has been establishes. Red mud comprising fine solid particles (average 5) is alkaline, poor in settling, and has 13-16m2/g specific surface area. Its mineral constituents are hematite, gibbsite, geothite, boehmite, rutile/ilmenite grains, specks of lithoretics like kaolinite, sillimanite, etc., and minor sodalite. Electron probe analysis on selective particles indicated the presence of up to 64-mole% of boehmite coexisting with gibbsite crystals and up to 18% of alumina in goethite. Red mud samples generated at different stages of the refinery, such as Digested Mud (DM), Deslicated Mud (DSM), Stlleter Mud (SM), Washer Mud (WM), and Mud to Pond (MTP) are more or less similar in their physical and mineralogical properties, but exhibit minor but distinguishable differences in their chemical characteristics. In the alumina refinery, some phases like dehydroxylated gibbsite, boehmite, alumo-goethite, Al-rich lithorelics, etc., do not dissolve during the industrial treatment of bauxite and get released as constituents of red mud and thereby reduce the recovery of alumina significantly.

Aluminum Reduction Technology: Cell Operation/Electrodes

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

Thursday AM — Room: Sewanee
March 16, 2000 — Location: Opryland Convention Center

Session Chair: Pierre Homsi, Aluminum Pechiney, Saint-Jean-de-Maurienne 73303 France

8:30 AM Invited

Graphite Cathode Wear Study at Alouette: *Pierre Reny*[1]; Sigfried Wilkening[2]; [1]Aluminerie Alouette, Inc., 400 Chemin de la Pointe-Noire, P.O. Box 1650, Sept-Iles, Quebec G4R5M9 Canada; [2]VAW Aluminium-Technologie GmbH, P.O. Box 2468, Bonn 53014 Germany

Between 1996 and 1999, graphitized cathodes were installed in the 264 AP- 30 pots at Aluminerie Alouette, Inc. aluminium smelter. During this period, an extensive cathode erosion study was undertaken with the primary objective of predicting potlife. A method to accu-

rately measure cathode erosion on an operating pot was developed. This method uses a surveyor's laser level equipment and is detailed in the paper along with measurements accuracy estimates. The study is still ongoing but the analysis of the preliminary results show evidence that mechanical erosion, at the tapping hole or elsewhere in the pot, is not the leading erosion mechanism. The large scale erosion pattern suggests a current density-driven phenomenon, which is in agreement with electrical models. Fixed interval measurements of selected cathodes also show that the erosion speed does not appear to vary with pot age, or potline amperage evolution as was the case at Alouette. The accuracy of this analysis method is such that it enables identification of areas where spalling of the cathode occurred at start-up.

9:00 AM

Ramming Paste Properties and Cell Performance: *Frank Hiltmann*[1]; Karl Heinz Meulemann[2]; [1]SGL Carbon GmbH, Griesheim Plant, Stroofstrasse 27, Frankfurt D-65933 Germany; [2]Alusuisse Technology & Management Limited, CH-3965 Switzerland

The importance of ramming paste is often underestimated for the performance of aluminum electrolysis cells. Four commercially available ramming pastes have been lab-tested for ramming behaviour, green and baked properties, expansion/shrinkage characteristics during heat-up, alkali resistance, and mechanical strength at elevated temperatures. The results are discussed with respect to the specific changes in a cell during start-up, with special focus on glued cathode blocks, and correlated to smelter results: a proper paste should exhibit a smooth expansion until binder carbonisation which should take place only late in the heat-up cycle and stay dimensionally stable afterwards until reaching cell operating temperature. When selecting a paste the particular cell technology and start-up conditions must be taken into consideration.

9:25 AM

Effect of Multiple Restarts on Cell Life: *Paul Desclaux*[1]; [1]Alcan International Limited, Reduction Tech. Svc., 1955 Mellon Blvd., P.O. Box 1250, Jonquière G7S4K8 Canada

It is a known fact that premature shut downs and restarts because of economic conditions or emergency situations do affect negatively potlinning life. Many plant data from various smelters were analyzed using a special statistical method based on the concept of risk and limit product estimate to evaluate the relation between the number of restarts and cell life. This method appears to be very well adapted to this precise situation where many cells from the plant population are still in operation. This analysis indicated clearly the negative and additive effect of each restart on the cell life average. Calculation of the mean residual lifetime for a given cell may support decisions on strategic cell relining scheduling.

9:50 AM

Cryolite Penetration Studies on Barrier Refractories for Aluminium Electrolytic Cells: *Don Harris*[1]; George Oprea[2]; [1]Clayburn Refractories Limited, 33765 Pine St., Abbotsford, British Columbia V2S5CI Canada; [2]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, British Columbia V6T1Z4 Canada

In order to assess the behavior of a dense barrier refractory towards cryolite penetration, two main parameters, open porosity and gas permeability, were considered and studied on various bricks, castables and dry granular materials. The penetration and corrosion results were correlated with the testing parameters, the cryolitic bath chemistry and the refractories' microstructure. The mechanisms of corrosion were discussed, particularly emphasizing the reactivity of different mineralogical components of the refractory materials, when tested in oxidizing and reducing atmospheres.

10:15 AM Break

10:25 AM

Thermal Conductivity Measurements of Cathode Insulation Materials: *Flemming Bay Andersen*[1]; Jørgen Mikkelsen[1]; [1]Skamol A/S, Rsrch. and Dev., Ostergade 60, Nykobing, Mors DK-7900 Denmark

Cathode insulation in aluminium electrolysis cells plays a very important role in the pot design even though only a small amount of the total heat is dissipated through the cathode bottom. The amount of insulation determines the temperature gradients in the cathode and the choice of insulation can have a large influence on pot life. The thermal conductivity is the key property of insulation and a range of materials have been examined. The so-called Hot Disk Method has been used to measure the thermal conductivity of several kinds of insulation material with different properties: Calcium silicate and vermiculite slabs, diatomacous earth and perlite bricks and finally insulating firebricks of aluminosilicate. The thermal conductivity dependence on density, temperature and anisotropy of the microstructure is examined and discussed.

10:50 AM

Anode Improvements at Alcan Brazil's HSS Potlines: *Rui Oyama Homma*[1]; [1]Alcan Aluminio do Brasil Ltda, Reduction Area, Av. Am'erico Rene Gianetti 521, 35.400-000-Saramenha, Ouro Preto, Minas Gerais, Brazil

During the last 20 years at the Alcan Brazil's HSS potlines, as in many other smelters, several efforts and process developments have been made aiming at power and raw materials consumption factors improvement. The anode plays an important role in these consumption factors, and the anode quality and its operation are the basic elements for these improvements. The anode quality affects not only paste consumption, but the overall cell operational performance, and it is usually a function of raw materials quality (pitch and coke), paste formulation and paste production process. Besides that, the anode performance is also related to the cell (anode and cathode) operational procedures. This paper describes all the improvements implemented in the paste and anode operation over the last 20 years, at the Alcan Brazil's HSS potlines, which led to a reduction of around 50 kg/mt in paste consumption. The improvements on cell operations are also discussed.

11:15 AM

The Properties of Si_3N_4-Bonded SiC Material for Aluminium Electrolysis Cell: *Junguo Zhao*[1]; Zhiping Zhang[1]; Wenwu Wang[1]; Guohua Liu[1]; Z. Cheng[1]; [1]Luoyang Institute of Refractory Research, 43 Xiyuan Rd., Luoyang, Henan 471039 China

The seam of side-wall lining in aluminum electrolysis cell has lower corrosion resistance than that of Si3N4-bonded SiC brick. In order to service performance of side-wall lining, reducing the number of seams is one of the effective approaches by increasing the size of Si3N4-bonded SiC brick. Therefore, in this the paper, attention is focused on fabricating Si3N4-bonded SiC bricks with different sizes and weights by adjusting processing parameters, and on investigating the relationship of the size and weight with mechanical properties. Helpful information is provided based on experimental results to design a side-wall lining by using large size Si3N4-bonded SiC bricks as possible without sacrificing service performance.

11:40 AM

Aliminium Wettable Cathodes: An Update: *Rudolf P. Pawlek*[1]; [1]Technical Information Services and Consulting, Le Forum des Alpes, Avenue du Rothorn 14, Sierre CH-3960 Switzerland

During the last few years, the development of aluminium wettable cathodes has advanced considerably. Coating methods tried in laboratory and plant tests include titanium diboride electrode position, titanium diboride coating with reinforced fibres, titanium diboride plasma spray, titanium diboride carbon/graphite composites and colloidal alumina-bonded titanium diboride slurry. Techniques and results are reviewed.

Cast Shop Technology: Grain Refinement

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

Thursday AM — Room: Mississippi
March 16, 2000 — Location: Opryland Convention Center

Session Chair: Barbara L. Kidwell, Alcoa Warrick Operations, Rigid Packaging Division, Newburgh, IN 47630 USA, G. W. Boone, KB Alloys, Corp. Tech. Ctr., Robards, KY 42452 USA

8:30 AM Introductory Remarks

8:35 AM
Design of Grain Refiners for Aluminum Alloys–How to Improve Efficiency: *Arnaud Tronche*[1]; A. Lindsay Greer[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metallu., Pembroke St., Cambridge CB23QZ UK

The efficiency of a grain refiner can be quantified as the number of grains per nucleant particle in the solidified product. Even for very effective refiners in aluminium, such as Al-5Ti-1B, it is known from experiment that efficiencies are very low, at best 10^{-3} to 10^{-2}. It is of interest to explore the reasons for such low values, and to assess the prospects for increased efficiency though design of refiners. Recently it has been shown [1] that a simple recalescence-based model can make quantitative predictions of grain size as a function of refiner addition level, cooling rate and solute content. In the model, the initiation of grains is limited by the free growth from nucleant particles, the size distribution of which is very important. The present work uses this model as the basis for discussing the effects of particle size distribution on refiner performance. Larger particles (of TiB_2 in the case of present interest) promote greater efficiency, as do narrower size distributions. It is shown that even if the size distribution could be exactly specified, compromises would have to be made to balance efficiency (defined as above) with the required addition level of refiner. [1] A.M. Bunn, P.V. Evans, D.J. Bristow and A.L. Greer, in 'Light Metals 1998', edited by B. Welch (TMS, Warrendale PA, 1998) pp. 963-968.

9:00 AM
Nucleation Mechanisms of TiBAl Additions in Al-Ni-Si Alloys: *Peter Schumacher*[1]; Brian J. McKay[1]; Pavel Cizek[1]; Keyna Q.A. O'Reilly[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX13PH UK

Grain refiner additions in Al-Si in conventional casting practice require higher amounts of excess Ti than in wrought aluminum alloys, however, beyond 3 wt.% Si the grain refiner efficiency is greatly reduced. The effects of Si on the nucleation mechanism of TiBAl (Al-5wt.% Ti-1wt.%B) are unknown. A novel metallic glass technique permits the addition of refiner particles into a melt of Al70Ni13Si17 (at.%) which higher solute content facilitate glass formation on rapid cooling. Nucleation of Al occurs in the undercooled melt while growth is effectively halted at the glass transition temperature resulting in discrete nucleation and growth centres suitable for TEM investigations. Three types of nucleation centres have been identified in the as quenched structure: hexagonal TiB2 platelets, dendritic aluminides and hexagonal devitrification products. Interestingly at high Si levels the borides appear not to be covered in Al3Ti as in previous studies without Si addition and do not nucleate Al on basal faces while the dendritic and devitrification phase nucleate heterogeneously copious Al crystals. This suggests that the excess-Ti has been consumed within the melt and cannot act as stabilised Al3Ti layer on borides. However, a new phase is nucleated epitaxial at non-based faces of the boride indicating that crystallographic matching is an important factor for successful nucleation.

9:25 AM
Poisoning of Ti-B-Al Refiner Rod Additions in Al Melts Containing Zr: *Peter Schumacher*[1]; Pavel Cizek[1]; Alice Bunn[2]; Lindsay Greer[2]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX13PH UK; [2]University of Cambridge, Dept. of Matls. Sci. and Metallu., Pembroke St., Cambridge CB23QZ UK

Grain refiner additions containing TiB2 and Al3Ti particles were successfully added to Al-Ni-Zr melts which upon rapid cooling form glasses. In the temperature region of the undercooled melt nucleation of aluminium occurred and subsequent growth of Al was halted at the glass transition temperature resulting in nucleation and growth centres separated by an amorphous matrix. Similar to earlier observations, in which no Zr was present, a layer of the type Al3Ti adsorbed on basal faces of hexagonal TiB2 particles acted as a potent nucleation substrate. Poisoning was found to be a thermally activated process observed on prolonged exposure to Zr. Zr affected firstly adsorbed aluminide layers and then borides. The adsorbed layer of Al3Ti can be replaced by Al3Zr having a lower peritectic temperature than Al3Ti and hence lower potency. At higher processing temperatures it was found that TiB2 transformed to ZrB2 affecting the delicate epitaxial relationship between boride, aluminide and Al believed to enhance the potency of the aluminide-covered borides. The findings are discussed and found to be consistent with respect to conventional casting practice.

9:50 AM Break

10:00 AM
Strobloy–The New Combined Grain Refiner and Modifier for Hypoeutectic AlSi Foundary Alloys: *Eivind Bondhus*[1]; Trond Sagstad[1]; [1]Hydelko KS, Saheimsveien, Rjukan 3660 Norway

Hydelko presents a new combination alloy for use in hypoeuctectic aluminium silicon foundry alloys. Strobloy simplifies today's addition practice of grain refiner and modifier by reducing the number of additions from two to one. Strobloy is a combination product between the well-established TiBloy, and strontium. The alloy contains nucleating particles in the form of the mixed boride (Al, Ti)B2, which is beneficial regarding settling and grain refining efficiency. Strontium is present as fast-dissolving Al4Sr particles. Trials carried out indicate that Strobloy shows very good performance compared to separate additions of TiBloy/AlTi5B1 and AlSr master alloy. The grain refining efficiency and modification level is the same or better. This paper displays results collected from tests done with different strontium levels in an A356 alloy. Comparisons to separate additions of TiBloy and AlSr are presented.

10:25 AM
Structural Refinement of Hypoeutectic Al-Si Alloys by Electromagnetic Vibrations: *Alireza Radjai*[1]; Kenji Miwa[2]; [1]Japan Science and Technology Corporation, National Industrial Rsch. Insti. of Nagoya, Matls. Processing Dept., 1-1 Hirate-cho Kita-ku, Nagoya 462 Japan; [2]National Industrial Research Institute of Nagoya, Materials Processing Dept., 1-1 Hirate-Cho, Kita-ku, Nagoya 462 Japan

Simultaneous imposition of alternating electric and stationary magnetic fields on a conducting liquid will induce a vibrating motion in the liquid which can lead into the formation and collapse of cavities in the liquid and affect the solidification structure. This phenomenon and the effects of the two main parameters of frequency and intensity of vibrations have been studied in an Al-7% Si alloy. Based on a superconducting magnet, an experimental apparatus that enables the simultaneous application of an alternating electric field with a frequency of up to 50 kHz and a magnetic field of up to 10 T has been designed and assembled. The thorough investigation, which has been carried out over wide ranges of intensity (an electromagnetic pressure range of (0 to 2.25 X 10⁵ Pa) and frequency (0 to 50 kHz), clarified the effects of the two main parameters on the structural refinement brought about by electromagnetic vibrations. Microscopic observations have shown that the cavitation phenomenon, being a main factor behind the structural refinement, has been effective over a specific

range of frequency and only when the magnetic pressure has exceeded a specific value. The effects of mechanical vibrations of the experimental apparatus have been also investigated and found to have no contribution to the structural refinement observed.

10:50 AM

A Study of TiCAl 315® Grain Refinement in Roll Cast Aluminium Alloys: *Ming Yun*[1]; S. A. Lockyer[1]; J. D. Hunt[1]; R. Cook[2]; D. J. Bristow[2]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, England OX13PH UK; [2]London & Scandinavian Metallurgical Company Limited, Rotherham, England UK

A new carbon containing grain refiner, TiCAl 315® produced by London & Scandinavian Metallurgical Co Limited, has been used in roll cast aluminium alloys. The effect of the grain refiner on as-cast grain structure has been investigated, in conjunction with variations in strip thickness, roll casting speed, superheat of the melt and addition level of Ti. A conventional grain refiner, 5/1 TiBAl, has also been used in the roll casts for comparison. The results of the study show that TiCAl 315® is equally efficient in terms of grain refining ability compared with 5/1 TiBAl at equivalent Ti additions levels. However, the as-cast grain structure refined by TiCAl 315® rods is better than that produced using 5/1 TiBAl at higher roll casting speeds and thinner cast gauges.

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

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

Thursday AM — Room: Canal A
March 16, 2000 — Location: Opryland Convention Center

Session Chairs: J. K. Shang, University of Illinois, Dept. of Matls. Sci. & Eng., Urbana, IL 61801 USA; L. Llanes, Universitat Politecnica de Catalunya, Dept. de Ciencia dels Materials i Enginyeria Metallurgica, Barcelona E-08028 Spain

8:30 AM

Cyclic Deformation and Fatigue Fracture of Tungsten Monofilament-Reinforced Multicrystalline Copper Composites: *Jieping Zhang*[1]; Campbell Laird[2]; [1]Intel Corporation, 5000 W. Chandler Blvd., Chandler, AZ 85226 USA; [2]University of Pennsylvania, Dept. of Matls. Sci. and Eng., Philadelphia, PA 19104-6272 USA

Studies on cyclic deformation and fatigue fracture behavior have been conducted on tungsten monofilament reinforced multicrystalline copper composites. The paper introduces a simple model to link the cyclic stress-strain response of the multicrystalline composites to those of monolithic single crystals and fibers. This model not only represents the fiber reinforcement by the rule of mixtures, but also adopts Sachs model for the single crystal-polycrystal conversion factor. The results calculated by the model show very good agreement with the experimental data in all strain amplitudes at which the composites were fatigued. This encouraging outcome suggest that the new model could be applied to high-cycle fatigue of commercial continuous-fiber-reinforced polycrystalline metal matrix composites. The fatigue fracture is very sensitive to a fiber break, and to microstructural features, such as grain boundaries and dislocation structures. Once the fiber breaks into two segments, the composite will fail in a short period afterwards. The fatigue fracture mechanism of the composites highly depends on applied plastic strain amplitudes. Most cracks initiate at grain boundaries in the matrix at low and intermediate plastic strain amplitudes and the fiber greatly improves the fatigue behavior. At a high plastic strain amplitude, fatigue cracks initiate at the fiber, and the fiber seems ineffective for improving the fatigue life.

8:55 AM

Fatigue Properties of SiC/Ti-15-3 MMC: *C. Masuda*[1]; Y. Tanaka[1]; Y. -F. Liu[1]; [1]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba, Ibaraki, Japan

Silicon-carbide (SCS-6) fiber reinforced titanium alloy matrix composites are attractive for structural applications such as gas turbine engines, because of their high specific modulus and strength, and good stability at high temperature. But one major problem still remains: reaction layer occurs between fibers and matrix during processing and degrades fibers properties. Moreover the thickness of this layer has a strong effect on global properties loss. Finally we aim to develop new composites to reduce this effect. But before that, we must able to explain the effect of interfacial damage on global mechanical properties. So far, fatigue tests were conducted at room temperature was higher than that tested at high temperature. But the fatigue data tested at high temperature in vacuum is higher than that tested at high temperature. But the fatigue data tested at high temperature in vacuum is higher than that tested at room temperature. At room temperature damage evolution process as follows: crack initiation at the reaction layer, interfacial debonding between reaction layer and outer carbon layer, cracking the outer carbon layer, interfacial debonding between outer carbon layer and SiC fiber, SiC fiber breakage, and matrix cracking. At high temperature in vacuum, damage evolution was nearly the same. However, for the same level of stress, fatigue life was longer at high temperature, probably because debonding appeared more important. Those results show how important the interface region is in damage initiation and propagation. That why, in addition to fatigue tests, theoretical investigation has been conducted. At high temperature in air, damage evolution was nearly the same, but the outermost carbon coating layer of fiber and matrix alloy were oxidized and the interface bonding stress will be reduced and the matrix will be more brittle.

9:20 AM

The Low-Cycle Fatigue Behavior and Microstructural Evolution of Haynes® HR-120® Alloy: *P. K. Liaw*[1]; Y. H. He[1]; M. Huang[1]; L. Miller[1]; C. R. Brooks[1]; R. R. Seeley[2]; D. L. Klarstrom[2]; [1]The University of Tennessee, Dept. of Matls. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., P.O. Box 9103, Kokomo, IN 46904-9013 USA

The low-cycle fatigue behavior of HAYNES® HR-120® alloy was studied at room temperature, 75°C and elevated temperatures (1200°F-1800°). Test results indicated that the test temperature and strain range significantly affected the mechanical behavior and fatigue failure of the alloy. The alloy was observed to cyclically harden at the moderately high temperatures of 1200°F and 1600°F, but to cyclically soften at the temperatures of 1800°F and 75°F. The analyses of microstructures by means of optical microscopy and transmission electron microscopy (TEM) indicated that the mechanical performance of the alloy at high temperatures was greatly influenced by the precipitation of second phase particles. Since the kinetics of precipitation was rapid at 1600°F, the alloy exhibited noticeable cyclical hardening at all of the strain ranges evaluated. At 1200°F, the alloy cyclically hardened at a high rate and then reached a plateau. However, at 1800°F, the alloy exhibited cyclic softening in most cases, which resulted from the coarsening of the second phase particles. The reason that the alloy exhibited cyclic softening at room temperature is believed to be due to the presence of residual coldwork resulting from the final flattening

operations of the plate. It was observed from the SEM examination of the fracture surfaces that the number of crack initiation sites varied with temperature and strain range. At the highest temperatures, oxidation was found to play an important role in crack initiation. This work is supported by the Haynes International Ind. We also acknowledge the financial support of the National Science Foundation, the Division of Design, Manufacture, and Industrial Innovation, under Grant No. DMI-9724476, and the Combined Research-Curriculum Development Program, under EEC-9527527, to the University of Tennessee, Knoxville, with Dr. Delcie R. Durham and Ms. Mary Poats as program managers, respectively. We appreciate the financial support of the Center for Materials Processing and Office of Research Administration with Drs. C. McHargue and K. Walker as directors, respectively, at the University of Tennessee.

9:45 AM

Cumulative Fatigue Damage of 310 and 316 Stainless Steels Evaluated by AFM: *Jeffrey L. Evans*[1]; William W. Gerberich[1]; [1]University of Minnesota, Chem. Eng. and Matls. Sci., 151 Amundson Hall, 421 Washington Ave. SE, Minneapolis, MN 55455 USA

Surface damage, due to reversed bending fatigue cycling, was evaluated as a function of the number of fatigue cycles in the low-cycle fatigue regime for a 310 stainless steel and a 316 stainless steel. Using an atomic force microscope (AFM), surface displacements were investigated. These displacements were a result of slip band formation and subsequent extrusions and intrusions occurring at the free surface. The cumulative surface damage of the 316 stainless steel, a relatively stable alloy, was compared with that of the 310 stainless steel, a very stable alloy, to determine what effect, if any, of strain-induced martensite or stacking fault energy on the surface displacements. When predicting fatigue crack initiation, the amount of surface displacement due to slip band evolution is critical, therefore previous models of a microstructurally-based Manson-Coffin law of low cycle fatigue were tested.

10:10 AM Break

10:35 AM

Cyclic Electric Field-Driven Crack Growth in Ferroelectric Ceramics: *Xiaoli Tan*[1]; Jian Ku Shang[1]; [1]University Of Illinois at Urbana-Champaign, Dept. of Matls. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA

Ferroelectric ceramics are used in number of electric, electromechanical and electrooptic applications. The inherent electromechanical hysteresis in these ceramics make them highly susceptible to fatigue. The fatigue degradation may result from purely mechanical, purely electric or combined loading. In this study, fatigue crack growth driven by pure electric loading was examined in a lead zirconia titanate and a La-doped lead zirconia titanate. Crack growth behavior was found to depend on direction of the applied electric field, field strength, the field ratio, frequency, temperature, and size of the hysteresis loop. Analysis of the experimental results using the current fracture mechanics principles will be presented and comparison will be made between theoretical predictions and experimental observations.

11:00 AM

Low-Cycle Fatigue Behavior of a Precipitation-Hardening Stainless Steel: *Chih Kuang Lin*[1]; Chi Chih Chu[1]; [1]National Central University, Dept. of Mech. Eng., Chung-Li 32054 Taiwan

This study investigated the effects of mean stress on the low-cycle fatigue (LCF) behavior of a martensitic precipitation-hardening stainless steel in different tempers. Uniaxial LCF tests were conducted under strain control with three strain ratios R=-1, 0 and 0.5. LCF specimens were prepared in three different tempers, namely solution-annealed (SA), peak-aged (H900), and overaged (H1150) conditions. The effects of aging treatment on the LCF behavior are also discussed. Experimental results show that LCF specimens in these three tempers all exhibited cyclic softening at high strain amplitudes under a strain ratio of R=-1. At low strain amplitudes, the cyclic softening is less evident for SA and H900 temper while H1150 temper exhibits cyclic hardening. Under a strain ratio of R=-1, specimens in H900 temper had longer LCF life than those in SA and H1150 tempers. However, this advantage for H900 over SA and H1150 tempers disappeared at higher strain ratios (R=0 and 0.5) due to the greater sensitivity to mean stress effect in H900 temper. For a given temper at high strain amplitudes, the LCF lives among the three applied strain ratios did not show significant differences as a result of the mean stress relaxation effect. However, at low strain amplitudes, cyclic loading at R=-1 generated the lowest mean stress levels and longest LCF lives as compared to R=0 and 0.5. The LCF life data obtained for this precipitation-hardening stainless steel under various combinations of heat treatments and strain ratios could be well correlated by two proposed life-assessment approaches.

11:25 AM

High Frequency Metal Fatigue: The High-Cycle Fatigue Behavior of ULTIMET® Alloy: *L. Jiang*[1]; C. R. Brooks[1]; P. K. Liaw[1]; D. L. Klarstrom[2]; [1]The University of Tennessee, Dept. of Matls. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA

ULTIMET® alloy is a newly developed commercial Co-26Cr-9Ni (wt.%) alloy, which exhibits good resistance to both wear and corrosion. A state-of-the-art high-frequency fatigue testing system was used to study the high-cycle fatigue behavior of ULTIMET® alloy between 10^5 and 10^9 cycles. The advantage of a high-frequency fatigue testing system is the reduction in testing time, so that a great number of cycles can be obtained in a short time. Fatigue behavior of ULTIMET® alloy was investigated at a high frequency of 1000 Hz and conventional frequency of 20 Hz. The effect of high strain rate on fatigue crack initiation and propagation mechanisms was studied. The fractography studies showed that the main features of the fracture surface were similar for both high and conventional-frequency fatigue specimens. There were some differences on the S-N curve developed at both 1000 and 20 Hz. The variations between the high and conventional-frequency fatigue mechanisms were elucidated by the surface microscopy. Most of the fatigue fracture surfaces of ULTIMET® alloy indicated that the crack generally initiated on the specimen surfaces, and had a crystallographic appearance regardless the frequency. Research supported by Haynes International, Inc. and National Science Foundation (DMI-9724476 and EEC-9527527 with Dr. D. Durham and Ms. M. Poats as contract monitors, respectively). Note that ULTIMET® is a registered trademark of Haynes International, Inc.

11:50 AM

On the Fatigue Behavior of Duplex Stainless Steels: *L. Llanes*[1]; A. Mateo[1]; A. Girones[1]; M. T. Farre[1]; N. Salan[1]; M. Anglada[1]; [1]Universitat Politecnica de Catalunya, Dept. de Ciencia dels Materials i Enginyeria Metallurgica, ETSEIB, Av. Diagonal 647, Barcelona E-08028 Spain

Interest on the fatigue performance of duplex ferrite-austenite stainless steels (DSSs) has expanded significantly in recent years. This is primarily due to the increasing use of these materials within structural applications that involve cyclic loading. Hence, extensive fatigue testing and analyses, approaching both fundamental and service-related viewpoints, have been developed by several excellent research groups along the last fifteen years. The objective of this contribution is therefore to critically review such existing knowledge as well as to point out immediate and long-term needed information for continuous structural improvement involving DSSs to occur. In doing so, aspects dealing with each and every one of the fatigue stages of these materials are addressed: cyclic stress-strain response and its correlation to substructural evolution; influence of environment (gaseous atmosphere/aqueous media) and thermal aging on strain localization, crack nucleation and early growth, propagation of long cracks and number of cycles to failure; anisotropic behavior as related to given processing routes (induced microstructural and crystallographic texture); and fatigue endurance criteria, for both low- and high-strain amplitude regimes, in terms of the surface damage resulting from the corresponding mechanical coupling of the constitutive phases. Although the review is mainly driven by own results, in all cases attempts are made to consider reports found in the literature for related duplex and single-phase austenitic and ferritic steels.

12:15 PM

Fatigue Behavior of Interstitial Free Steels with Deep Draw Strains: *Benda Yan*[1]; [1]Ispat Inland, Inc., 3001 E. Columbus Dr., East Chicago, IN 46312 USA

It is well known that interstitial free (IF) steels are susceptible to Secondary Work Embrittlement (SWE) due to its lacking of interstitials, such as carbon and nitrogen. The SWE susceptability may cause cracking failure of deep drawn parts under impact loading at winter temperatures when the ductile brittle transition temperature (DBTT) is high. Since IF steels have been widely used in automotive industry and many automotive body structures and even body panels are subjected to cyclic loading, the effect of SWE susceptibility on the fatigue performance of the IF steels has been a serious concern. In this study, two IF steels and a non-IF steel were tested to study the effect of SWE susceptibility on the fatigue performance. One of the IF steels exhibited a very high DBTT, 20°C, whereas the other exhibited much lower DBTT, -40. The non-IF steel, DQSK, is not SWE susceptible with a DBTT of less than -60°C. Specimens with deep drawn strains were tested under bending fatigue conditions. Limited test results show that even with deep drawnstrains, the fatigue lives of the IF steels and non-IF steel are similar despite their significantly different SWE susceptibility. The fatigue fracture behavior of the steels tested is also discussed in detail.

High Temperature Processes for Waste Treatment & Minimization: II

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

Thursday AM — Room: Jackson A/B
March 16, 2000 — Location: Opryland Convention Center

Session Chairs: William K. O'Connor, Albany Research Center, Office of Fossil Energy, Albany, OR 97321 USA; Stephen Fox, TIMET, Henderson Technical Lab., Henderson, NV 89015 USA

8:30 AM

Pyrometallurgical Extraction of Alumina and Iron from Red Mud: *Brajendra Mishra*[1]; David Kirkpatrick[2]; [1]Colorado School of Mines, Metallu. & Matls. Eng., 1500 Illinois St., Golden, CO 80401 USA; [2]Kaiser Aluminum & Chemical Corporation, P.O. Box 3370, Gramercy, LA 70052 USA

The major waste product of the alkaline extraction of alumina from bauxite [Bayer Process] is known as red-mud. Approximately, a ton of red-mud is produced for every two tons of bauxite mined. The red-mud produced from Jamaican bauxite is rich in hematite, alumina and titanium oxide. It has been shown that over 90 wt. pct. alumina can be recovered from red-mud by soda-ash sintering and caustic leaching. Hematite can be carbothermically reduced with a degree of metallization of over 94 pct. At this stage, the product could be charged through the tuyeres in an iron blast furnace or smelted to produce pig iron. If smelted, the concentration of titanium oxide in the slag will be significantly high justifying its recovery by an acid-leach process. This presentation will describe the successful efforts of iron and alumina recovery and the plans for titanium oxide recovery from red-mud. The problems associated with the use of reduced red-mud as an alternative to direct-reduced iron [DRI] will also be discussed. Critical assessment of the recovery sequence chosen for the products will be described based on economics.

9:00 AM

Characterisation and Thermal Treatment of Fly-Ash from a MSW Incineration Plant: *Célia Maria Ferreira*[1]; Manuel Fonseca Almeida[2]; [1]Escola Superior Agrária de Coimbra, Dep. Ciencias Exactas e do Ambiente, Bencanta, Coimbra 3040-316 Portugal; [2]Faculdade de Engenharia, Demm, Rua Dos Bragas, Porto, Cedex 4099 Portugal

Fly-ashes from Municipal Solid Waste Incineration Plants are considered hazardous waste due to its high concentration of soluble heavy metals, chlorides and sometimes minor quantities of organic compounds. Thus, detoxification accomplished by removing these contaminants can be viewed as an important step of its treatment. In this paper a sample of fly-ashes from a semi-dry off-gas treatment system is characterised, namely by using NEN-7343 leaching protocol. Also, results of contaminants removal from this residue by thermal treatment under different conditions are also presented.

9:30 AM

Laboratory Research for High Temperature Vitrification: *Patrick R. Taylor*[1]; [1]University of Idaho, Metallu. Eng. Dept., McClure Hall, Moscow, ID 83844-3024 USA

Glass is used for immobilization of radioactive and hazardous wastes due to its durability and ability to accept a variety of waste constituents into its network structure. Alternative high temperature vitrification techniques have the advantage of increased throughput rate, increased waste loading and being applicable to variable and heterogeneous waste streams. However, the increased processing temperature places further concerns on the increased volatilization of contaminants, and increased corrosion and erosion of melter components. Database of melt properties at higher temperatures, such as partitioning, viscosity, density and electrical conductivity, in the temperature range of 1300-1600°C are being developed.

10:00 AM Break

10:15 AM

A Fundamental Study of Ag-Sb-Sn Alloys during Silver Recycling Processes: Yasushi Akahori[1]; Fumiko Nakai[1]; Tsuyoshi Kamata[2]; *Itaru Jimbo*[1]; [1]Tokai University, Dept. of Metallu. Eng., Hiratsuka, Kanagawa 259-1292 Japan; [2]Musashi Factory, Matsuda Sangyo Co. Ltd., Iruma, Saitama 358-0032 Japan

A cooperative research work on the silver recycling process is undertaken by Tokai University and Matsuda Sangyo Co., Ltd. in Japan. Silver wastes treated here are mainly from photograph industries where the antimony content in these wastes is increased in the recent years because of its increase in the printing paper to suppress the inflammability. The materials are concentrated and then treated pyrometallurgically, where the impurities such as antimony and tin are removed by evaporation and oxidation. In the present paper, the result of the fundamental studies on the removal of antimony and tin from Ag-Sb-Sn alloys will be discussed. Characteristics in the behavior of Sb and Sn during the removal process will also be discussed.

10:45 AM

Removal of Chromium from Industrial Waste Solutions: *Ignatius C. Okafor*[1]; *Ramana G. Reddy*[1]; [1]The University of Alabama, MTE Dept., A129 Bevill Bldg., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

Radioactive components processing facilities generate a lot of radionuclide wastes. There is a growing concern over the disposal of these wastes since they may cause incalculable harm in the nations portable water system or farm fields if disposed as is. It is therefore a national priority to find ways and means to remove the harmful toxic metals from the solutions. In this study the removal of chromium from solutions using zeolites was undertaken. The effect of pH of the solution, time, and type of zeolite used was studied. Four types of zeolite namely: chabazite, erionite, mordenite and clinoptilolite were investigated for the sorption of chromium. Optimal conditions for maximum removal of chromium were identified and the mechanism for sorption of chromium was proposed in terms of adsorption and intra-particle diffusion. Adsorption constants calculated from Langmuir and Freundlich equations indicate that the adsorption of chromium on chazabite was most favored.

Honorary Symposium for Professor Oleg D. Sherby: Superplasticity B

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

Thursday AM — Room: Bayou E
March 16, 2000 — Location: Opryland Convention Center

Session Chair: Terry McNelley, Naval Postgraduate School, Mech. Eng., Monterey, CA 93943-5000 USA

8:30 AM Keynote
Overview of Positive-Exponent Superplasticity: *Kenji Higashi*[1]; [1]Osaka Prefecture University, Dept. of Metallu. and Matls. Sci., College of Eng., 1-1, Gakuen-cho, Sakai, Osaka 599-8531 Japan

Recent studies have demonstrated that superplasticity can be found at strain rates over 10[0s?1] (Positive Exponent Superplasticity). Although the detailed mechanistic origins of positive-exponent superplasticity or high-strain-rate superplasticity (superplasticity found at strain rates over 10[-2s?1]) are not yet fully understood, some interesting experimental observations have been noted. Recently, Higashi et al proposed a model in which it is assumed that grain boundary sliding is the dominant deformation process, as in normal superplasticity, but with the present of accommodation helper such as a liquid phase at the interfaces or boundaries serving both to relieve the stress concentrations due to sliding and to restrict the build up of internal cavitation and subsequent failure by cavity interlinkage. In the current presentation, the previously reported data will be reviewed and further evidence will be presented to discuss the deformation mechanisms in positive exponent or high-strain-rate superplasticity. Finally the optimum design in microstructural control for the distribution of the accommodation helpers as well as the grain size refinement is demonstrated for the positive exponent or high-strain-rate superplastic materials.

9:00 AM Invited
High Strain-Rate Superplasticity of 2124 Al Alloy and SiC Reinforced 2124 Al Composite: *Woo-Jin Kim*[1]; Soon-Hyung Hong[2]; [1]Hong-Ik University, Sangsu-dong Mapo-ku, Seoul 121-791 Korea; [2]Korea Advanced Institute of Science and Technology, Matl. Sci. and Eng., Kusung-dong Yusung-ku, Taejon 373-1 Korea

Deformation behavior of high-strain-rate superplastic PM 2124 Al alloy and PM 20%SiCp/2124 Al composite was investigated over a wide range of temperature from 643 to 838K. The entire temperature range of investigation could be divided into two regions where grain boundary sliding (high-temperature range from 748 to 838K: Region I) and dislocation climb creep (low-temperature range from 643 to 693K: Region II) dominate the plastic flow, respectively. For the 2124 Al alloy, the true activation energies for Region I and II were close to that for the lattice diffusion in pure aluminum, QL. For the composite, however, true activation energy for Region I was considerably higher than QL, while the energy for Region II was close to QL. The grain-size compensated strength comparison indicates that composite is stronger than the unreinforced PM 2124 Al alloy in Region II but weaker in Region I. The threshold-stress behavior was investigated as a function of temperature.

9:20 AM Invited
Processing, Recrystallization, and Superplasticity in Aluminum Alloys: *Terry R. McNelley*[1]; [1]Naval Postgraduate School, Dept. of Mech. Eng., 700 Dyer Rd., Monterey, CA 93943-5146 USA

Superplasticity is exceptional ductility during tensile deformation of a material under appropriate temperature and strain rate conditions. Professor Oleg Sherby's preeminent research in elevated temperature deformation of metallic materials was instrumental in establishing the microstructure-property relationships applicable to the phenomenon of superplasticity. A prerequisite for it is a high strain rate sensitivity of the flow stress, which requires deformation under conditions where grain boundary sliding is the principal deformation mechanism, with accommodation of the sliding by either slip or diffusional processes. For this, the microstructure must be highly refined, with equiaxed, stable grains and mobile boundaries that are resistant to tensile separation. The necessary grain refinement in bulk aluminum alloys may be achieved only by recrystallization after plastic deformation and two distinct recrystallization routes that enable superplastic response in aluminum alloys have been identified. The present understanding of microstructural control by deformation and recrystallization of aluminum alloys will be reviewed. Results of investigations into the evolution of microstructures, microtextures and grain boundary misorientation distributions by means of computer-aided electron backscatter diffraction (EBSD) analysis methods for alloys representing each of these different routes will be summarized and implications to processing of Aluminum alloys for superplasticity will be discussed.

9:40 AM Invited
The Effect of Grain Size on the Threshold Stress for Superplastic Flow in Aluminum Alloys: Woo-Jin Kim[2]; *Dongwha Kum*[1]; [1]Korea Institute of Science and Technology, Matls. Eng., P.O. Box 131 Cheongryang, Seoul 130-650 Korea; [2]Hong-Ik University, Matls. Sci. & Eng., 72-1 Sangsoo-dong, Mapo-ku, Seoul 121-791Korea

Superplastic behavior of dispersion strengthened aluminum alloys and discontinuously reinforced aluminum composites at high strain rates has been interpreted by incorporating the concept of threshold stress, and the threshold stress exhibits strong temperature dependency. The threshold stress data in literatures fit well with an Arrhenius-type plot, and however the origin and exact meaning of the thermally activated process have not been fully understood. In this paper, a possibility of structural dependency of the threshold stress for superplasticity has been investigated. By considering the grain size as a structural factor, it is demonstrated that the threshold stress data for many superplastic aluminum alloys with different grain sizes and alloying chemistry fall into a narrow band in a modulus compensated threshold stress vs. grain size correlation. This analysis leads to a phenomenological equation with the grain size exponent of -1.1 for the threshold stress behavior in superplastic aluminum alloys.

10:00 AM Invited
Grain Size and Temperature Dependence of Superplastic Deformation in an Al-Mg Alloy Under Isostructural Condition: *Amit K. Ghosh*[1]; D. H. Bae[1]; [1]The University of Michigan, Dept. of Matls. Sci. and Eng., Ann Arbor, MI 48109 USA

Mechanical behavior of a superplastic Al-4.7%Mg-0.8%Mn-0.4%Cu alloy has been characterized by a new type of step strain-rate test which preserves the initial microstructure of the alloy (i.e. an isostructural test). Four different grain sizes of the alloy (8 to 30?m), prepared by variations in thermomechanical processing practice were examined. A sigmoidal relationship between log? and log? is observed for each isostructural condition. The value of maximum m? increased with increasing temperature and with decreasing grain size. The isostructural log? vs. log? data are evaluated using the grain-mantle based quantitative model proposed by Ghosh. In the dislocation creep region?, stress exponent is 4.55 and activation energy is close to that of lattice self-diffusion, but grain size exponent is non-zero (~ 0.37). In the grain mantle deformation region?, the value of stress exponent based on effective stress (s -so, where so is threshold stress) is ~ 1.7, and grain size exponent is 2.3; but interestingly activation energy is the same as that for dislocation creep. Grain mantle creep is now believed to be controlled also by dislocation glide and climb processes, but its rate is enhanced many times due to a high concentration of vacancies near grain boundaries. Computed so based on the model

shows that so increases with increasing grain size and with decreasing temperature.

10:20 AM Break

10:30 AM Invited

Impurity Segregation during Superplastic Flow: *Farghalli A. Mohamed*[1]; [1]University of California, Dept. of Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697 USA

The occurrence of micrograin superplasticity in metallic systems requires a stable and equiaxed grain size of less than 10 µm. This requirement along with the strong sensitivity of steady-state creep rates measured during superplastic flow to changes in grain size, has indicated that boundaries play an important role which is related to their ability to contribute to deformation through the process of boundary sliding. Recent analyses and experimental data have revealed another important role played by boundaries during superplastic flow. This role pertains to the ability of boundaries to serve as favorable sites for the accumulation of impurities, i.e. boundary segregation. It is the purpose of this paper to review the effects of impurities segregation at boundaries on superplastic deformation and cavitation.

10:50 AM Invited

Influence of Solute Additions on Superplastic Deformation: *John S. Vetrano*[1]; C. H. Henager[1]; S. M. Bruemmer[1]; [1]Pacific Northwest National Laboratory, MSIN P8-16, P.O. Box 999, Richland, WA 99352 USA

Fine-grained superplastic deformation is dominated by the process of grain boundary sliding (GBS). Though the exact mechanism of GBS is still being debated, there are certainly major contributions from grain boundary diffusion and localized movement of dislocations. We are investigating the role of solute atoms on these processes by measuring deformation behavior and grain boundary composition in Al-Mg alloys with and without the addition of 80 appm Sn. Scanning Auger Microprobe measurements of grain boundary composition showed that the Sn segregated to the boundaries and was present at amounts up to 10 at.% prior to testing. At temperatures below 500°C the presence of Sn had a negative impact on elongation to failure in both coarse- and fine-grained Al-Mg alloys. In coarse-grained samples it was noted that the fracture surface changed from a narrow point in the Al-Mg alloy to a "quasi-brittle" intergranular failure in the Al-Mg-Sn material. At temperatures of 500°C and 550°C, the material containing Sn showed consistently higher elongations, particularly at low strain rates. At 550°C and a strain of 1x10-4 s-1 the elongation to failure was increased from 600% to 740% by the addition of Sn. Preliminary microstructural examination of quenched samples indicate that the Sn is increasing GBS under all conditions, but at lower temperatures the material fails due to insufficient accommodation of the sliding. Work supported by the Materials Division, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC06-76RLO 1830.

11:10 AM Invited

An Analysis of Gas Pressure Forming of Superplastic Al 5083 Alloy: *Chol K. Syn*[1]; Donald R. Lesuer[1]; Oleg D. Sherby[2]; [1]Lawrence Livermore National Laboratory, P.O. Box 808, L-342, Livermore, CA 94551-0808 USA; [2]Stanford University, Matls. Sci. and Eng., Stanford, CA 94305 USA

Superplastic Al 5083 alloy disks were gas-pressure formed to hemispheres and cones at constant forming pressures with and without back pressure. The forming operation was performed using an in-house designed and built biaxial forming apparatus. The temporal change of dome heights of the hemispheres and cones were measured for the different forming and back pressures applied. Several cone samples were etched with circle grids prior to the forming, and local minor and major strains were measured after the forming was performed. The flow stresses and strain rates developed at the top of the dome during forming were shown to closely follow the flow stress-strain rate relation obtained from the strain rate change tests performed at the same temperature using uniaxial tensile samples. The local strain measurements on the circle-gridded samples, the data from the tensile tests performed on-site, and other data from literature sources were used to construct a partial forming limit diagram for superplastic forming of the alloy. Detailed experimental procedures and the results of their analyses will be presented. Work was performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under contract W-7405-ENG-48.

11:30 AM Invited

The Effect of Ternary Alloying Additions on Solute-Drag Creep in Aluminum-Magnesium Alloys: *Eric M. Taleff*[1]; Peter J. Nevland[1]; [1]The University of Texas, TX Matls. Instit., Austin, TX 78712-1063 USA

Solute-drag creep is observed in many aluminum alloys containing magnesium concentrations from as little as 2 wt pct to the limit of solubility at temperature. Detailed studies of this behavior in low-impurity, binary Al-Mg alloys are available in the literature, and established models have been successful in predicting observed behavior. Solute-drag creep in more complex Al-Mg alloys has received less study, yet is of practical significance because of the enhanced ductility which it affords. Tensile ductilities in excess of 100% are achieved repeatedly in commercial Al-Mg alloys deformed by solute-drag creep. Recent investigations have produced data for commercial alloys as well as low-impurity, ternary Al-Mg alloys containing Mn, Fe, and Zn. This article presents these data in comparison with data from binary Al-Mg alloys and pure Al. The effects of ternary alloying additions are analyzed for application to the design of future commercial alloys.

Liquid Metal Atomization: Fundamentals and Practice: Microstructure & Industrial Practice

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

Thursday AM — Room: Bayou B
March 16, 2000 — Location: Opryland Convention Center

Session Chairs: Iver E. Anderson, Iowa State University, Ames Lab., Ames, IA 50011-3020 USA; Steven P. Marsh, Naval Research Laboratory, Washington, DC 20375 USA

8:30 AM Invited

Solidification of Atomized Liquid Droplets: *John H. Perepezko*[1]; Jason L. Sebright[1]; Peter G. Höckel[1]; [1]University of Wisconsin, Matls. Sci. and Eng., 1509 University Ave., Madison, WI 53706 USA

An essential characteristic of liquid atomization processes is the dispersal of a liquid volume (i.e. stream) into a collection of droplets. Following subdivision, which also serves to isolate effectively internal nucleation catalysts, droplet thermal history is an important component of the solidification behavior and microstructure development. The thermal history is affected by droplet size not only due to external cooling, but also due to liquid undercooling. The undercooling behavior, the resulting nucleation onset, and the solid fraction and morphology that develop in flight are also key factors in determining droplet structure during a number of processes including deposition and coating development. In these cases, the basic nature of nucleation as a probabilistic process and the activity of various heterogeneous nucleation catalysts play a major role in determining the variation in droplet structure formation. The nucleation behavior can be evaluated most effectively in controlled undercooling measurements on droplet populations or with single droplet samples. The variability of the nucleation temperature about a range of 10°C or more even for a fixed droplet size is one consequence of the stochastic nucleation process that is usually not included in process models. Similarly, the strong

dependence of liquid lifetime on droplet size in both fully liquid and partially solid states is directly related to nucleant potency. Models are under development to describe nucleant activity in atomized droplets that can be included in overall process modeling strategies. The support of NSF (DMR-9712523) and ARO (DAAG55-97-1-0261) are gratefully acknowledged.

9:00 AM

Nanostructures in Gas Atomized Amorphizable Alloy Powders: *Andrea Zambon*[1]; Brando Badan[1]; Emilio Ramous[1]; [1]Universita' di Padova, Dept. of Mech. and Mgmt. Innov., Via Marzolo 9, Padova 35131 Italy

Nanostructured materials are gaining much interest in view of their high mechanical properties. A common way of obtaining such a microstructure is to induce devitrification of an amorphous alloy by means of a suitable heat treatment. Amorphization can be accomplished by rapid solidification processes or by stimulating solidification at high undercooling in bulk samples of suitable composition. Gas atomization can produce both high cooling rates and relatively high undercooling extents. As the cooling rate experienced by the atomized particles depends on their size, both amorphous and nanostructured powder can be found in an atomization batch if an amorphizable alloy is processed. The solidification morphologies obtained in gas atomized zirconium base alloys, investigated by means of X-ray diffraction, SEM and TEM examination, are related with their size and with computed cooling rates.

9:25 AM Invited

Powder Surface Microchemistry and Reactions During Atomization: *Lars Nyborg*[1]; [1]Chalmers University of Technology, Dept. of Eng. Mets., Horsalsvagen 7, Gothenburg SE41296 Sweden

Due to the high surface area to volume ratio of powder, the surface condition of powder is a crucially important factor that determines the use and further processing of the powder. Good processing control and powder handling procedures ensure that surface contamination (e.g. surface oxides, etc.) is kept at a minimum. However, despite the clean conditions and high cooling rates employed during atomization, surface reactions will take place. It is of general interest to be able to describe these reactions and to understand the fundamental aspects behind the formation of reaction products. The scope of this communication is to provide a general overview on this subject based on observations made by means of surface chemical analysis (XPS, AES, SIMS, etc. of atomized high-alloy powder. Discussion will focus on the role of atomisation method (gas or water), surface reactions with external impurity elements (e.g. oxygen), and surface reactions with internal impurity elements (e.g. sulphur) due to surface segregation phenomena. Factors controlling the possible amount of different reaction products will be discussed and special attention will be placed on how to distinguish between surface reactions that occur during atomization and powder handling. Some general conclusions are as follows. i) Strong oxide formers (such as Si, Mn, Al and Cr) are oxidized at high temperatures during atomization. ii) The base element (Fe or Ni) is mainly oxidized during cooling and subsequent powder handling. iii) Surface oxidation is controlled by oxygen availability during gas atomization, while cooling rate is rate-controlling during water atomization. iv) Particulate reaction products may form on the powder surfaces rather than layered products. v) Surface segregation of e.g. S depends on competing events such as surface oxidation.

9:50 AM Invited

Water Atomization of Ferrous Metals and Alloys: *K. S. (Sim) Narasimhan*[1]; C. T. Schade[1]; [1]Hoeganaes Corporation, 1001 Taylors Ln., Cinnaminson, NJ 08077 USA

Liquid metal atomization to produce particulates of definite size and shape distribution has advanced significantly during the last 30 years. Currently, powders of iron, stainless steel, and various prealloys of iron with chromium, manganese, nickel and molybdenum are routinely being produced to satisfy the needs of $6 billion parts industry. This presentation will cover various types of powder produced by water atomization and the advances in manipulation of particle morphologies by controlling atmosphere, molten metal, jet geometry and other parameters.

10:15 AM Break

10:30 AM Invited

CFD Simulation Interaction with Production Atomization Trials: *William B. Eisen*[1]; Michael J. Peretti[1]; Jason Ting[1]; Rocco A. Longo[2]; [1]Crucible Materials Corporation, Crucible Rsch. Ctr., 6003 Campbells Run Rd., Pittsburgh, PA 15205-1022 USA; [2]Crucible Materials Corporation, Crucible Compact. Met., 1001 Robb Hill Rd., Oakdale, PA 15071 USA

Crucible has two long term objectives to improve the operation of its production atomizers: 1) improve the yield of finer powder and (2) produce finer powder without any loss of productivity. The use of CFD simulations and aspiration pressure testing as a guide to the design of a gas delivery system to achieve these objectives is described. Data from the initial production trials are presented and compared with the current production methods on a 800 lb. and a 5500 lb. atomizer.

10:55 AM Invited

Synopsis of Commercial Atomizing Modes for Metal Powders: *Krishna B. Patel*[1]; Edul M. Daver[1]; [1]ACuPowder International, LLC, 901 Lehigh Ave., Union, NJ 07083 USA

A Cu Powder is engaged in the business of producing non-ferrous metal powders for 65 years. Various methods of atomizing processes have been practised at this facility. Essentially all processes were developed in house over the years. In general, this paper will characterize merits of vertical, horizontal and downward modes used to atomize metal powders. Specific examples will be narrated. Original patent for the atomization of aluminuim powder invented by the founder of the company will be addressed. Water atomization of copper powder will be described (without divulging proprietary information). The effects of some of the process variables onto the characteristics of copper powder for use into targeted market segments will also be described.

11:20 AM Invited

Commercial Atomization Processes for Aluminum Powder Manufacture: *George T. Campbell*[1]; Roy W. Christensen[1]; [1]Valimet, Inc., P. O. Box 6186, 431 Sperry Rd., Stockton, CA 95206 USA

Commercial atomization processes for aluminum powder manufacture range from air atomization to closed loop inert gas atomization. The shape of the particle produced as well as the surface chemistry of the particle will vary significantly depending upon the atomization process used. Particle size distribution, particle shape, and surface chemistry can have a significant impact on the response in subsequent processing of powder. This paper discusses the various commercial atomization processes currently in practice and the characteristics of the powder produced by these processes.

11:45 AM Invited

Research in Nozzle Systems to Produce Spray Formed Aluminum Automotive Sheet Products: *David D. Leon*[1]; Robert L. Kozarek[1]; Diana K. Denzer[1]; [1]Alcoa, Inc., Alcoa Tech. Ctr., Ingot & Solid. Platform, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

The U. S. Department of Energy-Office of Industrial Technology (DoE-OIT) has an objective to increase energy efficiency and enhance competitiveness of American metals industries. To support this objective, Alcoa, Inc. entered into a cooperative program to develop spray forming technology for aluminum. Alcoa has developed spray forming processes capable of scale-up for commercial production of aluminum alloy sheet products. This paper will review research with linear nozzles and the effect of nozzle system geometry and operating parameters on the as-cast deposit. Emphasis will be given to Al 6111 a commercially significant alloy in the automotive industry. For completeness, Alcoa research in computer process simulation, thermo-mechanical processing techniques, and economics will also be covered.

Magnesium Technology 2000: Wrought Alloys and Thixomolding

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

Thursday AM — Room: Bayou C
March 16, 2000 — Location: Opryland Convention Center

Session Chair: Lee T. Barnes, Spectrulite Consortium, Madison, IL 62060 USA

8:30 AM
Alternative Ways to Fabricate Magnesium Products: *Firoze E. Katrak*[1]; Jagdish C. Agarwal[1]; Francis C. Brown[1]; Michael J. Loreth[1]; [1]Charles River Associates, Inc., John Hancock Tower, 200 Clarendon St. T-33, Boston, MA 02116-5092 USA

Magnesium demand will grow even faster if and when alternate cost-effective fabrication methods are designed. Today most of the growth is driven by diecastings, which cannot be used in a broader set of potential applications that need strength. Conventional rolling of magnesium mill products is unlikely to be cost-effective because magnesium has an HCP structure. Thus, the application of extrusion to the fabrication of a range of magnesium parts has tremendous potential. CRA will discuss in this paper the issues related to fabrication costs, and how the cost of extrusion compares to those of other fabrication methods. We will also discuss major limitations and opportunities in technology development for magnesium extrusions.

8:55 AM
Extrusion of AZ31: Modeled Distribution of Stress, Strain Rate, Temperature and Microstructure: *H. J. McQueen*[1]; M. M. Myshlyaev[2]; E. V. Konopleva[1]; M. Sauerborn[1]; [1]Concordia University, Dept. of Mech. Eng. H-549, 1455 de Maisonneuve Blvd. W., Montreal, Quebec H3G1M8 Canada; [2]Baikov Institute of Metallurgy, RAS, Moscow 117911 Russia

Following torsion tests of Mg-3Al-1Zn in the ranges of 180-450°C and 0.01-1 /s to determine the dependence of flow stress, ductility and microstructure on temperature and strain rate, axisymmetric extrusion was modeled by Deform(TM) finite element software. The incidence of twinning across the entire range and the increased level of dynamic recovery and recrystallization as temperature rose were observed by optical and scanning electron microscopy. Application of the sinh-Arrhenius constitutive equation with activation energy 145 kJ/mol enabled calculation of the distributions of temperature, strain rate and stress throughout the billet for extrusion ratio 31, ram speed 5 mm/s and insert temperature from 300 to 450°C. The maximum of these variables occurred at the die exit corner so that the occurrence of surface cracking was estimated from the measured ductility. Streamlines from billet to extrudate were plotted and the variation of temperature and strain rate along lines at 1/4, 1/2, 3/4, and full radius were determined. The microstructure development at various depths were derived. The relevance of these results to industrial processing is discussed.

9:20 AM
Deformation Characteristics of Wrought Magnesium Alloys AZ31, ZK60: *A. Ben-Artzy*[1]; A. Shtechman[1]; N. Ben-Ari[2]; [1]Rotem Industries Limited, Dead Sea Magnesium Ltd., P. O. Box 75, Beer-Sheva 84100 Israel; [2]Ben-Gurion University, Beer-Sheva, Israel

The use of magnesium alloys in the automotive industry is expanding rapidly due to the introduction of new air pollution regulations, implemented in the western world and Japan. Cast magnesium alloys such as AZ91 and AM50 are occupying the essential part of the automotive magnesium parts demands. Magnesium alloys used for car structural parts have to be produced from energy absorption materials, characterized in good elongation, high yield strength and high impact energy. Wrought magnesium alloys have the potential to serve these needs better than the die cast Mg alloys. Open hot forging of cylindrical samples, in the temperature range of 290°C÷420°C, strain in the range of 0.1÷0.5 and strain rates 0.001÷0.5(1/sec) were used to plot FLD (Forming Limits Diagram) for the AZ31 and Zk60 magnesium alloys. The influence of forming temperature and strain rate on the microstructure and mechanical properties was determined. AZ31 was found to be sensitive to strain rate in the mid temperature range. ZK60 has no sensitivity to strain rate within the tested temperature range, due to the grain refinement caused by the presence of zirconium. The DRV (Dynamic Recovery) and DRX (Dynamic recrystallization) were found to be dominating plastic deformation behavior of the wrought magnesium alloys in elevated temperature range.

9:45 AM
Environmental Effects on the HCF Behavior of the Magnesium Alloys AZ 31 and AZ 80: *Matthias Hilpert*[1]; Lothar Wagner[1]; [1]Technical University of Brandenburg at Cottbus, Matls. Tech. and Phys. Metallu., P.O. Box 101344, Cottbus 03013 Germany

The fatigue performance of two widely used magnesium alloys AZ 31 and AZ 80 was studied in various environments. HCF tests were performed on electrolytically polished hour-glass shaped specimens in fully reversed (R=-1) axial loading using a resonance tester at frequencies of about 100 Hz. Tests were done in vacuum, ambient air and in an aqueous 3.5% NaCl solution. In addition, a few tests were performed on specimens which had been mechanically surface treated by shot peening and roller-burnishing. Results will be explained by environmental effects on fatigue crack nucleation and microcrack propagation.

10:10 AM Break

10:20 AM
Structure and Mechanical Properties of Friction Stir Weld Joint of Magnesium Alloy AZ31: *Takeshi Nagasawa*[1]; Masahisa Otsuka[1]; Takeo Yokota[1]; Tadahiro Ueki[1]; [1]Shibaura Institute of Technology, Dept. of Matls. Sci. and Eng., Shibaura 3-9, Minato-ku, Tokyo 1088548 Japan

The applicability of friction stir welding to hot rolled sheet of commercial magnesium alloy AZ31 sheet has been investigated. Friction stir weld joint showed mechanical strength comparable to that of base material, though the ductility remained at one half of that of the latter. This is consistent with the fact that the bond layer was composed of fine and equiaxed grains which had recrystallized during friction stirring. It is found that both anodizing treatment and insertion of aluminum foil between batting faces do not degrade the joint properties at all. The results suggest that friction stir welding is potentially applicable to magnesium alloy.

10:45 AM
New Developments in Magnesium Production Technology: *Dieter Brungs*[1]; [1]Honsel AG, Fritz-Honsel-Strasse, Merschede 59872 Germany

Magnesium pressure die castings have been introduced successfully into light-weight automobile body structures. In many cases, the special design opportunities and properties of magnesium alloys compensate the higher base metal cost compared with aluminium. However, applying pressure die casting machines up to 50.000 kN locking force, limitations regarding the maximum dimensions of the highly integrated die cast components become visible. On the other hand, Magnesium extrusion technology has reached an advanced status to produce complex, thin walled sections. Focus will be on the presentation of an advanced extrusion process for magnesium alloys. Depending on the alloy applied, mechanical properties and production costs of the Mg-extrusions are assessed. The combination of Mg-extrusions and die castings will create new light-weight design opportunities especially for the body structure of vehicles. Possible applications will be discussed, considering component recycling, productivity, cost situation,

mechanical properties and joining technologies like fusion welding and friction stir welding.

11:10 AM

Mechanical Properties and Microstructure of Heat-Resistant Mg-Al-Ca Alloys Formed by Thixomolding Process: *T. Tsukeda*[1]; A. Maehara[1]; K. Saito[1]; M. Suzuki[2]; J. Koike[2]; K. Maruyama[2]; H. Kubo[2]; [1]The Japan Steel Works Limited, 1-6-1 Funakoshi-Minami, Aki-ku, Hiroshima City, Hiroshima-pref. 736-8602 Japan; [2]Tohoku University, Japan

Thixomolding is a new process in which the metallic slurry is injected into a die cavity at semi-solid temperature to form near net-shape products from the solid feed stock in one step. The relationship between the chemical composition and the mechanical properties of heat-resistant Mg-6mass%Al-04mass%Ca alloys was investigated. The effect of process condition such as barrel temperature was also studied. It was found that the addition of Ca was effective to improve the yield strength and the creep resistance at elevated temperatures. The ductility decreases with increasing Ca content. The microstructure of specimens observed by SEM and TEM showed a fine hypoeutectic structure consisting of granular Mg solid solution and phase ($Mg_{17}Al_{12}$) networks incorporated with Ca. The morphology of these networks varies depending on Ca content.

11:35 AM

Developments of Semi-Solid Molded Magnesium Components from Alloys with Improved High Temperature Creep Properties: *Robert D. Carnahan*[1]; Raymond F. Decker[1]; Eric A. Nyberg[2]; Russell H. Jones[2]; Stan G. Pitman[2]; [1]Thixomat, Inc., 620 Tech. Dr., Ann Arbor, MI 48108 USA; [2]Pacific Northwest National Laboratory, Matls. Res., Battelle Blvd., P.O. Box 999, Richland, WA 99352 USA

In this study magnesium alloy ZAC8506, known for its high temperature creep resistance, has been formed using the semi-solid forming process known as Thixomolding®. The ZAC alloys have not proven to be amenable to processes such as die-casting because of their relatively poor castability as compared to standard die-casting alloys. However, it was shown that with proper die design and molding parameters, Thixomolding® of the ZAC alloy is possible. Moldability was evaluated by evaluating the formability of an automotive electrical cover box and a spiral fluidity test. The electrical cover box was evaluated for porosity and room temperature mechanical properties. The results indicate that the level of porosity is below that of typical die-cast components. This work is part of a joint program with the Pacific Northwest National Laboratory (Northwest Alliance for Transportation Technology-Partnership for Next Generation Vehicle Program) and Thixomat, Inc. to evaluate the high temperature application of Thixomolded magnesium components to automotive applications. Due to the promising formability results of the ZAC8506 alloy, a comparison of the creep performance between Thixomolded AZ91D and ZAC8506 was conducted using an instrumented bolt load retention component assembly. In addition, die-cast AZ91D and die-cast ZAC8506 properties were compared. The results of both the bolt load retention tests and the comparison with the die-cast materials will be reported.

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

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 Packing Technologies, San Jose, CA 95134 USA; Martin Weiser, AlliedSignal Electronic Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Thursday AM — Room: Lincoln C
March 16, 2000 — Location: Opryland Convention Center

Session Chairs: Yellapu V. Murty, Carpenter Technology Corporation, Rsch. & Dev., Reading, PA 19612-4662; Sungho Jin, Lucent Technologies, Appl. Matls. and Metallu. Grp., Murray Hill, NJ 07974 USA

8:30 AM

Tantalum Sputtering Target and Sputter Deposition of Ta/TaN Diffusion Barriers For Cu Interconnect: *Hao Zhang*[1]; [1]Tosoh SMD, Inc., 3600 Gantz Rd., Grove City, OH 43123 USA

Copper is considered as a new interconnect material to replace aluminum alloys in sub-0.25 micron IC devices because of its low resistivity, excellent electromigration and stress migration resistance. However, Cu can readily diffuse into Si and SiO_2-based dielectrics, causing degradation and failure in IC devices. Therefore, the application of Cu interconnect requires an effective diffusion barrier which can prevent Cu from diffusing into Si and SiO_2. Among many barrier materials have been studied, Ta and TaN are reported to have excellent diffusion barrier properties between Cu and Si. Sputtering is an effective method to deposit Ta and TaN in the Cu/barrier/Si or Cu/barrier/SiO_2 structures, and has attracted considerable attention. In this study, Ta and TaN films with different thickness were sputter deposited from high-purity Ta targets. The performance of the Ta targets was evaluated. The effects of process parameters such as N_2/Ar flow ratio on film properties such as electrical resistivity, mechanical stresses and film uniformity were studied. The properties of Ta and TaN barriers are strongly dependent on the sputtering process. In addition, the Ta film thickness distribution across 200 mm Si wafer was simulated by using SIMBADTM package (SIMBADTM is a trademark of Alberta Microelectronic Center), and compared to the experimental results. The phases and the crystallographic texture of the Ta and TaN films were studied by using X-ray diffraction and pole figure analysis.

9:00 AM Keynote

Mainstreaming Photonics: Strategic Technology for the New Millennium: *John L. MacWilliams*[1]; [1]US Competitors, LLC, 561 Upper Pike Creek Rd., Newark, DE 19711 USA

The talk will summarize significant industry and technology trends in fiber optics. Photonics is now a multi billion-dollar thread woven into the $1.75 trillion information technology market. The market segments, industry dynamics, and global competition will be presented as an overview. The associated component technologies vital to this highly sophisticated markets will be described.

9:25 AM

Antiquity Lead, Source of No Alpha Lead (NAL): *Rick Jensen*[1]; [1]Sea Recovery Limited

Applications for lead in the Roman Empire; applications clustering large volumes of lead; historic dispersion factors; problems associated with finding intact clusters; European country policies on the export of antiquity lead; Roman applications resulting in Sea Recovery Ltd lead clusters, lack of dispersion factors, surveyed quantities, recovery issues. Radioactive decay in antiquity lead; ingot size/weight and appearance; cutting samples from ingots without alpha contamination; alpha particle emission testing and results; 74 element non-radioactive impurity testing and results (chart); non-radioactive impurities of concern to the semiconductor industry; refining issues: semiconductor industry specifications, eliminating sources of radioactive contamination, certification, physical forms of the end product. Chart of the alpha levels of Low Alpha Lead (LAL) purchased by the semiconductor industry since 1997. Emergence of NAL applications. Future of NAL applications.

9:50 AM

Modification of Thermal Expansion Behavior of Materials for Microelectronic Applications: *Sungho Jin*[1]; Hareesh Mavoori[1]; [1]Bell Laboratory Lucent Technologies, Rm. 1A-123, 600 Mountain Ave., Murray Hill, NJ 07974 USA

In microelectronic devices and assemblies, the thermal expansion behavior of materials, especially the mismatch in the CTE among various component materials is an important issue in terms of device performance and reliability. The CTE is generally considered to be an intrinsic property of materials. However, there are some novel ways of modifying the thermal expansion behavior, e.g., so as to obtain very small, very large, near-zero, or negative CTE values by utilizing magnetic transition or phase transition near room temperature. Some examples of CTE control in electronic materials and composite structures, as well as their potential device applications will be discussed. The heat sink materials commonly used today such as Cu and Al alloys have a much higher coefficient of thermal expansion (CTE) than Si. CTE mismatch between the various materials in an electronic package can lead to stresses that can trigger complex failure mechanisms seriously degrading the device reliability and lifetime. Therefore, it is highly desirable to minimize the CTE mismatch by developing new heat sink materials with CTEs close to that of Si using either a composite or alloying approaches, while preserving much of the thermal conductivity in the elemental heat sink materials.

10:15 AM

Ohmic Contacts to p-GaN: *Sujit Pillai*[1]; Eicke R. Weber[1]; [1]University of California, Matls. Sci. and Eng., 161M Cory Hall, Berkeley, CA 94720 USA

The group III nitrides, especially GaN, are attractive materials for optoelectronic devices because of the success in commercialization of high brightness blue/cyan and green light emitting diodes and violet Laser Diodes. Ohmic contacts to GaN are very important because the performance of these devices such as operating voltage is strongly influenced by the contact resistance. A very low-resistance ohmic contact to n-type GaN has been demonstrated using Ti/Al/Ni/Au. However, ohmic contacts to p-type GaN are still a challenge. Various metal contacts have been applied to p-type GaN, and all of the reported specific contact resistances are in the range from 2.1 × 10^2 to 9.6 × 10^- 4 ohm.cm^2. These values are too high for high performance devices. Low contact resistances to p-type GaN are difficult to obtain because of the difficulty in achieving high hole concentrations in p-type GaN and the lack of metals with high work functions compared to the band gap and electron affinity of GaN. Ohmic Contacts to p-GaN may be formed in 4 different ways: 1. High workfunction metals; 2. Transparent hole injecting oxides; 3. Nitride Forming metals; 4. Hydride forming metals. We will discuss the above strategies with examples. The tradeoff between optical transmissivity and contact resistance will be discussed for contacts to LEDs. Novel contact structures will also be discussed. Demonstrations of InGaN LEDs, including the white LED, will also be included.

10:40 AM

Roles of Self-Assembled Monolayers in Slow Cracking of Polymer-Metal Interfaces: *Tianbao Du*[1]; Jian Ku Shang[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA

Polymer-metal interfaces are built into a wide range of microelectronic devices. The adhesion of polymer to metal can degrade gradually under the combined action of internal/external stress and environment. In this study, the use of self-assembled polymer monolayers was explored to inhibit interfacial stress corrosion in epoxy/Cu and epoxy/Al systems. Self-assembling phosphonic acids with various end groups were synthesized and monolayers of the synthesized polymers were introduced at the polymer-metal interface. The resistance of the modified polymer-metal interfaces to stress-corrosion cracking was determined using interface-fracture mechanics techniques, and compared to that of the unmodified interface. Striking differences were observed in short-term adhesion and in long-term durability between the modified and unmodified interfaces. These differences are discussed in terms of the changes in crack growth mechanisms.

11:05 AM

Stresses and Deformation in Miniature Structures and Micro-Electro-Mechanical Systems (MEMS): *T. A. Venkatesh*[1]; Subra Suresh[1]; [1]MIT, Matls. Sci. and Eng., Rm. 8-139 77 Massachusetts Ave., Cambridge, MA 02139 USA

Small-volume structures, Micro-Electro-Mechanical Systems (MEMS), and MEMS-based functional devices collectively represent a rapidly growing field with a large potential for far-reaching technological impact, the realization of which depends critically on identifying, and developing solutions to, the multi-disciplinary problems that limit the design, fabrication and operational reliability of these devices. It is increasingly becoming evident that the optimization of electro-mechanical, thermal, optical, fluidic or magnetic functionality of MEMS devices requires a comprehensive understanding of several materials and mechanics issues in MEMS, particularly those dealing with the prediction, measurement and control of processing-induced and service-induced stresses. In this work, we propose a novel classification of miniature structures based on their geometry (1-D, 2-D or 3-D), structural environment (unconstrained, partially constrained or fully constrained), and coupling characteristics (pure mechanical, piezoelectric or magnetostrictive). This framework is developed with the objective of addressing the uncoupled and coupled mechanical response of MEMS in a unified fashion. Theories for predicting stresses and deformation in a wide variety of applications involving small-volume structures are presented and discussed within the context of length-scales and size-dependence of material properties. The existing techniques for the quantitative determination of the geometry-dependent, thermo-mechanical properties (elastic, plastic, fracture and fatigue), residual stresses and tribological characteristics are also briefly examined for their respective advantages and limitations.

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

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

Thursday AM — Room: Knoxville B
March 16, 2000 — Location: Opryland Convention Center

Session Chairs: Boyd Mueller, Howmet Corporation, Whitehall, MI 49461 USA; Kuang Oscar Yu, RMI Corporation, R&D, Niles, OH 44446-0269 USA

8:30 AM

The Effect of Interface Heat Transfer on Solidification, Microstructure Evolution, and Mold Wear During Permanent Mold Casting of Ti-6Al-4V: *Pamela A. Kobryn*[1]; S. Lee Semiatin[1]; [1]Air Force Research Laboratory, Met., Cer., and Nondestruct. Eval. Div., AFRL/MLLM Bldg. 655, 2230 Tenth St., Ste. 1, Wright-Patterson AFB, OH 45433-7817 USA

Computer simulation capabilities have been developed for predicting characteristics of solidification, microstructure evolution, and mold wear for permanent mold casting of Ti-6Al-4V. The solidification modeling package ProCAST™ was used throughout. A combination of physical and numerical experiments was used to determine interface heat transfer coefficients and the validity of using the chosen modeling approaches and input parameters to simulate various casting geometries. Laboratory and in-plant casting trials were conducted to obtain casting data. Thermocouple data were compared to simulation results to determine interface heat transfer coefficients for "shrink off" and "shrink on" casting geometries. Both a conventional thermocouple technique and a novel microstructure-based mold temperature signature analysis technique were used to determine mold temperatures for model validation. The validated models were used to predict microstructure via solidification mapping and solid state grain growth. The importance of properly accounting for the casting-mold interface contact condition was stressed throughout.

8:55 AM

The Effects of Compositional Variation and Aging Temperature on the Tensile Properties of Cast Ti-15V-3Cr-3Al-3Sn: *Donald R. Clemens*[1]; [1]Howmet Research Corporation, Adv. Tech., 1500 S. Warner St., Whitehall, MI 49461 USA

Ti-15V-3Cr-3Al-3Sn (Ti-15-3) is a metastable beta titanium alloy that is age hardenable. Minor variations in both composition and aging temperature cause a dramatic difference in the morphology and amount of alpha precipitation during the solution and age cycles. This in turn effects the tensile properties, which can range from very high strength and low ductility, to low strength and high ductility. This study investigated three compositional variants of Ti-15-3: an alpha rich composition, a nominal composition, and a beta rich composition. All three were within the typical range of acceptable compositions for Ti-15-3. After hot isostatic pressing and solutioning, a number of single, double, and triple step aging cycles were investigated. Tensile results will be discussed along with recommendations for optimizing the composition and aging temperature to obtain an alloy with high strength while maintaining adequate ductility. The majority of this work was conducted using investment casting, however, vacuum die cast material was also tested. The finer grainsize obtained with vacuum die casting had a positive effect on the tensile properties.

9:20 AM

Dental Investment Castings of Beta-Type Titanium Alloy: Hisao Fukui[2]; *Mitsuo Niinomi*[1]; Tsutomu Takeuchi[3]; Shigeki Katsura[4]; Keiichi Fukunaga[1]; Jiro Hasegawa[2]; Shinya Yoshitani[1]; Daisuke Kuroda[1]; [1]Toyohashi University of Technology, Dept. of Product. Sys. Eng., Toyohashi 441-8580 Japan; [2]Aichi-Gakuin University, Dept. of Dental Matls. Sci., Nagoya 464-8650 Japan; [3]Tekeuchikatan Limited, Toyohashi 441-8132 Japan; [4]Yamahachi Dental MFG Company, Gamagori 443-0105 Japan

Dental investment casting processes of newly developed beta-type titanium alloy composed of non-toxic elements, Ti-29Nb-13Ta-4.6Zr for orthopedic instrumentations were investigated. The effects of investment materials on the surface reaction and tensile properties of Ti-29Nb-13Ta-4.6Zr castings were, in particular, examined in this study. The mold reaction of Ti-29Nb-13Ta-4.6Zr is greater when alumina with phosphate system materials is used for investment materials than when magnesia with non-phosphate system one is used. The mold reaction observed on the surface of Ti-29Nb-13Ta-4.6Zr is greater than that of conventional biomedical pure titanium and Ti-6Al-4V. Tensile strength of Ti-29Nb-13Ta-4.6Zr casting is smaller than that of Ti-6Al-4V while elongation of Ti-29Nb-13Ta-4.6Zr castings is greater than that of Ti-6Al-4V castings.

9:45 AM Break

10:00 AM

Thermochemical Processing of Ti-6Al-7Nb Castings for Biomedical Applications: *Mitsuo Niinomi*[1]; Ryosuke Isohama[1]; Toshikazu Akahori[1]; Akihiro Suzuki[2]; [1]Toyohashi University of Technology, Dept. of Product. Sys. Eng., Toyohashi 441-8580 Japan; [2]Daido Steel Company Limited, R&D Lab., Nagoya 455-0811 Japan

Thermomechanical processing, that is, hydrogenation and de-hydrogenation process was investigated in order to improve the balance of strength and ductility of investment castings of Ti-6Al-7Nb for dental applications. Elongation of Ti-6Al-7Nb castings conducted with conventional thermochemical processing is smaller than expected value, over 10%, although strength is enough. The good balance of strength and ductility can be achieved by adopting proper heat-treatments after de-hydrogenation in the thermochemical processing. The microstructure of the heat-treated alloy after dehydrogenation is a little coarser than that of the un-treated one. However, the microstructure of the heat-treated alloy after de-hydrogenation is a little more spherical than that of the un-treated one.

10:25 AM

Phase Transformations in Ti-6Al-4V-xH Alloys: *Javaid I. Qazi*[1]; Oleg N. Senkov[1]; Francis H. Froes[1]; William M. Mullins[2]; [1]University of Idaho, IMAP, Mines Bldg. Rm. 321, Moscow, ID 83844-3026 USA; [2]US Army Research Office, AMSRL-RO-PM, P.O. Box 12211, Research Triangle Park, NC 27709-2211 USA

Ti-6Al-4V alloy samples were alloyed with 10, 20 and 27 at.% hydrogen by holding the samples in a hydrogen atmosphere at 780°C. The different hydrogen alloying was achieved by varying the hydrogen partial pressure in the chamber during hydrogenation. Phases and temperatures of phase transformations in the hydrogenated samples were determined by X-ray and microstructural analyses. TTT diagrams for decomposition of metastable beta and martensitic phases were also determined using similar approach. Using the results obtained, conditions of heat treatment of the hydrogenated samples were optimized leading to a refined grain structure and improved mechanical properties after dehydrogenation.

10:50 AM

Sintering Behavior of a Titanium Powder Produced by a Novel Continuous Process: *Stephen J. Gerdemann*[1]; David E. Alman[1]; [1]Albany Research Center-DOE, Therm. Treat. Tech., 1450 Queen Ave. S.W., Albany, OR 97321 USA

The sintering behavior of titanium powder that was produced by a novel reduction process was characterized. Green specimens were die pressed into cylindrical compacts and vacuum sintered. The influence of green density, sintering temperature and sintering time on the final

density and microstructure was evaluated. Once these experiments were concluded, near-net-shape tensile bars were pressed and consolidated using the determined optimal sintering conditions, and room temperature tensile properties were measured. The results were compared to the properties of material produced from commercially available sponge fine powders and cast and wrought titanium.

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

Thursday AM — Room: Lincoln E
March 16, 2000 — Location: Opryland Convention Center

Session Chairs: Brajendra Mishra, Colorado School of Mines, Kroll Instit. of Extract. Metallu., Golden, CO 80401 USA; Ramana G. Reddy, University of Alabama, Metallu. and Matls. Eng., Tuscaloosa, AL 335487 USA

8:30 AM

Organophosphorus Reagents in Actinide Separations: Unique Tools for Production, Cleanup and Disposal: *Kenneth L. Nash*[1]; [1]Argonne National Laboratory, Chem. Div., 9700 S. Cass Ave., Argonne, IL 60439-4831 USA

Interactions of actinide ions with phosphate and organophosphate reagents have figured prominently in nuclear science and technology, particularly in the hydrometallurgical processing of irradiated nuclear fuel. Actinide interactions with phosphorus-containing species impact all aspects from the stability of naturally occurring actinides in phosphate mineral phases through the application of the bismuth phosphate and PUREX processes for large-scale production of transuranic elements to the development of separation and environment restoration processes based on new organophosphorus reagents. In response to the need for complete actinide recovery in processing, organophosphorus extractants, aqueous complexants, and ion exchange resins have been developed over the past 15-20 years. Recently, thiophosphinic acids have been identified as potentially important reagents for lanthanide-trivalent actinide separations necessary for actinide transmutation. An overview of the unique role of organophosphorus compounds in actinide production, disposal, and environment restoration will be presented. The broad utility of these reagents and their unique chemical properties will be emphasized. Work performed under the auspices of the U.S. DOE Office of Basic Energy Sciences, Division of Chemical Sciences under contract number W-31-109-ENG-38.

9:00 AM

Characterization of Actinide-Containing Metallic High-Level Nuclear Waste Forms: *Dennis D. Keiser*[1]; Wharton Sinkler[1]; Daniel P. Abraham[2]; [1]Argonne National Laboratory-West, Nuclear Tech., P.O. Box 2528, Idaho Falls, ID 83403-2528 USA; [2]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439-4803 USA

Small amounts of U, Pu, and Np are leftover from an electrometallurgical process being developed by Argonne National Laboratory to treat spent nuclear fuels. This process is employed to extract usable uranium. Some of the residual actinides are incorporated into a metallic waste form that is destined for disposal in a geologic repository. Besides actinides, the metallic waste form consists of stainless steel, zirconium, and metals noble to the process (e.g., Tc, Ru, and Pd). The baseline alloy composition is stainless steel-15 wt.% Zr. This paper discusses the microstructural development in alloys generated from actual irradiated material that is residual from electrometallurgical treatment. Focus is given to the actinide behavior in the alloys. The actinides are found to congregate into one of the two major alloy phases, viz. an Fe2Zr Laves intermetallic. The actinides are found to segregate into specific regions of this phase, and this is probably due to the actinides favoring one Laves phase polytype over the others. Comments will be made as to why this actinide behavior is observed.

9:30 AM

Vitrified Magnesia Dissolution and its Impact on Plutonium Residue Processing: *Keith W. Fife*[1]; Jennifer L. Alwin[1]; [1]Los Alamos National Laboratory, Nuclear Matls. Tech. Div./Actinide Process Chem., P.O. Box 1663 MS-E511, Los Alamos, NM 87545 USA

Aqueous chloride operations at the Los Alamos Plutonium Facility cannot directly dispose of acidic waste solutions because of compatibility problems with existing disposal lines. Consequently, all hydrochloric acid must be neutralized and filtered prior to exiting the facility. From a waste minimization standpoint, the use of spent magnesia pyrochemical crucibles as the acid neutralization agent is attractive since this would involve taking a stream destined for transuranic waste and using it as a reagent in routine plutonium residue processing. This presentation discusses our experience in defining appropriate size reduction equipment and presents our results in using the magnesia crucibles for hydrochloric acid neutralization.