MONDAY AM

AIME/TMS KEYNOTE ADDRESS
"FutureView...A Look Ahead"
Daniel Burrus
11:30am-1:00pm
Convention Center, Presidential Ballroom

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Tutorial Luncheon Lecture
"Advanced Rechargeable Batteries: A Materials Science Perspective"
Donald R. Sadoway
12:00noon-1:30pm
Convention Center, Lincoln C

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AIME & TMS Banquet & Awards Presentation
6:00pm Reception
7:00pm Dinner
Convention Center, Presidential Ballroom

+Indicates Student

Advanced Technologies for Superalloy Affordability: Development of New Technology

Sponsored by: Structural Materials Division, High Temperature Alloys Committee
Program Organizers: K. M. Chang, West Virginia University, Mechanical & Aerospace Engineering, Morgantown, WV 26506 USA; K. R. Bain, GE Aircraft Engines, Cincinnati, OH 45215 USA; D. Furrer, Ladish Company, Cudahy, WI 53110 USA; S. K. Srivastava, Haynes International, Kokomo, IN 46904 USA

Monday AM — Room: Canal C
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Keh-Minn Chang, West Virginia University, Morgantown, WV 26506 USA; John J. deBarbadillo, Special Metals Corporation, Huntington, WV 25705 USA

8:30 AM Keynote
Initiatives for Superalloy Affordability: *Malcolm C. Thomas*[1]; Robert E. Schafrik[2]; James C. Williams[3]; [1]Rolls-Royce Allison, P.O. Box 420, Indianapolis, IN 46206-0420 USA; [2]GE Aircraft Engines, One Neumann Way, MD H85, Cincinnati, OH 45215 USA; [3]The Ohio State University, 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Modern superalloy forgings and castings have enabled significant increases in turbine engine performance, which have historically been driven primarily by military requirements. The current situation is significantly different, with reduced military budgets for propulsion, and increasing demands from both civil and military customers for affordable solutions to performance requirements. One result of this shift in emphasis has been the initiation of several programs to reduce the cost of these increasingly complex materials. This paper will review some of the work currently being undertaken both at engine manufacturers, and at component suppliers in consortia such as the Engine Supplier Base Initiatives and the Metals Affordability Initiative. The creation of such consortia to reduce component cost has made significant progress possible in focusing the entire supply chain and the customer on this critical issue of affordability

8:55 AM Invited
Application of Lean Concepts to the Aerospace Forging Value Stream: *Dan Krueger*[1]; Rod Boyer[2]; David Furrer[3]; Mary Lee Gambone[4]; Beth Lewis[5]; [1]GE Aircraft Engines, One Neumann Way, MD H85, Cincinnati, OH 45215 USA; [2]Boeing Company, Commercial Airplanes Grp., Seattle, WA 98124 USA; [3]Ladish Company, Inc., 5481 S. Packard Ave., Cudahy, WI 53110 USA; [4]Rolls-Royce Allison, P.O. Box 420, Indianapolis, IN 46206-0420 USA; [5]Wyman Gordon Company, 244 Worcester St., Grafton, MA 01563 USA

Forged components comprise approximately one-third the cost of a propulsion system, and are major cost drivers for airframe systems. Traditionally, cost reduction efforts have focused on improving individual operations or steps in the value stream. A new U.S. Air Force ManTech program, Forging Supplier Initiative, has been initiated to achieve a significant reduction in cost and cycle time through improvements across the entire Supplier-OEM value stream for forged airframe and gas turbine engine components, from raw material order to the finished, ready to install component. Lean Manufacturing, which encompasses a total waste reduction strategy based on a thorough understanding of value for all manufacturing operations in the supply chain, along with forging, machining and modeling technology improvements, must be exploited to achieve the cost and cycle time reduction goals. This paper describes the Phase I approach and progress in the Forging Supplier Initiative program being performed by the General Electric-Lean Industrial Forging Team (LIFT) Consortium. The objective of Phase I, Analysis and Definition of Cost Reduction Opportunities, is to identify cost and cycle time drivers across the entire forged product value stream for representative airframe and propulsion system forgings, and demonstrate the feasibility and payoff of projects selected to address the major drivers.

9:20 AM Invited
Cost Modeling of Forged Turbine Engine Disks: *Kong Ma*[1]; Kenneth A. Green[1]; [1]Rolls-Royce Allison, P.O. Box 420, Indianapolis, IN 46206-0420 USA

As a part of the NASA funded IDPAT (Integrated Design/Processing Analysis Technology) consortium development program, Rolls-Royce Allison was responsible to develop a cost model for forging and machining of turbine engine disks. The objective of this task was to develop a geometric feature based software design tool, which can assist preliminary design to perform trade-off studies in a qualitative fashion. The system consists of generic cost related design/manufacturing rules and allows users to define their company/process specific variables associated with these rules. The user can use a graphic user interface to sketch the design section, then the system will report the cost impact based on the design changes from the base line.

9:45 AM Invited
ATS Advanced Turbine Airfoil Manufacturing Technology Program: *Mei Ling Carolyn Henne*[1]; John Brinegar[1]; Albert Hines[1]; [1]Howmet Research Corporation, 1500 S. Warner St., Whitehall, MI 49461 USA

The purpose of the Advanced Turbine Airfoil Manufacturing Technology Program is to develop single crystal and directionally solidified casting technologies to benefit Advanced Turbine Systems (ATS) industrial and utility gas turbine engines. Traditionally, single crystal investment casting technologies have been utilized for aircraft gas turbine blades where yields of 95% and higher are commonly achieved. These yields are the result of understanding the relationship between alloy, geometry and process conditions. These relationships have been developed iteratively over the past 30 years. The application of this knowledge to Industrial Gas Turbine (IGT) sized castings has demonstrated that the technology involved must be optimized to obtain similar quality and yields of the smaller aircraft turbine counterparts. The goal of this program is to develop the technology utilized for IGT components so that casting yields, furnace up time and direct costs are impacted. This presentation will focus on the thermal resistances to heat removal from an IGT part. Addressing these resistances to heat transfer in a casting process provide guidance on where improvements can be made. Efforts have been directed toward understanding these resistances by employing finite-element-modeling (FEM). Modeling has also been utilized to determine the effects of the thermal resistances on typical IGT, single crystal microstructural defects. Additional efforts involve benchmarking the state-of-the-art in IGT production via microstructural and crystal quality evaluations. Freckle and grain-associated defects are also examined in the evaluations.

10:10 AM Break

10:25 AM Invited

Spraycast-X® for Aerospace Applications: *Thomas Tom*[1]; Greg Butzer[2]; Kim Bowen[3]; [1]Howmet Research Corporation, 1500 S. Warner St., Whitehall, MI 49461-1895 USA; [2]Spraycast Technologies International, L.L.C., Whitehall, MI 49461-1895 USA; [3]Cannon-Muskegon Corporation, Muskegon, MI 48441 USA

Howmet Corporation has licensed and modified the Osprey®, or spray forming process to produce high quality nickel-base superalloys for gas turbine engine applications. The modified process, known as the "Spraycast-X®" process, combined vacuum induction melting technology with high purity argon gas atomization to produce ring and case preform products. This process yields a product with a fine grain homogeneous microstructure, improved machinability, ability to process previously non-forgeable alloys, and an economic benefit that indicate savings of up to 30% over its ring rolled counterpart. Additionally, the Spraycast-X® process results in lead-time reductions of up to 75% over ring rolled components. A description of the process, along with data that substantiates these claims, will be presented.

10:50 AM Invited

Enhanced Powder Metallurgy (P/M) Processing of UDIMET® 720 AE1107C (T406) Turbine Disks: *Gary A. Miller*[1]; Kenneth A. Green[2]; Tony Banik[3]; Joseph Lemsky[4]; [1]Concurrent Technologies Corporation, 1450 Scalp Ave., Johnstown, PA 15904 USA; [2]Rolls-Royce Allison, P.O. Box 420, Indianapolis, IN 46206-0420 USA; [3]Special Metals Corporation, 100 Industry Lane, Princeton, KY 42445 USA; [4]Ladish Company Inc., 5481 S. Packard Ave., Cudahy, WI 53110-8902 USA

Enhanced powder metallurgy (P/M) processing of Udimet 720 has been evaluated as an approach for improving the quality, and concurrently, reducing the cost of AE1107C (T406) turbine disks. Enhanced P/M processing combines several technologies: high-yield production of fine powder (-270 mesh); low extrusion ratio (3:1) for converting as-HIPed material to billets; isothermal forging of disk preforms to near-net shape; and selective ultrasonic inspection. Having achieved acceptable mechanical properties, the viability of this combination of technologies depends upon component performance in cyclic spin and spin burst tests and engine tests as well as verification of projected cost savings. Results obtained to date confirm the acceptability of material produced using this combination of technologies. Technology implementation hinges on the component and engine test results and completion of the cost/benefit analysis which remain to be performed. Technology assessment activities including defect migration and flow modeling; residual stress measurement, modeling, and mitigation approaches; HIP modeling; and seeding are discussed.

11:15 AM Invited

The Castability and Mechanical Properties of Nickel Superalloys Cast Using Thermally Controlled Solidification: *Sanjay Shendye*[1]; M. L. Gambone[2]; Paul P. Andrews[3]; Michael Tims[4]; [1]PCC Structurals Inc., 4600 S.E. Haney Dr., Portland, OR 97206 USA; [2]Rolls-Royce Allison, P.O. Box 420, Indianapolis, IN 46206-0420 USA; [3]Rolls-Royce, plc, P.O. Box 31, Derby DE248BJ UK; [4]Concurrent Technologies Corporation, 1450 Scalp Ave., Johnstown, PA 15904 USA

Thermally Controlled Solidification (TCS), a casting process patented by PCC Structurals, has potential to greatly reduce the cost of aerospace structural castings. The response of three nickel superalloys; INCO 718, INCO 939 and RS5; to casting via TCS was studied as part of a program supported by the National Center for Excellence in Metalworking Technology. (RS5 is a superalloy developed a by Rolls-Royce, plc.) The alloys were compared by such characteristics as the ability to fill thin-wall sections, propensity to hot tear, and weld reparability. TCS microstructures were analyzed, and mechanical properties; including tensile, creep, fatigue and damage tolerance; were measured for each of the TCS cast alloys. The castability evaluation plan will be presented in this paper as well as the results and their implication.

Deformation and Stress During Solidification

Sponsored by: Materials Processing and Manufacturing Division, Solidification Committee, Jt. Processing Modeling Analysis & Control Committee, Shaping and Forming Committee

Program Organizers: Brian G. Thomas, University of Illinois, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Christoph Beckermann, University of Iowa, Department of Mechanical Engineering, Iowa City, IA 52242 USA; Matthew J.M. Krane, Purdue University, School of Materials Engineering, West Lafayette, IN 47907 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Monday AM — Room: Johnson A/B
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Brian G. Thomas, University of Illinois, Mech. & Industrial Eng., Urbana, IL 61801 USA; Christoph Beckermann, University of Iowa, Dept. of Mech. Eng., Iowa City, IA 52242-1527 USA; Matthew J. Krane, Purdue University, School of Matls. Eng., West Lafayette, IN 47907 USA

8:30 AM

An Experimental Study of the Deformation of a Directionally Solidified SCN-ACE Alloy Mush: Ernandes M.S. Rizzo[2]; Venkatesh Raghavendra[1]; Houfa Shen[1]; *Christoph Beckermann*[1]; [1]University of Iowa, Dept. of Mech. Eng., 2412 SC, Iowa City, IA 52242 USA; [2]Centro Federal de Educacao Tecnologica do Espirito Santo, C.P. 5139, Vitoria, ES 29040-333 Brazil

The deformation of a directionally solidified mush of a succinonitrile-acetone (SCN-ACE) alloy is studied experimentally. This alloy not only solidifies dendritically like a metal alloy, but also has mechanical properties that are similar to those of metals near the melting point. A test cell was designed that allows for the directional solidification of the alloy and the controlled compression of the mush that forms. Measurements during the deformation include the local displacement and deformation rate of the solid, the flow patterns in the liquid, temperatures, and liquid concentrations. Results are presented for a

range of initial compositions, cooling rates, and deformation amounts. The measurements are suitable for validation of future models. The experiments are relevant, for example, to the deformation of a partially solidified strand during continuous casting of steel slabs.

8:50 AM

Relation Between Tensile and Shear Strengths of the Mushy Zone in Solidifying Aluminium Alloys: Stephen S. Instone[1]; Taro Sumitomo[1]; *Arne K. Dahle*[1]; [1]The University of Queensland, Dept. of Ming., Mins. and Mats. Eng., Brisbane, Qld 4072 Australia

Strength measurements reported in the literature show a significant deviation in results for semi-solid material tested in shear and in uniaxial tension. This therefore raises the question of whether different deformation mechanisms apply and which are the important with respect to predicting the formation of various defects, such as hot tearing, surface cracking and burst feeding. In this work, the strength development during solidification of various aluminium alloys has been measured with two different techniques-horizontal tensile testing and direct shear cell testing. The strength results from the two methods correspond to a much higher degree than suggested by the results presented in the literature. Constitutive equations for the mechanical properties of the mushy zone over the whole solid fraction regime, ie. from coherency to complete solidification, can be developed based on these strength measurements. These equations can be used for the prediction of stress development as well as defect formation. An important component of the analysis of the mechanical properties of the mushy zone is the fraction of the cross-sectional area that is capable of supporting an applied load through the formation of a continuous solid network. Consequently, the formation of solid-solid bridges and their contribution to the strength of the mushy zone was investigated by comparing mushy zone strength to measurements of tensile strength at temperatures just below the solidus temperature. This information was then used to estimate the fraction of the cross-sectional area of the mushy zone where solid-solid bridging had occurred.

9:10 AM

Tensile Deformation Behaviour at Above Solidus Temperatures in Aluminium Alloy 5083: *John Anthony Spittle*[1]; Jonathan James[1]; Stephen G.R. Brown[1]; Michael E. Keeble[1]; [1]University of Wales, Matls. Eng. Dept., Singleton Park, Swansea, Wales SA28PP UK

A tensile testing technique has been developed specifically for examining true stress/true strain behaviour at above solidus temperatures. ASSET (the Advanced Semi-Solid Elongation Test) applies AC Joule heating to rapidly reheat specimens from DC-cast ingots to the test temperatures. Specimen and grip geometries are designed using a finite difference thermal model, such that a test temperature is maintained at ~+/-2 degrees K over a gauge length of 20mm. Strain measurements are carried out using a non-contact linescan camera method. The fraction liquid at a given test temperature is determined using a thermodynamic model assuming Scheil non-equilibrium freezing. The test has been applied to the study of 5083 aluminium alloy at several test temperatures and crosshead movement rates. The data has been evaluated and plotted as true stress/true strain and the curves have been analysed using a simplex approach. The influences of fraction liquid, strain rate, strain and microstructure on deformation characteristics are described and discussed.

9:30 AM

Mechanical Behavior of Carbon Steels in the Temperature Range of Mushy Zone: *Dong Jin Seol*[1]; *Young Mok Won*[1]; *Tae-jung Yeo*[1]; *Kyu Hwan Oh*[1]; [1]Seoul National University, Matls. Sci. and Eng., San 56-1 Shinrim-dong, Lab. of Matls. Deformation and Processing, Seoul 151-742 Korea

Tensile strength and ductility of carbon steels have been measured in the temperature range of mushy zone by the in-situ melting tensile test technique with Gleeble system. The specimen was melted and cooled to the test temperature before the tensile deformation in order to get the mechanical properties subject to the continuous casting process. During hot tensile test, a ceramic fiber tube was used to reduce the radial temperature gradient in the heated specimen. Tensile strength of carbon steels in the temperature range of mushy zone increased with decreasing test temperature, and was well described by the modified yield criterion for porous metals. The measured zero strength temperature (ZST and zero ductility temperature) (ZDT) were related to the solid fractions evaluated by the numerical simulation of microsegregation. The characteristic solid fractions of ZST and ZDT which corresponded to 0.75 and 0.99, respectively, were well described by the prediction equation on ZST and ZDT at given steel compositions and cooling rates. KEY WORDS: mushy zone; zero strength temperature (ZST zero ductility temperature) (ZDT).

9:50 AM Break

10:20 AM

A New Hot Tearing Criterion: Application to DC Casting of Aluminum Alloys: *Jean-Marie Drezet*[1]; Michel Rappaz[1]; [1]Laboratoire de Metallurgie Physique, Ecole Polytechnique Federale de Lausanne, MX-G, Lausanne CH-1015 Switzerland

Hot tear is one of the most serious defects which a casting can suffer. It represents a major limitation to the production of foundry cast parts and to the productivity of continuous casting processes such as the direct chill casting of aluminum alloys. As an example, the casting speed of the direct chill casting of billets is limited for some aluminium alloys because of their high propensity to develop hot tears which initiate at non zero liquid fraction at the bottom of the sump. In order to predict the occurrence of hot tears in solidifying parts, a hot tearing criterion based on the ability of the interdendritic flow of liquid to compensate for the thermally-induced deformation of the roots of dendrites has been recently derived by Rappaz, Drezet and Gremaud. Based upon a mass balance performed over the liquid and solid phases, this criterion accounts for the deformation of the solid skeleton perpendicularly to the growing columnar dendrites and for feeding of the interdendritic liquid: it allows the calculation of the maximum strain rate that the roots of the dendrites can undergo without initiation and/or propagation of hot tears. The present paper gives a summary of the main features and assumptions of the new hot tearing criterion. The equations defining the hot cracking sensitivity index in the particular case a thermally-induced deformation rate which is uniform in the mushy zone are presented. Then, the model is applied to the particular case of the DC casting of billets of aluminum alloys. The maximum strain rate sustainable by the mushy zone is derived at the bottom of the sump and in the primary cooling zone. It is demonstrated that the bottom of the sump is more sensitive to hot tearing than the primary cooling zone, thus limiting the casting speed and therefore the productivity of the process.

10:40 AM

Application of Solidification-Stress Model to Predict Critical Shell Thickness for Breakouts During Continuous Casting of Steel: Chunsheng Li[1]; *Brian G. Thomas*[1]; [1]University of Illinois, Mech. & Industrial Eng., 140 Mech. Eng. Bldg., 1206 W. Green St., Urbana, IL 61801 USA

During continuous casting, excessive gap formation in the mold can lead to a locally thin solidified shell with a higher surface temperature. This can cause a breakout if the strength of the shell is insufficient to withstand the ferrostatic pressure at mold exit. A finite element model has been developed to simulate thermal and mechanical behavior of the solidifying shell during continuous casting of steel both in and below the mold. It features an elastic-viscoplastic creep constitutive equation that accounts for the different responses of the semi-solid, delta-ferrite, and austenite phases. The model is applied to predict temperature, stress, and strain in a section through the steel shell cast under conditions that lead to varying degrees of shell growth and surface cooling. At mold exit, ferrostatic pressure is applied and the mechanical response is predicted. The results suggest critical conditions that lead to excessive strain and failure of the shell for different steel grades.

11:00 AM

Cracking Phenomena of AISI 304 Stainless Steel Produced by Twin Roll Strip Casting Process: D. K. Choo[1]; *S. I. Jeong*[1]; M. J. Ha[1]; S. H. Kim[1]; S. Lee[2]; [1]Research Institute of Industrial Science & Technology, Strip Casting Project Team, P.O. Box 135, Pohang, Kyongbuk 790-600 Korea; [2]Pohang University of Science & Technology, Matl. Sci. & Eng., San 31 Hyojadong, Pohang, Kyongbuk 790-600 Korea

Cracks occurring on the surface of AISI 304 stainless steel produced by strip casting process was investigated and the causes for their occurrence were postulated. The sections of as-cast strip near the cracked region were cut and polished to reveal microstructural change, and the cracked region was fractured to investigate the location of crack initiated area using optical and scanning electron microscopes. The fractography showed that the crack was initiated at the tip of dendrite of solidified shell and propagated along the path to the segregated liquid film between primary dendrites and the fractured surface was heavily oxidized from atmosphere, as this was typical in solidification cracking at high temperature. The reason for the occurrence of solidification cracking in the strip casting process was uneven stress distribution in the solidified shell during solidification: when the local tensile stress in the delayed solidification region exceeded the critical strain limit of the shell at high temperatures, the crack would be formed at delayed solidified shell. The causes for the uneven solidification were local heat transfer barriers on roll surface, such as contaminated oxide debris on roll surface, oxide scum entrapped at meniscus and melt level fluctuation. The tendency of crack occurrence was also dependent on the chemical composition of molten steel and the cooling rate of the solidified strip.

11:20 AM

Effect of Gap Distance on the Cooling Behavior and Microstructure of Indirect Squeeze Cast and Gravity Die Cast 5083 Wrought Al Alloy: *Jong Hyeon Lee*[1]; Hyoung Seop Kim[1]; Chang Whan Won[1]; Seong Seock Cho[1]; Byong Sun Chun[1]; [1]Chungnam National University, Rapidly Solidified Matls. Rsch. Ctr., 220 KungDong, Taejon 305-764 Korea

An indirect squeeze casting process applied to a wrought Al alloy (Al-4.7Mg-0.7Mn) was investigated experimentally and numerically. A two-dimensional finite element computer code for fully coupled heat transfer and deformation analysis, ABAQUS, was used to simulate the cooling curves obtained from the experiments. Thermal contraction of the material during solidification creates a cavity between the mold and the cooling material. The formation of this cavity is explained using the calculated results. The experimental and predicted results are discussed in conjunction with the relationships between the cooling rate, microstructure, die geometry and applied pressure. The effect of applied pressure on the macrosegregation is also discussed.

11:40 AM

Computer Based Analysis of Thermal Stresses for Continuous Steel Casting Rate Increase: *Alexey N. Lozhko*[1]; *Viktor M. Olshanski*[1]; *Vladimir I. Timoshpolski*[2]; [1]State Metallurgical Academy of Ukraine, Thermal Eng. Dept., 4 Gagarin Prosp., Dniepropetrovsk UA 320635 Ukraine; [2]Byelorussian State Politechnical Academy, 2 Kondrat Krapiva Str., Minsk BA 220117 Belarus, Republic

The maximum increase in continuous steel casting rate is predetermined by economical reasons. Thermal stresses acting as the factors limiting the process, arise as a result of cooling rate inconsistency between different ingot points. Underestimation of this effect brings forth the appearance of inner cracks or residual stresses. By changing the crystallizer profile it is possible to sufficiently diminish thermal stresses effect on casting rate. The experimental research of thermal stresses during solidification is very costly (in Ukraine). That is why adequate computer modeling of the process is becoming vital. The conjugate 3-D thermo-mechanical state model of the bar-crystallizer system was the basis of the multi-fold analysis of the process. The paper presents the crystallizer profiles computed for certain occurances and the results of their experimental implementation.

Dislocations and Microscale Plasticity Modeling: Theory and Modeling of Dislocations

Sponsored by: ASM International: Materials Science Critical Technology Sector, Materials Processing and Manufacturing Division, Structural Materials Division, Jt. Mechanical Behavior of Materials, Jt. Computational Materials Science & Engineering

Program Organizers: Elizabeth Holm, Sandia National Laboratories, Albuquerque, NM 87185-1411 USA; Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA; Jeffrey Rickman, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18105-3195 USA; David J. Srolovitz, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Vaclav Vitek, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA

Monday AM — Room: Lincoln A
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Vaclav Vitek, University of Pennsylvania, Materials Science & Engineering, Philadelphia, PA 19104 USA

8:30 AM Invited

Dynamic Simulation of Dislocation Microstructures: *Richard LeSar*[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, P.O. Box 1663, Los Alamos, NM 87545 USA

One approach to bridge the gap between the atomistic scale and macroscopic materials response is a simulation in the intermediate, or mesoscopic, regime that focuses on the collective properties of the defects. For problems in materials deformation, a promising approach is dislocation dynamics, in which the dynamic motion and patterning of the dislocations themselves are simulate. There are a number of challenges in implementing dislocation dynamics, including the accurate treatment of the long-range interactions, the non-linear-elastic short-range interactions, etc. We will briefly review progress to date. We will then present results from a recently-developed three-dimensional method for calculating dislocation microstructures based on Monte Carlo and Kinetic Monte Carlo approaches. The advantages and disadvantages to this approach relative to the more standard dislocation dynamics simulations will be discussed. Time permitting, we will present an alternative approach to simulating dislocation microstructures that yields coarse-grained structures and properties.

9:10 AM

Three-Dimensional Dislocation Dynamics Simulations of Stacking Fault Tetrahedra Formation using Anisotropic Elasticity: *Moono Rhee*[1]; Brian D. Wirth[1]; James S. Stölken[1]; [1]Lawrence Livermore National Laboratory, Chem. and Matls. Sci. Directorate, P.O. Box 808, L-356, 7000 E. Ave., Livermore, CA 94551 USA

The formation of Stacking Fault Tetrahedra (SFT) in Face-Centered Cubic (FCC) metals is studied using both Molecular Dynamics (MD) based on embedded atom method potentials and Dislocation Dynamics (DD) simulations. MD simulations suggest that SFT form via the spontaneous decomposition of Frank partial dislocation loops into stair-rod partial dislocations and Shockley partial dislocations, which subsequently glide to form the SFT; as initially proposed by Silcox and Hirsch. Three-dimensional DD simulations utilizing fully anisotropic elasticity are performed to study the influence of disloca-

tion mobility, stacking fault energy, and elastic anisotropy on the formation mechanism for a variety of FCC metals. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

9:30 AM

Simulation of Equilibrium Distribution of Dislocation Structures in Bcc Single Crystals under Cyclic Loading: *S. B. Biner*[1]; J. R. Morris[1]; [1]Ames Laboratory, Iowa State University, Metallu. and Ceramics, Ames, IA 50011 USA

Collective motion of large number of discrete edge dislocations in bcc single crystals under cyclic loading is investigated using a numerical method that combines the finite element method and multi pole expansion algorithm. The dislocations are modeled as line defects in a linear elastic medium. At each instant, superposition is used to represent the solution in terms of the infinite-medium solution for the discrete dislocations and a complementary solution that enforces the boundary conditions. Annihilation of dislocations, generation of new ones and dislocation pinning at obstacles are simulated through a set of constitutive models. The evolution of Bauschinger-effect and hardening and softening behavior is correlated with the cyclic load levels and the parameters of the constitutive model for the collective behavior of dislocations. The details of the implementation of the numerical method for parallel computing in a cluster environment will also be elucidated. This work was performed for the United States Department of Energy by Iowa State University under contract W-7405-Eng-82.

9:50 AM

Kinetics of Slip and Plasticity Including a Distribution of Obstacle Sizes and Load Shedding: *Glenn S. Daehn*[1]; [1]The Ohio State University, Dept. of Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

Over the past few decades there has been much interest in describing plastic deformation as a classical kinetic problem of the thermal activation of a dislocation over a barrier, where an external stress provides a driving force tending to push the dislocation over an obstacle. The approach almost invariably implicitly assumes that the obstacles are of a single uniform size and shape and the driving force at each obstacle is also fixed. These are both clear oversimplifications. Presently the effects of a spectrum of obstacle sizes as well as slip-induced load shedding will be studied and are shown to produce significant changes in the modeled material behavior. A stochastic cellular automaton model is used to construct the model. One of the most exciting aspects of this approach is that using only very simple and reasonable assumptions many commonly-observed creep and plasticity phenomena (such as anelastic backflow and power-law strain-time creep transients) naturally emerge and aspects of this behavior can be correlated to the assumptions in a given model.

10:10 AM Break

10:30 AM

Study of the Dislocation Cores in Bcc Transition Metals Using Bond-Order Potentials: Importance of Directional Bonding: M. Mrovec[1]; R. Porizek[1]; D. Nguyen-Manh[2]; D. G. Pettifor[2]; M. Sob[3]; *V. Vitek*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. and Eng., 3231 Walnut St., Philadelphia, PA 19104 USA; [2]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX13PH UK; [3]Institute of Physics of Materials, Academy of Sciences, Zizkova 22, Brno, Czech Republic

The core structure of screw dislocations in the 4d (Nb and Ta) and 5d (Mo and W) transition metals is studied by computer simulation using the recently constructed bond-order potentials based on the real-space parametrized tight-binding method. In this framework the energy consists of the bond part that comprises contributions of d electrons, the central-force many-body part that reflects the environmental dependence of overlap repulsion arising from the valence sp electrons and a repulsive pair-wise contribution; the calculations scale linearly with the system size. The potentials have been tested for the accuracy and transferability by evaluating the energy differences of alternate structures and investigating several transformation paths and comparing these calculations with the ab initio results. The study begins with calculations of the gama-surfaces and is followed by simulations of the structures of screw dislocations. The results are compared with analogous studies made using the central-force many-body potentials of the Finnis-Sinclair type. This comparison enables us to assess the importance of directional bonding on the dislocation core structure and thus mechanical behavior of the 4d and 5d transition metals. This research was supported in part by the Advanced Strategic Computing Initiative of the U.S. Department of Energy through LLNL, grant no. B331542 (MM and VV).

10:50 AM

The Barrier Strength of Mismatch Dislocations: *Satish I. Rao*[1]; Peter M. Hazzledine[1]; [1]UES Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

In order for layered materials to yield, mobile dislocations must be able to cross the layer interfaces. The barrier to motion through the interface contains several components. In semicoherent multilayers one of the most significant components is the set of mismatch (van der Merwe) dislocations. The separation of these dislocations, d, depends on the lattice parameter mismatch and the degree of coherency, hence on the layer thickness. The barrier stress has two closely related parts, first the requirement to bow the mobile dislocation between the mismatch dislocations and second, the requirement to leave a 'difference' dislocation at the interface. The paper describes EAM atomistic simulations designed to test and refine the analytical estimate for this stress of a Gb/d.

11:10 AM

Systematic Analysis of Dislocation Junction Reactions in Bcc Metals Using Anisotropic Elasticity: *James S. Stölken*[1]; [1]Lawrence Livermore National Laboratory, Chem. and Matls. Sci. Directorate, P.O. Box 808, L-356, 7000 E. Ave., Livermore, CA 94551 USA

Anisotropic elasticity calculations were performed to estimate the intrinsic strength of dislocation junction reactions in both Group VB-VIB transition metals, and Iron. A dislocation phase space description is used to systematically categorize junction formation reactions for dislocations on {110}, {112}, and {123} slip planes: identifying <24 unique junction reactions that comprise the 1176 possible reactions. Regions in which repulsive or neutral dislocation reactions (i.e. jogs) may occur are also identified. The calculations form a critical link between microscale phenomena of dislocation interaction and the formulation of mesoscopic models of crystal plasticity by establishing a taxonomy of dislocation interactions. The resulting catalog of dislocation reactions indicates the relative strength of dislocation junctions, imposes certain symmetry restrictions for physics based hardening models, and suggests specific latent hardening experiments to measure the influence of junction formation on the work hardening of BCC metals. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

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Primitive Dislocation Loops in Face-Centered Cubic Crystals: *Craig S. Hartley*[1]; [1]U.S. Department of Energy, Office of Science, SC-131, Germantown, MD 20874 USA

The effect of crystal structure on atomic displacements near the dislocation core cannot be included in local continuum models of dislocations. Atomic models of dislocations using appropriate interatomic potentials can provide this information, subject to limitations imposed by the choice of interatomic potential and boundary conditions on the atomic array surrounding the dislocation line. An intermediate approach, the simulation of dislocations by non-local force arrays, shows promise as a method for modeling the atomic arrangements near dislocations and other crystal defects. This method relies on the definition of a "primitive dislocation loop", a non-local continuum analogue to the infinitesimal dislocation loop of local continuum theory. The primitive loop consists of an array of forces applied to the nearest neighbors of an atom. Magnitudes and directions of the forces are chosen so that the far-field displacement field of the array is identical to that of an infinitesimal dislocation loop centered on the atom. The locations of the forces are determined by the crystal structure of the medium, i.e. by the coordination group surrounding an atom. Dislocation lines can then be constructed by superposition of these primitive loops to form various shapes. Since the singular character of the force array is concentrated in the points of application of the forces, whose displacements can be estimated from atomic force constants, there is no singularity in stress or strain at the core of the

dislocation. This model has been employed to determine the displacement fields and energies of dislocations, kinks and jogs in the case of an elastically isotropic, simple cubic lattice, but extension to real structures relies on the construction of appropriate primitive loops. Such loops have been constructed for the body-centered cubic lattice in anisotropic crystals. The present work extends this construction to the face-centered cubic lattice and describes the technique for determining properties of finite dislocation configurations.

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Self-Consistent Modeling of Polycrystal Plasticity: B. Clausen[1]; *Carlos N. Tome*[1]; F. Jean-Prost[1]; M. A.M. Bourke[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

The utilization of self-consistent modeling (SCM) of polycrystalline plasticity to predict internal and residual stresses in structural materials has increased significantly in the last decade. The constitutive equations for elastic-plastic and visco-plastic self-consistent schemes will be presented and the advantages and limitations of the models will be discussed. At Los Alamos National Laboratory the SCM of several materials has been correlated with in situ neutron diffraction measurements of internal and residual elastic lattice strains. Furthermore, the SCM scheme has been incorporated into finite element codes enabling predictions of complex non-uniform loading and arbitrary geometries. We also discuss the use of SCM as a tool for interrogating the microstructural mechanisms, such as type and characteristics of slip and twinning systems, and their interactions.

General Abstracts: Aluminum and Texture

Sponsored by: TMS

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

Monday AM — Room: Knoxville A
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Mike O'Brien, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA

8:30 AM

Comparison of Microstructure and Texture of AA3XXX Direct Chill Cast Ingot and Strip Cast Slab: J. T. Liu[1]; Y. Liu[1]; *J. G. Morris*[1]; [1]University of Kentucky, Light Metals Resrch. Labs., Dept. of Chem. and Mats. Eng., 177 Anderson Hall, Lexington, KY 40506 USA

Direct chill (DC) ingot and strip cast (SC) slab present significantly different features in microstructure and texture. Both the microstructure and texture of AA3105 DC ingot and AA3015 SC slab were observed in this work for the purpose of determining the difference in microstructure and texture between DC ingot and SC slab. The results show that both the DC ingot and SC slab present a random texture and nearly the same grain structure, however, the dispersion of particles and the solute supersaturation condition of the alloying elements for DC ingot and SC slab are very different. Consequently, the dispersion of particles and the solute supersaturation condition of the alloying elements significantly contribute to the great difference in microstructure and texture evolution between DC ingot and SC slab in the following thermomechanical processing of these materials.

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Earing Behavior of AA6010 Aluminum Alloy with Different Preferred Orientations: Yansheng Liu[1]; Jiantao Liu[1]; Xiang-Ming Cheng[1]; *James G. Morris*[1]; [1]University of Kentucky, Light Metals. Resrch. Labs., Dept. of Chem. and Mats. Eng., 117 Anderson Hall, Lexington, KY 40506 USA

Texture inhomogeneity through the thickness has been found in AA6010 aluminum alloy hot band. Annealing does not remove the inhomogeneity but produces different textures. In order to understand the earing behavior of the materials, samples for earing test were prepared by slicing the original hot band in planes parallel to the rolling plane. Samples with the typical texture at the midplane of the hot band and with the textures at the surface of the hot band were obtained separately. Earing of both the as received samples and annealed samples were measured. The results show that earing of different samples and annealed samples were measured. The results show that earing of different samples are typically controlled by their textures. However, earing of the original hot band can not be simply designated as the average of the several sliced samples.

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Anisotropy and Texture Evolution of Cold-Rolled Al-Mg Alloys: *Seiichi Hirano*[1]; Masaru Nomura[1]; [1]Sumitomo Light Metal Ind., Limited, Rsch. and Dev. Ctr., 1-12 3-chome, Chitose Minato-ku, Nagoya, Aichi 455-8670 Japan

Texture evolution which leads to the earing behavior of a drawn cup of cold-rolled Al-Mg alloy sheet was investigated by two different process materials, with and without intermediate annealing during cold rolling. The samples without intermediate annealing had 6 or 8 ears on the cups, while ones with intermediate annealing had 4 ears. Ears at 0° and 180° to the sheet rolling (RD) are related to the intensity of the Cube and RD rotated Cube texture. 45° ears were related to beta fiber components of the rolling texture, and the ratio of Brass and Cu component intensities along the beta fiber was different between with and without intermediate annealing samples. Texture and microstructure changes associated to the intermediate annealing conditions are also discussed.

9:30 AM

Analysis of Tensile/Compressive Deformation in Zirconium Bent Beams: *G. C. Kaschner*[1]; T. A. Mason[1]; J. F. Bingert[1]; P. J. Maudlin[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Previous work has demonstrated that the deformation mechanisms of this highly anisotropic low-symmetry metal may be dominated by either slip or twinning depending on strain rate, temperature, and orientation. In the current study, we have loaded basal fiber textured zirconium beams of two orientations in 4-point bending to produce a continuous stress-strain gradient across the thickness of the sample. The macroscopic shape of the cross-sections was used to validate predictive modeling efforts. The predictive modeling is based on an anisotropic yield criterion that is a function of texture and deformation mechanism, ie., slip or twinning. Automated EBSP methods were used to analyze twinning deformation modes as a function of position, and hence sign and magnitude of strain, in the beam. These measurements corroborated the basis for constructing our yield function. Research supported by the US DOE, Basic Energy Sciences, Division of Materials Sciences.

9:50 AM

The Cooling and Solidification Behaviour of Aluminum and Aluminum Alloys Weld: *Kimioku Asai*[1]; Eisaku Tokuchi[1]; [1]Musashi Institute of Technology, Tokyo, Japan

Experiments on weld solidification were successfully carried out in TIG arc spot welding of thin plate. This second research was conducted to establish a practical, convenient method for evaluating the hot cracking susceptibility in Al-alloys weld. In addition to the temperature measurement in welding thermal cycle, the behaviour of the liquid film solidifying in the grain boundary was investigated by revealing dynamically the liquid films in breaking surfaces with a newly developed high-speed breaking machine, and by observing them prudently with SEM. All those results including on solidification rate, cooling rate, temperature gradient, and fraction solid were quantitatively gath-

ered in a diagram named CCSP (Continuous Cooling Solidification Process) diagram. The result of the Varestraint Test carried out to make hot cracking occur was also considered with the same diagram. This series of operation enabled us to discuss the cooling and solidification behaviour of weld in relation to the hot cracking susceptibility.

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Fatigue Crack Initiation in Aluminum Alloys: *Peter S. Pao*[1]; Steven J. Gill[1]; Jerry C. Feng[1]; [1]Naval Research Laboratory, Washington, DC 20375 USA

Fatigue crack initiation from pre-existing corrosion pits of 7075 and 7050 alloys was investigated using blunt-notched WOL specimens. The presence of corrosion pits not only reduces the fatigue crack initiation lives but also lowers the fatigue crack initiation thresholds by half. The effect of aging was studied by comparing the fatigue crack initiation of peakaged 7075-T651 to that of overaged 7075-T7351. At high stresses, 7075-T651 exhibits longer fatigue initiation lives than does 7075-T7351. However, at lower stresses, the difference between the fatigue crack initiation diminishes. The effect of orientation was investigated by comparing the fatigue crack initiation of SL 7075-T651 to that of ST 7075-T651. At high stresses, SL 7075-T651 demonstrates longer fatigue initiation lives than does ST 7075-T651. Again at lower stresses, such differences disappear. Fatigue crack initiation and identification of initiation sites and mechanisms are discussed.

10:50 AM

Improvements of 6063 Al-Alloy for Rapid Extrusions and Possibility of Reducing Their Tails: *Abdel Haleem Ghaneya*[1]; [1]Assiut University, Mining and Metallu. Dept., Egypt

This study was applied on an industrial scale of EgyptAlum Company It is principally divided into two main branches. The first is concerned with the transformation of beta phase into alpha phase, and at the same time, the dissolution of Magnesium silicide precipitate. Both processes lead to ease the fabricability of the alloy by extrusion, even at a rapid rate. The second branch deals with the possibility of reducing ingot tails. Manufacturers require ingots of 6063 free of non-metallic inclusions of both ends. Our trial was carried out to achieve minimum non-metallic inclusions number and size, as well as their distribution per unit area at both ends of ingot. According to the first branch, the temperature of homogenization was found to be 580°C for holding time of 12 hours. This is sufficient for obtaining the alpha phase and for the dissolution of Magnesium silcide precipitate. Cooling was performed by using three fans, each providing 60000 meter cube of air per hour. By this specific cycle (heating, holding, cooling), the beta phase transformation and magnesium silicide precipitation and its coalescence are prevented. As for the second branch, our trials give inconsistent results. Nevertheless 20% of the removed part can be saved.

11:10 AM

Characterization of Oriented Structure in Copper Electrocoatings: Yuliya O. Proshenko[1]; Evgenii P. Kalinushkin[1]; *Oleg B. Girin*[2]; [1]State Metallurgical Academy of Ukraine, Dept. of Phys. Metallu., Prospekt Gagarina 4, Dnipropetrovsk 320635 Ukraine; [2]Ukrainian State University of Chemical Engineering, Dept. of Matls. Sci., Prospekt Gagarina 8, Dnipropetrovsk 320005 Ukraine

A comprehensive study into structure evolution in copper electrolyte coatings at the stages of nucleation and growth of the major component of texture was carried out using nonconventional X ray diffraction techniques in combination with scanning electron microscopy and electron probe analysis. Following thermodynamic considerations were found to exert dominating influence on texture formation in copper electrocoats, namely grain boundary energy alone at the nucleation stage, and grain boundary energy on equal terms with bulk energy at the grain growth stage. Anisotropy of oriented structure was observed even at the texture formation onset and involved not only element size but also element shape. Thus for the major component the nuclei height was almost four times greater than the average of the two other dimensions. A comprehensive analysis of evolution of texture, substructure, microstructure and surface morphology in the course of copper layer growth revealed that the oriented structure of copper electrocoats features texture-related effects of anisotropy of substructure and nonhomogeneity of granular structure.

General Abstracts: Ferrous and Refractory Metals

Sponsored by: TMS

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

Monday AM — Room: Canal A
March 13, 2000 — Location: Opryland Convention Center

Session Chair: George Spanos, Naval Research Laboratory, Washington, DC 20375-5000 USA

8:30 AM

Measurement of Burst Disc Properties Using Nanoindentation: *Neville R. Moody*[1]; Steven L. Robinson[1]; Mike Chiesa[1]; [1]Sandia National Laboratories, P.O. Box 969, MS9403, Livermore, CA 94551-0969 USA

Stainless steel burst discs are used in many high hydrogen pressure applications to insure safe operation. These discs are formed to a dome-like geometry and scored to produce a thin-walled, high strength ligament beneath the score. However, little is known about how structure and properties in this ligament control burst pressure. As a consequence we used nanoindentation to measure properties and finite element analysis to map stresses and strains in a 316L burst disc. The nanoindentation results showed a more than two-fold increase in strength due to scoring in good agreement with finite element results obtained using a strain hardening relationship to describe stresses in 316L. These results strongly suggest that the burst pressure is controlled by dislocation interactions. The test techniques and results will be discussed and used to show how a materials and mechanics approach can provide accurate measures of small volume properties.

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Void Nucleation at Inclusions in Ultra-High Strength Steels: *Luana E. Iorio*[1]; Warren M. Garrison[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. and Eng., Pittsburgh, PA 15213 USA

We have investigated the effects of carbon content on the strength and toughness of AF1410 type steels when the sulfur has been gettered as either chromium sulfide or as titanium carbosulfide. After tempering at 510°C for five hours the yield strengths were about 1500MPa, 1600MPa and 1710MPa for carbon levels of 0.16, 0.20 and 0.25wt.%, respectively. At a carbon level of 0.16wt.% the Charpy impact energy is about 180J when the sulfur is gettered as particles of titanium carbosulfide and about 100J when the sulfur is gettered as particles of chromium sulfide. As the carbon and yield strength increase the toughness decreases much more rapidly when the sulfur is gettered as titanium carbosulfide than when the sulfur is gettered as chromium sulfide which could be due to the void nucleation resistance of the titanium carbosulfide particles decreasing with increasing matrix strength while the particles of chromium sulfide have low resistance to void nucleation resistance at all strength levels. In this work the void nucleation resistance has been determined for titanium carbosulfide and chromium sulfide particles at strength levels ranging from about 1500MPa to 1710MPa. This work was funded by the Division of Materials Research of the National Science Foundation.

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Use of Metallic-Glasses in MoSi2-Stainless Steel Joining: *Rajendra U. Vaidya*[1]; Partha Rangaswamy[1]; [1]Los Alamos National Laboratory, MST-6, MS G770, Los Alamos, NM 87545 USA

Residual stresses due to mismatch in elastic and thermal expansion properties in ceramic-metal joints can lead to failure at the interface or within the brittle ceramic. Low temperature brazing techniques coupled with ductile interlayers alleviate this problem. However, the use of precious metal based brazes and the incorporation of the interlayer add to the complexity and cost of the joining process. We have overcome these problems with the use of metallic-glass brazes. This is a new idea that eliminates the need for separate interlayers in ceramic-metal joining. We present the results of our preliminary joining experiments using metallic-glasses. Stainless steel 316L and molybdenum disilicide were successfully brazed using a cobalt based metallic glass. Issues pertaining to the interfacial chemistry, joint strength and residual stresses are presented here.

9:30 AM

Stress-Rupture Strength and Creep Behaviour on Molybdenum-Rhenium Alloys: *Bernd Fischer*[1]; Dietmar Freund[1]; Samantha Baxter[1]; Jan-C Carlen[2]; Todd A. Leonhardt[2]; [1]Fachhochschule Jena, Dept. of Matls. Tech., Univ. of Appl. Sci., Jena D-07745 Germany; [2]Rhenium Alloys Inc., P.O. Box 245, Elyria, OH 44036-0245 USA

Due to the outstanding properties of molybdenum and rhenium, which have very high melting points and excellent strength at elevated temperatures, molybdenum-rhenium alloys are valuable materials in applications where high strength at high temperature is required. The manufacturing processes of Mo-Re alloys can be tailored so as to enhance the mechanical properties of the part. The design engineer and manufacturer need exact information on the creep behavior, and stress-rupture strength at elevated temperatures. After an extensive literature search of the technical literature, relatively few reports were found on the mechanical properties of molybdenum-rhenium alloys, so an investigation was set forth using a special test facility for measuring high melting materials at temperatures up to 3273K under a protective atmosphere. The stress-rupture diagrams were determined for the molybdenum-rhenium alloys with rhenium contents between 41 and 51 weight percent at test temperatures of 1473K, 1873K and 2273K at 0.1 to 10 hours creep rupture times.

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Effect of the Carbide Solution Heat Treatment of ASTM-75 Alloys on the Wearing Behavior of the UHMWPE Material: *Edgar Guerra Martinez*[1]; H. M. Mancha[1]; A.J. U. Perez[2]; H. Lopez[3]; [1]Centro de Investigacion y Estudios Avanzados del IPN, Tribology, Carr. Saltillo-Monterrey Km 13, P.O. Box 663, Saltillo, Coahuila 25000 Mexico; [2]Universidad Autonoma de Nuevo Leon, Monterrey, NL; [3]University of Wisconsin, Milwaukee, WI USA

Today, one of the most important problems that face metallic hip and knee prostheses researching is the wear of polymeric and metallic materials used manufacture orthopedic implants to rehabilitate patients with no longer functional joints. The aim of this researching work was to study the effect of some surface modifications on an ASTM-F75 alloy on the wear rate of UHMWPE in order to decrease the amount of particles produced by sliding of the metal against the polymer. The desired amount of particles being that which can be removed via the patient lymphatic system. To study the wear properties, specimens of ASTM-F75 alloy were prepared by precision investment casting process, surface treated, and tested against UHMWPE at 37°C, applying two types of lubricant solutions (distilled water and Ringer solution).

10:10 AM Break

10:30 AM

Microstructure and Impact Toughness of Heat Affected Zones of an SA 508 Steel: *Sangho Kim*[1]; Sunghak Lee[1]; Soon Ju Kwon[1]; Joo Hag Kim[2]; Jun Hwa Hong[2]; Nack Joon Kim[1]; [1]Pohang University of Science and Technology, Matls. Sci. and Eng., San 31 Hyoja-dong, Nam-gu, Pohang, Kyoungbuk 790-784 Korea; [2]Korea Atomic Energy Research Institute, Nuclear Matls.Tech. Dev., Dajeon 305-600 Korea

In this study, microstructures of a heat affected zone (HAZ) of an SA 508 steel were identified by Mossbauer spectroscopy in conjunction with microscopic observations, and were correlated with impact toughness. Specimens with the peak temperature raised to 1350°C showed mostly martensite. With the peak temperature raised to 900°C, the martensite fraction was reduced, while bainite or martensite island were formed because of decrease in a prior austenite grain size and the slow cooling rate from the lower austenite region. As the martensite fraction increased, hardness and strengths tended to increase, whereas impact toughness decreased. However, impact toughness of the subcritical HAZ with the peak temperature raised to 650°C-700°C was seriously reduced after post-weld heat-treatment (PWHT) since carbide particles were of primary importance in initiating voids.

10:50 AM

Nanocomposite Magnets: *D. J. Branagan*[1]; [1]Bechtel BWXT Idaho LLC, Idaho Nat. Eng. and Environ. Lab., 2351 N. Blvd., Idaho Falls, ID 83415-2218 USA

The realization of high energy densities in permanent magnetic materials requires careful control of both the composition and the processing conditions in order to develop appropriate microstructures capable of storing high energy densities. It will be shown that nanoscale metal matrix composite microstructures can be developed in modified Nd-Fe-B alloys by careful selection, manipulation, and control of the alloying elements. The development of composite microstructures in hard magnetic materials is a novel approach resulting in many beneficial effects on the resulting structure/processing/property relationships

General Abstracts: Materials Processing and Fundamentals

Sponsored by: TMS

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

Monday AM — Room: Canal E
March 13, 2000 — Location: Opryland Convention Center

Session Chair: A. K. Vasudevan, Office of Naval Research, Code 332, Arlington, VA 22217-5660 USA

8:30 AM

Experimental Comparison of the Intrinsic Jump Frequency Formalism with the Traditional Darken-Manning Formalism for Describing Diffusion in Cu-Ni-Zn: *Robert T. DeHoff*[1]; Nagraj Kulkarni[1]; [1]University of Florida, Dept. of Matls. Sci. and Eng., Gainesville, FL 32611 USA

Earlier presentations derived the following expression for the intrinsic flux of component k in a multicomponent system: where Ck is the molar concentration of component k, ÿk is the composition dependent jump frequency of component k determined in a tracer diffusion experiment, ÿ is the jump distance and ÿk is a factor that reports the extent to which the atom jumps are biased. All of the information in this equation is determinable from independent experiments except ÿk. The traditional Darken-Manning approach to the description of multicomponent diffusion uses tracer diffusion coefficients, thermodynamic information and a contribution derived from the vacancy wind effect to compute intrinsic diffusion coefficients in the phenomenological description of the diffusion process. In this presentation these two disparate descriptions have been used to compute the ex-

perimental observables in a diffusion experiment (composition paths and profiles, Kirkendall shifts) for the system Cu-Ni-Zn. A simulation based upon these sets of equations permits separate computation of the experimental observables with each of the various influences assumed to operate in each formalism. This allows a definitive assessment of the effect that each of these factors has in predicting the experimental observables and a definitive comparison of the two approaches.

8:50 AM

Asymptotic Decay Analysis of Phase Coarsening Data: *Steven P. Marsh*[1]; [1]Naval Research Laboratory, Code 6325, 4555 Overlook Ave. SW, Washington, DC 20375-5343 USA

The progress of phase coarsening is generally quantified by the increase in the average particle size. However, coarsening is essentially a relaxation process driven by a reduction in the excess interfacial energy of a multiphase structure. Analysis of coarsening data via the decay of a global property, such as specific surface area, introduces a non-arbitrary temporal parameter that relates the experimental clock to an asymptotic time scale. This approach permits the fitting of data at earlier times, where the driving force is greatest, and more accurate determination of the power-law rate constant. Implications of this approach on measurement and interpretation of coarsening kinetics will be discussed.

9:10 AM +

Microstructural Evolution of Solid-Liquid Mixtures: *Victoria A. Snyder*[1]; Jens Alkemper[1]; Peter W. Voorhees[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

The coarsening of solid Sn particles in a Pb-Sn eutectic liquid was studied under microgravity conditions during the Microgravity Science Laboratory-1 mission. A clustered structure of particles exists at the beginning of the experiment for samples containing 10% and 20% volume fractions of coarsening phase. The coarsening of such a clustered structure of particles was studied via numerical simulations of coarsening using the approach developed by Akaiwa and Voorhees. Theoretically predicted particle size distributions and spatial correlation functions will be directly compared to the results from the spaceflight experiment. Furthermore, the microstructure in samples containing 50% and 70% coarsening phase (where a percolated structure of particles is present) was examined in orientation space using orientation imaging microscopy. Electron back-scattered diffraction patterns were used to determine the misorientation between contacting particles as well as the evolution of the misorientation distribution function with coarsening time.

9:30 AM

Elevated Temperature Mechanical Properties and Microstructure of a Ag-Cu-In-Ti Active Metal Braze Alloy: *John J. Stephens*[1]; Thomas E. Buchheit[1]; [1]Sandia National Laboratories, Matls. Joining Dept., P.O. Box 5800, MS0367, Albuquerque, NM 87185-0367 USA

Commercial Ag-Cu-In-Ti active metal braze alloys such as Incusil ABA (TM of WESGO Metals, Inc.) are attractive for metal-ceramic brazing applications because they eliminate the need for metallization of the ceramic and have relatively low braze process temperatures (~750°C). We have studied the elevated temperature mechanical properties of Incusil ABA using compression testing over the temperature range 150-550°C. For the case of constant load creep data, the minimum strain rate as a function of stress and temperature can be represented by the Garofalo sinh equation. Based on compressive stress-strain results, the 0.2% offset yield stress and work-hardening slope for this alloy are observed to have a maximum in the temperature range of 100-150°C. This effect is discussed in view of the microstructure of this alloy, which contains a significant volume fraction of Cu2InTi ordered intermetallic compound. Low force nanoindentation test results will be presented which quantify the relative hardness of this phase compared to the Cu-rich phase and the Ag-rich matrix of this braze alloy. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. Dept. of Energy under contract DE-AC04-94Al85000.

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Thin Film Coated Glass Soldering: *F. Michael Hosking*[1]; [1]Sandia National Laboratories, Dept. 1833, P.O. Box 5800, MS0367, Albuquerque, NM 87109-0367 USA

Conductive adhesives and solders are generally used to join soda-lime glass to itself and other materials. Soldering usually requires metallization of the glass. Two thin film glass coatings, Cr-Pd-Au and Cr-Ni-Sn, were investigated. Glass-to-glass test specimens, metallized with Cr-Pd-Au and soldered with 60Sn40Pb solder, had shear strengths approaching 16.5 MPa. Similar Cr-Ni-Sn coated specimens gave higher strengths, 20-22.5 MPa, with failures primarily in the glass. Surface roughness of the glass pieces also affected the coating uniformity and bond strength. Solder wetting and shear test results are discussed. The work was conducted at Sandia National Laboratories, a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

10:10 AM

Preparation of Silica Fibers from a Suspension: *Hiroyuki Nakamura*[1]; Akira Shimizu[2]; Yoko Suyama[2]; Eiichi Abe[1]; Noriyuki Yamada[1]; [1]Kyushu National Industrial Research Institute, Dept. of Inorganic Matls., 807-1, Shuku, Tosu, Saga 841-0052 Japan; [2]Shimane University, Dept. Matl. Sci., 1060, Nishikawazu, Matue, Shimane 690-8504 Japan

Fibrous silica was prepared by drying silica particle suspension. The silica fibers were obtained only on walls, which were wetted well by the suspension. The fibers were made of roughly close-packed silica particles. Widths of the fibers were affected by some preparation conditions. In this study, wider fibers were obtained under these conditions: higher silica particles concentration, larger particle diameter, and lower drying temperature. From an observation of a generation of fibers, the formation mechanism was considered as following; When a dispersion of silica was dried, a silica film made of silica particles were generated on a wall. During drying, since the shrinkage ratio of the film depends on the distance from the surface of the suspension, stress whose direction is parallel to the suspension surface arises. Because of the horizontal stress, the cracks are considered to proceed to the downward to generate silica fibers.

10:30 AM Break

10:50 AM

Formation of Cu-Ni-Zr Amorphous Powders with Significant Supercooled Liquid Region by Mechanical Alloying Technique: *Pee Yew Lee*[1]; Chung Kwei Lin[2]; G. S. Chen[2]; [1]National Taiwan Ocean University, Instit. of Matls. Eng., 2 Pei-Ning Rd., Keelung 202 Taiwan; [2]Feng-Chia University, Matls. Sci., Taichung 400 Taiwan

Recently, the studies on amorphous alloys were focused on the formation of bulk amorphous with a distinct supercooled liquid regions by using the traditional melt quenching technique, even water quenching are quite active. However, the composition of the material for the production of bulk amorphous alloys by quenching method is limited by the necessary low cooling rate. Changes in composition, therefore, strongly affect the glass forming ability and hence the required cooling rates to obtain bulk amorphous samples. In comparison to quenching techniques, amorphization by mechanical alloying process offers an alternative way which might be a promising method for the formation of a bulk amorphous with an obvious supercooled liquid region through the powder metallurgy route. In this study, we have investigated the possibility of preparing amorphous Cu-Ni-Zr powders by mechanical alloying. The results indicated that several amorphous alloy samples were found to exhibit a wide supercooled liquid region before crystallization. This is believed to be the first evidence for the appearance of a supercooled liquid region for mechanically alloyed Cu-Ni-Zr amorphous powders. The origin of the significant supercooled liquid region of these new alloys is discussed.

11:10 AM

A Thermodynamic Model for Mechanical Alloying: *Saheb Nouari*[1]; Abdul Razak Daud[1]; Shahidan Radiman[2]; Redzuwan Yahaya[2]; [1]University Kebangsaan Malaysia, Dept. of Matls. Sci., Faculty of Physical and Appl. Sci., Bangi, Selangor 43600 Malaysia; [2]University

Kebangsaan Malaysia, Dept. of Nuclear Sci., Faculty of Physical and Appl. Sci., Bangi, Selangor 43600 Malaysia

Mechanical alloying is a useful technique to produce different materials. Despite it's importance a few attempts has been made to model the fundamental process on one hand and to explain thermodynamically the mechanism of mechanical alloying on the other hand. In the present study, an attempt to develop a thermodynamic model for solid solution formation by mechanical alloying is presented. The main objective of the work is to describe from a purely thermodynamic point of view the evolution of solution during mechanical alloying based on the regular solid solution model, taking in to account the energy rise during alloying due to the generation of different defects.

11:30 AM

A Study of Casting Filling Process Using Simulation Tools: *Pongsakd Dulyapraphant*[1]; Patcharin Poosanaas[1]; Supparit Lounkosonchai[1]; Harit Sutabutr[1]; Panya Srichandr[1]; [1]National Metal and Materials Technology Center, Manufacturing and Design Tech. Ctr., 9th Floor, Gypsum Metropolitan Bldg., 539/2 Sri-Ayudhya Rd., Bangkok 10400 Thailand

The application of computer simulation are being widely used and becoming extremely beneficial in improving casting process and in tooling design. The use of simulation provides an insight into how each process related parameters affecting the quality of the casting. In this study, the influence of filling process on the quality of casting will be investigated by using a casting simulation package. Different types of gating systems will be simulated in order to identify how each component of the gating system contribute to the filling process. The comparison between simulation results and experimental results will be presented in order to verify the simulation results. Furthermore the use of simulation results to detect some flow related defects will be studied as well.

11:50 AM

The Story of the Safety Related Problems in the Titanium Industry in the Last Millennium: *Eldon R. Poulsen*[1]; [1]TI + MG Consultant, 4360 Malaga Dr., Las Vegas, NV 89121 USA

The titanium industry dates back to the turn of the century. The actual commercial production of the metal actually started in about 1950. By the end of the century, the industry was producing at the rate of over 100-million pounds per year. From a safety standpoint, the best judge of the industry is to evaluate each of the fatalities that have been experienced during the last 50 years. These as well as other major safety related problems such as fires will be discussed. Early on the problems was lack of knowledge with regards to furnace design and related explosions. The knowledge at the time was based on steel technology. The addition of the problem of hydrogen explosions was a totally new problem. When molten titanium reacts with water, the water breaks down and liberates the hydrogen which results in major explosions. During the first 5 years of the industry, furnace explosions killed a total of 6 employees. The next problem that plagued the industry was that of fires and explosions from sponge and fines fires. Fines fires and explosions killed a total of 3 employees. The third problem was confined space entry. Fatalities resulted from argon, nitrogen, and other inert gases. A total of 5 fatalities have been recorded to date from these causes. This paper discusses each of the fatal incidents and explains the cause and effect. Safety committees were formed and safe operating equipment and procedures were developed. Three separate lists of safety recommendations are included, var furnace design and operation, handling and storage of titanium fines and sponge and confined space entry based on OSHA standards.

General Non-Ferrous Pyrometallurgy: Industrial Operations and Recycling

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee

Program Organizers: Robert L. Stephens, Cominco Research, Trail, British Columbia V1R 4S4 Canada; Pekka Taskinen, Outokumpu Research Oy, Pori FIN-28101 Finland

Monday AM — Room: Bayou B
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Robert L. Stephens, Cominco Research, Trail, British Columbia V1R 4S4 Canada

8:30 AM

The Use of Ausmelt Technology at the Minsur Tin Smelter and Refinery: *Colette M. Ng*[1]; Ken R. Robilliard[2]; [1]Ausmelt Limited, 12 Kitchen Rd., Dandenong, Victoria 3715 Australia; [2]Funsur S.A., KM 240 Panamericana Sur, Pisco-Ica, Peru

The Peru-based tin mining company, Minsur S.A., commissioned Ausmelt Limited to design, supervise construction, and commission a tin smelter and refinery for production of refined tin metal. The plant is located 240 km south of Lima, near Pisco, Peru, and is operated by Funsur S.A., a wholly-owned subsidiary of Minsur. Selection of Ausmelt Technology followed a series of crucible scale test work and pilot plant trials conducted at Ausmelt's facility in Dandenong, Australia and two feasibility studies undertaken by Ausmelt in 1991 and 1993. Ausmelt began the plant design in 1994 with the construction phase following in 1995. Commissioning of the plant commenced early in 1996 with a Phase 1 target throughput of 30,000 tonnes per annum of concentrates. In mid-1998, oxygen enrichment of the Ausmelt lance air was introduced to expand the capacity to the Phase 2 throughput of 40,000 tonnes per annum of concentrates. The process route involves the use of an Ausmelt furnace for primary concentrate smelting, followed by a conventional tin pyro-refining circuit to produce high-grade tin suitable for sale. Further work continues between Ausmelt and Funsur to investigate the use of an alternative, more economical fuel source and the installation of a second Ausmelt furnace. This paper reviews the project to date with details of ongoing operations and developments at the Minsur Tin Smelter.

8:55 AM

Treatment of Industrial Waste in Reverberatory Furnace at Onahama Smelter: *Takayuki Sato*[1]; Nobuo Kikumoto[1]; Kiyotaka Abe[1]; Michio Nishiwaki[1]; [1]Onahama Smelting & Refining Company Limited, Smelting, 1-1 Nagisa Onahama, Iwaki, Fukushima 971-8101 Japan

Recently, the treatment of industrial waste materials has been a serious social problem here in Japan. Landfilling is now strictly prohibited due to a limited land area and incineration method is also regulated so as to not generate dioxins and furans (DXN). With this background, Onahama Smelter has begun to treat automobile shredder dust in reverberatory furnaces to recover various metals such as copper, gold, silver and palladium without generation of DXN. The total volume of these dusts is expected to be more than 1 million metric tonnes a year in Japan. Onahama Smelter is now planning to treat about 20% of them in the near future by using tonnage oxygen. In this paper, the heat balance and control method of DXN in reverberatory furnaces and treatment of these dusts are described.

9:20 AM

Precursors for PCDD/F Formation During Combustion of Electronic Scrap: *Menad Nourreddine*[1]; [1]Luleå University of Technology, Div. of Process Metallu., Luleå Se-97187 Sweden

The flame retardant contained in the electronic scrap can form dioxins (PCDDs) and furans (PCDFs). Their formation can be dependent on the quantities of carbon, hydrogen, chlorine, and oxygen present in a given system, as well as on parameters such as temperature

and pressure. Inadequate supply of combustion air (incomplete oxidation), low combustion temperatures (incomplete combustion), and insufficient turbulence in the combustion are factors which can favour their formation. Dioxins and furnans were also produced calatytically from chlorinated phenol precursors, non-chlorinated compounds that were chemically modified, and reaction of phenol with inorganic chloride. In this paper, different precursors such as combinations of C, H, O, Cl, rapid formation/combustion intermediates, and routes to PCDD/Fs are reviewed, and the influence of the amount of PVC on the formation of these isomers is discussed.

9:45 AM

Synergetic Effects During Phosphorous Production in Submerged-Arc Furnaces: *Markus A. Reuter*[1]; Diekske van der Pas[1]; Rob de Ruiter[2]; [1]Delft University of Technology, Faculty of Appl. Earth Sci., Mijnbiuwstraat 120, Delft, The Netherlands; [2]Thermphos, Postbus 65, Vlissingen, The Netherlands

In this paper, the kinetics of phosphorous production in submerged-arc furnaces will be discussed as a function of temperature, as well as various ore and reductant types. Interesting positive synergetic effects on the rate of reaction could be observed when mixing different types of ores. The results will be discussed with reference to a kinetic model, microprobe analyses, etc. The effects these results have on the control of the furnace will also be discussed in detail, indicating how these could be incorporated into the electrode and metallurgical control systems for submerged arc-furnaces.

10:10 AM Break

10:25 AM

Peirce-Smith Converter Hood Design Analysis Using Computational Fluid Dynamics Modeling: *Paykan Safe*[1]; [1]Gas Cleaing Technologies Inc., 4950 North O'Connor Rd., Ste. 250, Irving, TX 75062 USA

Computational fluid dynamics (CFD) modeling provides a powerful tool to assist with the design of ventilation and fume control systems in smelters and other high temperature metallurgical facilities. For this paper, this tool has been used to analyze the off-gas flow patterns exiting the mouth of a Peirce-Smith converter into a water-cooled hood and drop out box. The effects of various process and physical plant design parameters on process gas and fume capture and potential build up on the converter hoods was examined, and the optimum design and operating parameters were determined.

10:50 AM

Process Control Improvements at the Kennecott Utah Copper Smelter: *Robert M. Leary*[1]; Marielle A.S. Siraa[2]; [1]Kennecott Utah Copper Corporation, Smelter, P.O. Box 329, 12000 W. 2100 S., Magna, UT 84044 USA; [2]Rio Tinto plc, Tech. Svcs. Ltd., P.O. Box 50, Castlemead Lower Castle St., Bristol, England BS997YR UK

The Kennecott Utah Copper Smelter operates two Outokumpu flash furnaces: a smelting furnace for production of high-grade matte and a converting furnace for production of blister copper. Flash furnaces typically possess several characteristic features that must be considered in process control development such as long lag times associated with settler volume, non-linear input and output responses, large process gains, and unmeasured disturbances. Additionally, the Kennecott smelter possesses certain unique characteristics which must be considered such as a single mine concentrate source, small concentrate blending capacity, feed preparation facilities that introduce significant lags between furnace control parameters and manipulated set points and converter slag chemistry. In order to improve furnace control and on-line time, a process control system was developed which features feed forward and feed back control. The feed forward control module is based on a steady-state heat and mass balance that is executed in response to furnace feed changes. The feed back control module is based on Proportional-Integral control equations that were developed by statistical inference and are executed in response to furnace product grade and temperature deviations from set point. To provide regular feed back information to the control room operators, new sampling methods were developed which allow the operators to obtain molten metal samples at hourly intervals and allow for rapid laboratory turn around. The control system was implemented in May 1999 and a review of it's operation, adjustments, and future work will be discussed.

11:15 AM

Reversing the Philosopher's Stone: Recovering Iron from Copper Slags and Residues (Adeptus Ineptus): *Larry M. Southwick*[1]; [1]L. M. Southwick and Associates, 992 Marion Ave., Ste. 306, Cincinnati, OH 45229 USA

Interest has been expressed over the years in recovering iron from copper smelting and leaching residues. In the United States, this interest resulted in commercial operations and pilot plant tests by, among others, Lakeshore Copper on leaching residues, Phelps Dodge on copper matte, United Verde on granulated slag, and the USBM on molten slag. In most case, these were directed at producing sponge iron suitable for cementation of copper from spent leach solutions. Cementation of copper on iron from acid mine drainage solutions in the "Alchemists' Age" is likely what gave rise to mythical stories of a "Philosopher's Stone". While none of the above facilities continue in operation nor is cementation of great interest in current flowsheets, there does remain the need to treat acidic mine and tailing pond drainage. In a related field, there has also been increasing interest within the steel industry to recover iron from various steel plant iron oxide wastes to supplement a tightening supply of pig iron and scrap. Many steel waste plants propose to use technologies and processing concepts similar to those practiced earlier on the copper industry. This paper will review the processes and designs used to make sponge iron from copper residues, identify the factors that led to success or failure in these approaches, and then apply those results to evaluate potential operating difficulties with the technologies being proposed and installed for steel plant wastes. While there has been no visible effort to cross-fertilize technologies between the ferrous and non-ferrous industries in this field, this paper will identify where such information transfer may be desirable.

11:45 AM

The Case for the Copper Mini Smelter: *Rolf J. Wesley*[1]; [1]Kvaerner Metals, 12657 Alcosta Blvd., San Ramon, CA 94583 USA

In recent years the trend in Copper smelting has been to expand existing smelters to increase their throughput. New smelters that are being considered are very large. Despite this trend to larger smelters, there are still a number of conditions where a small smelter can be economically viable. The paper examines these conditions and outlines the process concepts for a small smelter. The paper outlines several options for treatment methods and presents a summary of the economic conditions that would lead to a successful small smelter project.

High-Temperature Superconductors: BSCCO Tapes & Applications

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

Program Organizers: U. Balu Balachandran, Argonne National Laboratory, Argonne, IL 60439 USA; Pradeep Haldar, Intermagnetics General Corporation, Latham, NY 12110-0461 USA; Chandra Pande, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5000 USA

Monday AM — Room: Canal D
March 13, 2000 — Location: Opryland Convention Center

Session Chair: U. (Balu) Balachandran, Argonne National Laboratory, Ceramics Section, Argonne, IL 60439 USA

8:30 AM Invited

High-Tc Conductor Development for Use in Electric Power Devices: *Heinz-Werner Neumueller*[1]; [1]Siemens AG, ZT EN4, P.O. Box 3220, Erlangen D-91050 Germany

Demonstration of HTS technology in power systems requires development of practical and robust conductors on technical scale. Meanwhile several manufacturers offer elementary conductors like 2223 tapes having reasonable engineering current densities in quantities sufficient for the manufacturing of representative power devices like transformers and current limiters for use in first pilot systems. Similar to the LTS conductor development the realization of larger high current HTS power devices need advanced cabled conductors designed for AC-application. Robel bars have been manufactured consisting up to 13 tapes of BPSCCO 2223 multifilamentary tapes. The production line delivers the conductor material for the low voltage winding of the 1 MVA railway model transformer currently being under construction and avoids complicated and expensive coil winding. In course of the resistive fault current limiter development a continuous production of switching elements has been established. Design considerations, quality measures and test results of the 1 MVA model will be presented.

9:10 AM Invited

Fatigue Behavior of Multifilamentary BSCCO Wire Used in Superconducting Ac Motors for Navy Ship Propulsion: *Donald U. Gubser*[1]; [1]Naval Research Laboratory, Matls. Sci. and Tech. Div., Code 6300, 4555 Overlook Ave. S.W., Washington, DC 20375 USA

A new type of ac superconducting motor is under development for the US Navy. This motor uses BSCCO conductor, wound into racetrack magnets, mounted on the rotor. Such magnets will be subjected to considerable cyclic stress, both during operation and during thermal cycling. It is well known that a monotonic strain of more than a few tenths of a percent can degrade the critical current in high temperature superconductor wires. A much lower strain, if applied repeatedly, also can degrade the critical current through cumulative damage effects. The possible fatigue damage of HTS wires is a significant concern for the reliability and service life issues in the development of motors and other power equipment. We report on measurements of the critical current fatigue in commercial BSCCO/Ag multi-filamentary wires. Long (250 mm) length sections of the wire were measured in liquid nitrogen using a conventional servo-hydraulic mechanical test system and with sinusoidal loading at a frequency of 10 Hz and zero load ratio. Strain ranges were from 0.01 to 1.0 percent. Critical current failure, using a stringent 0.1 microvolt-centimeter field criteria, was measured as a function of the number of load cycles at each strain level. The fatigue limit is determined from the strain-life curves. The relationship of the fatigue limit to the monotonic critical strain is discussed. In addition, systems advantages of superconducting motors for Navy ships will be discussed. Work is supported by the Office of Naval Research.

9:50 AM Break

10:00 AM

Grain Morphology of High Tc Superconducting Wires for Superconducting Motors: *C. S. Pande*[1]; K. L. Zeisler-Mashl[1]; R. A. Masumura[1]; [1]Naval Research Laboratory, Matls. Sci. Div., Washington, DC 20375 USA

Superconducting properties of practical high Tc superconductors are strongly influenced by their grain morphology. Towards this goal, texture measurements were conducted for BSCCO 2212 (Bismuth-Strontium-Calcium-Copper Oxide) tapes made by dip coating or by powder-in-tube techniques. BSCCO 2212 grain texture measurement was performed for both c-axis using (001) pole figures from (008) reflections and a-b axes using (115) pole figures. Current transport properties of these tapes were also measured and correlated with texture parameters obtained from contours of the texture plots. Grain morphology was inferred from the texture plots and was found to be consistent with a model based on global alignment of the c-axis and the presence of colonies of grains differing mostly in c-axis twist. There is a good correlation between the c-axis texture and Jc. Surprisingly on a global basis the system showed little a-b texture. However the material did give indications of local a-b texture in addition to the c-axis texture. These results far as the grain morphology is concerned can be explained in terms of colonies of grains. Inside a typical colony, the grains differ mostly in their small amount of twist along c-axis. These colonies are connected by complicated grain boundary structures.

10:40 AM

Histographic Analysis of the Microstructure of Ag/Bi-2223 Composite Conductors*: *Roxanne Baurceanu*[1]; Nazarali N. Merchant[1]; Albert K. Fischer[1]; Victor A. Maroni[1]; Ronald D. Parrella[2]; [1]Argonne National Laboratory, Chem.Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]American Superconductor, Two Technology Dr., Westborough, MA 01581 USA

A group of procedures has been developed to identify, quantify (in terms of area fraction), and spatially map the nonsuperconducting second phases (NSPs) that are formed during heat treatment of Ag/Bi-2223 multifilament composite conductors. These procedures involve (1) extracting the gray scale histogram from scanning electron microscope images of transverse cross sections of Ag/Bi-2223 after varying degrees of thermal treatment, (2) correlating selected ranges of gray scale values with the energy dispersive x-ray scans of those regions (to obtain metallic element ratios), (3) performing Raman microscopy analyses of the same regions (to identify the phases present), and (4) making systematic measurements of the Bi-2223 grain colony size and texture factor as they relate to NSP size, content, and composition. From these results we have developed quantitative correlations between key heat treatment parameters (such as, temperature, oxygen partial pressure, and treatment time) and the chemical form of the NSPs, their size distributions, their area fractions, and their impact on the final microstructure of the filaments. We find that oxygen partial pressures and temperatures on the high end of the Bi-2223 stability range tend to encourage the formation of the 14/24 alkaline earth cuprate, whereas oxygen partial pressures and temperatures on the low end of that range tend to encourage the 2/1 alkaline earth cuprate. The quality of the Bi-2223 grain colony microstructure is most sensitive to heat treatment temperature and the presence of large NSPs. The optimum temperature for achieving a robust grain colony microstructure tends to increase with increasing oxygen partial pressure. *Work sponsored by the U.S. Department of Energy, Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, under Contact W-31-109-ENG-38.

11:20 AM Invited

Development of Bi-2212/Ag Conductors and Coils: *Hiroaki Kumakura*[1]; Hitoshi Kitaguchi[1]; Hanping Miao[1]; Kazumasa Togano[1]; Tsutomu Koizumi[2]; Nozomi Ohtani[2]; Takayo Hasegawa[2]; Katsumi Ohata[3]; Junichi Sato[3]; Kazuhide Tanaka[4]; Michiya Okada[4]; [1]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]Showa Electric Wire & Cable, 2-1-1 Odasakae, Kawasaki, Kanagawa 210-8660 Japan; [3]Hitachi Cable Limited, 3550 Kidamari, Tsuchiura, Ibaraki 300-0026 Japan; [4]Hitachi Limited, 7-1-1 Ohmika, Hitachi, Ibaraki 319-1292 Japan

Development of two types of Bi-2212/Ag superconductors with excellent current carrying capacities is now in progress. One is a surface-coated multilayer tape conductor prepared by applying Pre-Annealing and Intermediate Rolling (PAIR process) before a heat treatment. The other is Bi-2212 multifilamentary wires fabricated by applying Rotation Symmetric Arranged Tape-in-tube (ROSAT process, where Bi-2212 tapes were arranged with triple rotation symmetry. We evaluated current carrying characteristics of these Bi-2212 conductors at various temperatures and fields. Excellent Ic and Jc values of about 380A and 2?~105A/cm2 were obtained in a field of 30T at 4.2K for short tapes cut from the PAIR processed 100m tape. E(electric field)-J(current density) characteristics was also improved by the PAIR process. Jc values of pancake coils prepared with 100m-class PAIR processed tapes were much higher than those of the no-PAIR processed tape. The ROSAT wires show very small Jc anisotropy with respect to the field orientation in spite of excellent Jc values. A 990-filament wire showed Ic and Jc of 360A and 105A/cm2 in 28T at 4.2K. Recently, we fabricated 400m-length ROSAT wires, and constructed a solenoid magnet using these wires. Test result of the magnet will be reported.

International Symposium on Iridium: Mechanical Properties

Sponsored by: Structural Materials Division, Refractory Metals Committee

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

Monday AM — Room: Jackson A/B
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Evan K. Ohriner, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6083 USA; William J. Barnett, U.S. Department of Energy, Office of Space and Defense Power Systems, Germantown, MD 20874-1290 USA

8:30 AM Invited

Micro- and Macro-Alloying of Ir-Base Alloys: E. P. George[1]; *C. T. Liu*[1]; [1]Oak Ridge National Laboratory, Mets. and Cer. Div., P.O. Box 2008, Oak Ridge, TN 37830-6115 USA

Ir-base alloys are of interest for structural applications at high temperatures because of their high melting point (2443°C) and good oxidation and corrosion resistance. A major concern for structural use is their low tensile ductilities when tested at conventional strain rates at ambient temperatures and at high strain rates at elevated temperatures. Microalloying has been used to strengthen grain boundaries and suppress brittle intergranular fracture in Ir alloys. Auger studies indicate that Th has a strong tendency to segregate to Ir grain boundaries and suppress intergranular fracture at elevated temperatures. Among macroalloying elements, Hf is found to be the most effective in improving the strength of Ir alloys at room and elevated temperatures. Ir-W alloys doped with Th have been successfully developed as cladding materials for space power applications at temperatures to 1400°C. The current studies of Ir-base γ-γ alloys and intermetallic alloys will be also briefly reviewed.

9:00 AM

Comparing Experimental Measurements with Ab Initio Simulations of the Elastic and Plastic Behavior of Single Crystalline Iridium: *T. J. Balk*[1]; O. N. Mryasov[2]; Y. N. Gornostyrev[3]; P. Panfilov[4]; A. J. Freeman[2]; K. J. Hemker[1]; [1]Johns Hopkins University, Dept. of Mech. Eng., 200 Latrobe Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Northwestern University, Dept. of Physics and Astronomy, Rm. F275, 2145 No. Sheridan Rd., Evanston, IL 60208-3112 USA; [3]Instit. of Metal Physics, 18 S. Kovalevskaya St., Ekaterinburg 620219 Russia; [4]Urals State University, Instit. of Physics and Appl. Math., Ekaterinburg 620083 Russia

Iridium is one of two face-centered cubic (FCC) metals that is known to undergo brittle fracture, in stark contrast with the normal ductile failure of other FCC metals. The occurrence of brittle fracture, which follows significant plastic deformation of up to 70%, is thought to result from the energetics of the dislocation core. Ab initio first-principles methods have been used to predict fundamental characteristics that govern the mechanical behavior of iridium, e.g., elastic constants, dislocation structure and fracture parameters such as surface and unstable stacking energies. The elastic behavior and strength of single crystalline iridium have been measured experimentally. These measurements and transmission electron microscopy (TEM) observations of the dislocation core structure will be compared with the first-principles predictions, in order to better understand the fundamental mechanical behavior of iridium.

9:20 AM

Influence of Trace Impurities on the High-Temperature Mechanical Properties of Iridium: B. Fischer[1]; *Andreas Behrends*[1]; D. Lupton[2]; J. Merker[2]; [1]Fachhochschule Jena, Univ. of Appl. Sci., Tatzendpromenade 1b, Jena 07745 Germany; [2]W. C. Heraeus GmbH & Company KG, Matls. Tech. Div., Heraeusstrasse 12-14, Hanau 63450 Germany

Iridium is used as a high temperature material at temperatures up to 2000°C, for example, for high-strength components in space technology. Trace impurities can favour the formation of intercrystalline cracks (processing problems) and reduce the necessary strength and ductility of iridium for this extreme field of application. Because no detailed data on the influence of trace impurities have been published, examinations were first carried out on the doping effect of the elements iron and silicon on high-purity iridium in different concentrations (27-1300 ppm). By means of a suitable production process, it is possible to avoid grain boundary segregations of trace elements which are difficult to remove under industrial conditions. The metal is now easier to process, the tendency to intercrystalline cracking is reduced and a larger amount of trace impurities is tolerable than was previously assumed. Only a small effect of the added elements on the stress-rupture strength of iridium at 2200°C in the short time range (<10 hours) could be observed. However, distinct differences in the creep behaviour of the doped heats were found relative to pure iridium.

9:40 AM

Brittle Transcrystalline Fracture in Plastic Face Centered Cubic Metal Iridium: *P. Panfilov*[1]; [1]Urals State University, Lab. of Strength, Ekaterinburg 62001 Russia

The main puzzle of iridium is an inclination to cleavage after severe plastic deformation. Evolution of transcrystalline cracks in bulk crystals and thin foils of iridium is the subject for discussion in this paper. Tested crystals did not content dangerous non-metallic impurities and level of metallic contaminants was minimal what allows considering brittleness as inherent property of iridium. Bulk crystals failed after considerable elongation, however, necking did not appear in them. It was shown that octahedral slip gives the main contribution to plastic deformation, therefore, mechanical twinning or non-octahedral slip could not be a cause for cleavage. Sharp cracks appeared in strengthened material near power concentrators of stresses. Sometimes, plastic deformation accompanied crack growth, but this did not lead to crack tip blunting. Motion of cracks on primary ({100}) and secondary ({210}) cleavage planes were observed. Evolution of cracks in thin foils was considered in detail. It is unbelievable, but cracking of iridium foils looks like fracture of ductile metal: microcracks emit both twins and perfect dislocation, dislocation emission causes crack tip blunting, and dangerous cracks possess zigzag profiles. In contrast with usual f.c.c.-metal, iridium foils contain high dense <110> dislocation nets, which bother emitted dislocation to move from crack tip, and, as a result, crack leaves ability to emit dislocations. After that crack should either stop or continue its growth without dislocation emission like crack in brittle crystal. Microcrack will have an opportunity to transform in zigzag crack if dislocation nets are absent around it. Observations of cracks have shown that brittle transcrystalline fracture is property of bulk iridium crystals only. TEM study allows revealing microscopic cause of brittleness in iridium, while its thin foils failed by ductile manner. This is its ability to store high dense dislocation nets. During preliminary deformation, plastic iridium crystal exhausts the resource of plasticity (nets cover all volume of crystal) and further loading should induce brittle fracture of sample.

10:00 AM Break

10:10 AM

Strength Behavior of Ir-Based Refractory Superalloys: *Y. Yamabe-Mitarai*[1]; *Y. Gu*[1]; Y. Ro[1]; S. Nakazawa[1]; T. Maruko[2]; H. Harada[1]; [1]National Institute for Metals, HTM21Project, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]Furuya Metal Company Limited, No.1915, Morizoeshima, Shimodate, Ibaraki 308-0861 Japan

We have proposed "refractory superalloys" using platinum group metals, especially Ir, as ultra-high temperature materials. The refractory superalloys are defined as the alloys with an fcc and L12 two-phase coherent structure and with yet higher melting temperature than

Ni-based superalloys that are used as high temperature materials like turbine blade. High strength of Ni-based superalloys at high temperature is attributed to the fcc and L12 two-phase coherent structure. Compression strength, creep properties, and microstructure up to 1800°C were investigated in the Ir-V, Ir-Ti, Ir-Nb, Ir-Ta, Ir-Hf, and Ir-Zr binary systems. The fcc and L12 two-phase coherent structure formed in these alloys after heat treatment. The strengths of the two-phase alloys are higher than those of fcc or L12 single-phase alloys. This shows that the main factor contributing to strengthening is precipitation hardening. Precipitate shape depends on lattice misfit between the matrix and the precipitates. Plate-like and cuboidal precipitates formed in the alloys with large (2%) and small (0.3%) lattice misfit, respectively. Precipitation hardening is more effective for plate-like precipitates because shearing or bypass of dislocation is difficult for plate-like precipitates and coherency strain at the interface between matrix and precipitates is high by large lattice misfit. Deformation mechanism is discussed by observation of dislocation structures of deformed samples.

10:30 AM

Effect of Ir Addition on High-Temperature Strength of NiAl Single Crystals: *A. Chiba*[1]; T. Ono[1]; X. G. Li[1]; S. Hanada[2]; T. Sugawara[2]; [1]Iwate University, Dept. of Matls. Sci. and Tech., Morioka 020-8551 Japan; [2]Tohoku University, Instit. for Matls. Rsch., Sendai 980-77 Japan

In order to obtain the knowledge on mechanisms of enhancement of high-temperature strength of NiAl by Ir addition, compression and creep tests of soft [-223] oriented single crystalline NiAl and Ir-doped NiAl (Ni0.9Ir0.01Al) have been conducted in the temperature range from room temperature to 1473 K. CRSSs of Ni0.9Ir0.1Al single crystal are about a factor of 3, 4, and 6 higher than those of binary NiAl single crystal at room temperature, 1073 K and 1473 K, respectively. The Ni0.9Ir0.1Al single crystal deforms by kinking instead of slip of b=(-110)[001] dislocation which normally glide in binary NiAl single crystal with soft [-223] crystal axis. Secondary creep rate of Ni0.9Ir0.1Al single crystal is about a magnitude of 4 lower than that of binary NiAl single crystal at 1273 K; stress exponent of secondary creep for Ni0.9Ir0.1Al and NiAl is approximately 3.0 and 4.8, respectively. Dislocation substructures of crept Ni0.9Ir0.1Al single crystal consist of subboundaries and gliding dislocations, indicating that dislocations of Ni0.9Ir0.1Al single crystal are more difficult to move than those of binary NiAl single crystal. It is likely that addition of Ir to NiAl enhances the Peierls stress of (-110)[001] slip system of NiAl and leads to the activation of non-b=(-110)[001] dislocation and (or) kinking.

10:50 AM

Microstructure and Mechanical Properties of Quaternary Ir-Nb-Ni-Al Alloys: *X. Yu*[1]; Y. Yamabe-Mitarai[1]; T. Yokokawa[1]; M. Osawa[1]; Y. Ro[1]; H. Harada[1]; [1]National Research Institute for Metals, High Temp. Matls. 21 Project, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

Many attempts have been made in order to improve the temperature capability of Ni-based superalloys by adding more refractory elements into them, such as Re, W, Mo, Ta, et al. Single crystal Ni-based superalloys are classified by the level of Re content. But the biggest problem is phase stability. Since Re has a low diffusivity and tends to be segregated in matrix, the addition of Re assists in the formation of a topologically close-packed (TCP) phase. Another question is the melting temperature of Ni (1450°C). It can not be used at higher temperature. Developing new alloys whose matrix elements are of higher melting temperature is another way. Ir attracts researcher's attention due to the higher melting temperature (2240°C) and superior oxidation and the fcc/L12 coherent structure in Ir-based alloys. The fcc/L12 coherent structure provides a challenge for combining Ir- and Ni-based alloys to prepare new alloys. Therefore, we do the efforts to investigate quaternary Ir-Nb-Ni-Al in the last two years. The previous results indicated the two kinds of fcc/L12 (fcc/Ir_3Nb and fcc/Ni_3Al) coherent structure formed in some alloys and strength also increased drastically compared to Ni-based superalloys. These alloys are promising to be used at higher temperature. However, the microstructure evolution of quaternary Ir-Nb-Ni-Al alloys is not clear, element distribution and lattice misfit of two kinds of fcc/L12 in the same quaternary Ir-Nb-Ni-Al alloys have never been studied. Therefore, in the present paper, microstructure evolution, element distribution and lattice misfit of two kinds of fcc/L12 as well as 0.2% flow stress at room temperature and 1200°C in quaternary Ir-Nb-Ni-Al will be investigated.

11:10 AM

The Effects of Nb Content and Third Element Additions on the Fracture Behaviours of Polycrystalline Ir-Nb Two-Phase Alloys: *Y. Gu*[1]; *Y. Yamabe-Mitarai*[1]; *H. Harada*[1]; [1]National Research Institute for Metals, HTM 21 Project, 1-2-1 Sengen, Tsukuba, Ibarake 305-0047 Japan

Iridium (Ir) and its single-phase alloys have been tried as structural materials for ultra-high temperature applications, such as solar thrusters in lightweight solar concentrators for use in space and as postimpact containment claddings for radioactive fuel. Ir and its alloys were chosen for these applications because of their high melting point, excellent oxidation resistance, and welding suitability. However, polycrystalline Ir and its alloys normally exhibit grain-boundary (GB) fracture with limited ductility in tensile tests at temperatures below 800°C. Moreover, alloys with single-phase normally show less resistant to creep deformation, a property that is important for structure materials at high temperatures. Recently, we developed a new class of two-phase superalloys, namely, refractory superalloy, based on platinum group metals (PGMs). These refractory superalloys have a coherent fcc-L12 structure, similar to γ/γ' microstructure in nickel-base superalloys, and have good potentiality as structural materials used at ultra-high temperatures up to 2000°C. Preliminary results showed that, of these refractory superalloys, Ir-based fcc-L12 two-phase alloys, such as Ir-15 at % Nb, Ir-15 at % Hf, and Ir-15 at % Zr alloys, were superior in high temperature strength and oxidation resistance. Despite the importance of the relationship between the microstructures and deformation behaviours of these refractory superalloys for practical applications, the fracture behaviour was also needed to be understood. The aims of the present study are: (i) to examine the effects of various niobium (Nb) contents on the microstructures and fracture behaviours of Ir-Nb two-phase refractory superalloys, (ii) to identify third element (Ni, Mo, C, and B) addition on the microstructures and fracture behaviours of Ir-15 at % Nb alloy, and (iii) to determine the relationship between the microstructure and fracture behaviour of the alloy. Our results showed that the intergranular fracture occurred in Ir single-phase alloys could be governed by controlling the microstructures around grain-boundary (GB) and alloyed with some third elements.

11:30 AM

Mechanical Behavior of Ir-Sn Layered Crystals: *P. Panfilov*[1]; Y. L. Gagarin[1]; A. V. Yermakov[2]; [1]Urals State University, Lab. of Strength, Ekaterinburg 62001 Russia; [2]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia

Iridium based compounds are prospective materials for exploitation in aggressive environments, but poor plasticity and brittleness make considerable doubts for their industrial applications. Elaboration of layered iridium compounds would help to solve the problem of technological brittleness, in as much as layered structure of sample could suppress crack growth and provide plastic deformation even in brittle crystals. Single crystals of Ir-Sn compound were grown by means of high temperature synthesis in vacuum. Silver lustrous and metallic electric conductivity was inherent to crystals. Chemical analysis has shown that material contains Ir and Sn in proportion of 1:1. Samples possessed tetragonal lattice with ratio of c/a<<1 and their morphology was similar to "sandwich" formed from one hundred thin square plates (thickness of each plate is 10^{-3}mm). These features did not allow to built simple crystallographic model for the compound. Samples were indented by Vickers diamond pyramid at room temperature. Deep holes remained on the surface after indentation, but this did not lead to the separation of crystal. Sliding of plates or single crystal layers is the main mechanism, which provides severe plastic deformation in vicinity of indents. Twin lamellas in <110> direction and cracks along <100> and <110> were observed in deformed single crystal layer. Cracks have only appeared near indents, while twins were detected on the whole crystal. It was revealed that cracks in the first layer could not pass in depth of crystal.

Kleppa Symposium on High Temperature Thermochemistry of Materials: Session I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Extraction & Processing Division, Thermodynamics & Phase Equilibria Committee, Process Fundamentals Committee

Program Organizers: Ray Y. Lin, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA; Y. Austin Chang, University of Wisconsin, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Dr. Susan Meschel, The University of Chicago, Chicago, IL 60637 USA; Ramana Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487 USA

Monday AM — Room: Lincoln E
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Susan V. Meschel, University of Chicago, Chicago, IL 60637 USA; J. C. Gachon, Universite Henri Poincare, Lab de Chimie du Solide Mineral, Vandoeuvre, Cedex 54506 France

8:30 AM

Some Like it Hot: Evolution and Applications of High-Temperature Reaction Calorimetry at University of Chicago from 1952 to 2000: *Ole J. Kleppa*[1]; [1]University of Chicago, James Franck Instit., Chicago, IL 60637 USA

The author will present a review of his work in the field of high-temperature reaction calorimetry. This review will be partly historical, partly scientific. He will touch on his work on low-melting liquid alloys, on the thermochemistry of some Hume-Rothery type binary alloys, on the heats of mixing of molten salts, on the development of oxide melt solution calorimetry, on the thermodynamics and thermochemistry of metal-hydrogen systems, and on the development and construction of a number of different reaction calorimeters suitable for work at temperatures up to about 1200°C. Finally he will give an introduction to his more recent investigations of refractory intermetallic and related compounds. This section will include an outline of the special technique of solute-solvent drop calorimetry, a technique which is particularly suitable for very refractory compounds. However, most recently his studies of refractory compounds have emphasized the thermochemistry of congruent melting compounds with melting points below about 2000°C using direct synthesis calorimetry at 1200°C. More detailed discussions of these investigations will be presented in the invited papers given by his co-workers S. V. Meschel and Qiti Guo.

9:15 AM

Contributions to Molten Salt Chemistry by Ole J. Kleppa: *Milton Blander*[1]; [1]Quest Research, 1004 E. 167th pl., South Holland, IL 60473-3114 USA

Ole Kleppa and associates have accurately measured the enthalpies of mixing of a large number of binary molten salt systems. These results have also been proven to be useful in the analyses of phase diagrams and in creating a reliable database on free energies of mixing of a large number of systems. He made a general confirmation of a specific result by Hildbrand and Salstrom that excess free energies of mixing of simple binary molten salts could be represented by a simple polynomial, an important result for ionic systems with very long range ionic pair interactions. His measurements of the enthalpies of mixing of all binary alkali nitrates led to an expression which was close to that deduced form a simple model by Forland. This result also catalyzed the Conformal Ionic Solution Theory, the only theory fundamentally valid for molten salts. This theory later led to methods for accurately predicting the solution properties of multicomponent molten salt systems from data on the subsidiary binaries and the pure component salts. His measurements of binary systems with the two components having the same cation and two different anions indicated that deviations from ideality were generally very small. A theoretical extension of this conclusion to silicates and other polymeric slags led to reliable predictions of the solubilities (and sulfide capacities) of ionic compounds. Kleppa's work has had a major influence on molten salt and slag chemistry.

9:45 AM

Thermochemical Studies of Oxynitrides by Oxidative High Temperature Solution Calorimetry: *Alexandra Navrotsky*[1]; [1]University of California, Dept. of Chem. Eng. and Matls. Sci., Thermochem. Facility, Davis, CA 95616 USA

Thermodynamic properties of nitrides and oxynitrides are poorly known, despite technological importance and scientific interest. Recent advances in oxidative drop solution calorimetry of nitrogen-containing materials in molten oxide solvents at 973-1073 K enable the determination of enthalpies of formation. The calorimetric methodology is described and applied to three groups of materials: the beta sialons in the Si-Al-O-N system, cubic oxynitrides in the Zr-O-N and Zr-M-O-N(M = Y, Ca, Mg) systems, and phosphorus oxynitrides in the P-O-N and Li-P-O-N systems. The energetics of oxygen-nitrogen substitution these systems is discussed in terms of crystal chemistry, bond strengths, and short range order.

10:15 AM Break

10:30 AM

What Will Be Done in the Future with the Enthalpy Data Set of O. J. Kleppa: *Jean Hertz*[1]; [1]Universite Henri Poincare-Nancy I, Lab. de Thermodyn. Metallu., Umr Cnrs 7555, Chimie du Solide Mineral BP 239, Vandoeuvre, les Nancy-Cedex 54506 France

After 40 years or more of experimental work devoted to thermochemistry of metallic alloys it will be time to wonder: what will be done in the future, by the scientific community, with the patrimony of data I have obtained? This question is of particular importance for O. J. Kleppa with his fantastic panel of consistent enthalpy data, obtained by his group for more than 45 years of unbroken calorimetry experiments relative to more than 200 different binary systems. The progress of phase diagram calculation of multicomponent systems, is significant as far as industrial applications for new alloys development and metallurgical processes are now in due course. That will be the main topic of the near future metallic thermodynamics. That means that very simple models are generalised: the Redlich-Kister multicomponent model, coming from the petroleum industry, and the Hillert sublattice model for intermediate phases provide a very large domain of applications, useful for the main multicomponent metallic systems. Unfortunately such works cannot start out of nothing, but out of consistent thermodynamic data used to fit the binary systems first, then the ternaries. Higher order terms are generally not needed. The enthalpy of mixing of the liquid phase in the whole range of composition and the enthalpy of formation of compounds are of significant interest to obtain a set of coherent multicomponent data bank. In the 80[ies] we have trusted the progress of band theory and physical quantum mechanics to provide a lot of "ab initio" or semi-fitted energetic data set. Unfortunately the relative phase stability in a multicomponent system depends on only some hundredth of eV and the precision of the calculated cohesive energy will not attain 0.1 eV in the more accurate results. The interest of this part of the physical thermochemistry remains for understanding the metallic bonding, but not for practical applications. In my opinion and for a very long period the only source of information for realistic metallic multicomponent phase diagram previsions will be the good experimental binary and ternary data: enthalpy and chemical potential measurements, each one needed to calibrate the two enthalpy and entropy contributions of the Gibbs-function. Experimental equilibrium lines and tie-lines in phase diagrams could be considered as equivalent to a Gibbs-function information, when calorimetric data are available. For this reason the large Kleppa enthalpy data set patrimony will be of crucial interest for a

very long future period. We will illustrate this prognosis with various multicomponent diagram previsions in the field of low melting metals.

11:00 AM

Excess Thermodynamic Functions of Systems with N (N>6) Components: Measurements and Calculations: *M. Gambino*[1]; J. P. Bros[1]; Z. Moser[2]; M. Hoch[3]; [1]Universite de Provence, Iusti, Ura-Cnrs 6995, Rue Enrico Fermi 5, Marseille, Cedex 13 13453 France; [2]Instytut Podstaw Metallurgii im., Alexandra Krupkowskiego Pan, Krakow, ul Reymonta 25 30-059 Poland; [3]University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA

We measure the enthalpy of mixing along a line connecting the center of the system with one component, and the partial enthalpy at the center of the system for all components. The calorimeter is a high temperature Calvet type calorimeter, operating between 700 and 1000 K. For the calculations we use the Hoch-Arpshofen model, which uses only binary interactions. The agreement between calculated and measured data is better than ±5%. Thus only a relatively few measurements are needed to understand the complete system. We discuss here the 7 component system (Bi-Cd-Ga-In-Pb-Sn-Zn). Measurements and calculations of Zn activity in the system (Bi-Cd-Pb-Sn-Zn) show similar excellent agreements.

11:30 AM

Thermochemistry of Alloys of Transition Metals and Lanthanide Metals with Some IIIB and IVB Elements in the Periodic Table-An Overview: *S. V. Meschel*[1]; O. J. Kleppa[1]; [1]The University of Chicago, James Franck Instit., 5640 S. Ellis Ave., Chicago, IL 60637 USA

We have in this laboratory conducted systematic studies of the thermochemistry of transition metal and rare earth alloys by high temperature calorimetric methods. An overview of the thermochemistry of the alloys of the transition metals and lanthanide metals with elements in the IIIB and IVB columns of the periodic table will be presented. The enthalpies of formation of most of these compounds were determined by high temperature direct synthesis calorimetry. This review will summarize the trends between the enthalpies of formation of the TR-X and LA-X alloys (where X is a IIIB or IVB element) and the atomic numbers in each transition metal and lanthanide metal family. We will compare our measured enthalpies of formation of each alloy family for the 3d, 4d and 5d transition metal elements. We will also compare our experimental measurements with predicted values on the basis of Miedema's semi-empirical model. This review will show examples of a correlation between the enthalpies of formation of the alloys of the lanthanide elements with the non-metal elements in the IIIB and IVB columns in the periodic table. We will also show some comparisons of our measured enthalpies of formation with the predictions by Gschneidner for the lanthanide alloys.

12:00 PM

Abstract Text Is Unavailable: *F. Sommer*

Light Metals Division Plenary Session: Aluminum Plenary

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Ray D. Peterson, IMCO Recycling Inc., Rockwood, TN 37854 USA

Monday AM — Room: Presidential Ballroom
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Ray D. Peterson, IMCO Recycling Inc., Rockwood, TN 37854 USA

8:00 AM Introductory Remarks
Dr. Ray Peterson, IMCO Recycling Inc., Rockwood, TN 37854 USA

8:10 AM

An Overview of the Aluminum Industry: *Richard B. Evans*[1]; [1]President, Alcan Global Fabricatin Group, 6060 Parkland Blvd., Cleveland, OH USA

An overview and outlook of the global aluminum industry will be presented. A focused look on aluminum's two largest markets, packaging and transportation, will be given.

8:40 AM

Materials Used in Aluminium Smelting: *Harald A. Oye*[1]; [1]Norwegian University of Science and Technology, Instit. of Chem., Trondheim N-7491 Norway

The production of alumina, anode and cathode carbon will be reviewed from a historic and the present point of view. Problems, challenges and new future advances will be discussed.

9:10 AM

Advancing the Hall Heroult Electrolytic Process: *Barry Welch*[1]; [1]The University of Auckland, Dept. of Chem. and Matls. Eng., Private Bag, Auckland 92019 New Zealand

The design and operating advances achieved in the last quarter century will ensure the Hall Heroult technology will maintain a competitive advantage over alternative aluminium production processes for some time into the future. The advances were lead by magnetic compensation and computerised process control coupled with electrolyte optimization. These enable larger more economic cells to be designed. During the same time the fundamental studies have enabled a better understanding of the subtleties of the processes and secondary reactions that were ignored in the theoretical understanding whilst the cells performed poorly. These secondary processes, including reactions associated with impurities and anodes consumption, those leading to onset of anode effect, and alumina dissolution kinetic have not been fully exploited yet. Today as the focus shifts to higher productivity, the dynamics of the cell and the impact operations have on the very finely tuned heat balance becomes more important. With the high current efficiencies and low margins for error in modern cells the present challenge is to refine designs, control strategy and operating practices so that further marginal gains and economic performance can be achieved. This will include better management of aluminium fluoride, prevention of anode effects, and earlier detection of the increasingly prevalent anode spikes. Super structure design changes coupled with improved control and practice will also enhance productivity of future generations of cells.

9:40 AM Break

10:00 AM

A Perspective on Aluminum Melting and Metal Treatment: *C. Edward Eckert*[1]; [1]Apogee Technology Inc., 1600 Hulton Rd., P.O. Box 101, Verona, PA 15147-2314 USA

Essentially all commercially significant aluminum produced shares a common processing history; melting and metal treatment. These two operations therefore have a monumental impact on production costs, the ultimate quality of end-use products, and also have an ancillary influence on environmental issues. Melting and metal treatment processes understandably remain developmentally topical. The technological heritage of aluminum melting and metal treatment will be chronicled, and, importantly, significant technical milestones identified. These milestones are associated with specific product/commercial imperatives or a revolutionary development. An example of the former is the emergence of the aluminum beverage container in the 1960 timeframe, while the latter is exemplified by rotary impeller in-line treatment. Finally, an inventory will be provided of contemporary aluminum melting and metal treatment technology, critical needs assessed, and a projection offered of future development.

10:30 AM

Aluminum Solidification Processing-Prospective and Retrospective Views of the Industry and the Field: *Diran Apelian*[1]; [1]Worcester Polytechnic Institute, Aluminum Cast. Rsch. Lab., Met. Proc. Instit., Worcester, MA 01609 USA

During the last 50 years, aluminum has evolved into one of the most important societal materials; it is used in a variety of diverse

applications-construction, automotive, aerospace, packaging, furniture, jewelry and a vast number of products, which once were made from ferrous or other materials. Specifically, during the last decade, we have seen significant increases in the use of cast aluminum net-shaped manufactured components. For example, in 1980 there were 800,000 tons of aluminum casting shipments in North America versus 1,800,000 tons in 1998. In this plenary lecture, a prospective review of the science and technology of aluminum metalcasting will be presented, for both primary aluminum production as well as metalcasting. This will be followed with a retrospective presentation of the emerging technologies and challenges we face. The plenary lecture will be a holistic review of solidification processing of aluminum, addressing: where we have been, where we are going, and what are the exciting frontiers facing the industry.

11:00 AM

Aluminum Fabrication and Applications: *Elwin L. Rooy*[1]; [1]Elwin L. Rooy and Associates, 461 Ravine Dr., Aurora, OH 44202 USA

Modern manufacturing strategies combine solidification and thermomechanical process technologies for optimum efficiency, product quality and reliability, and product performance. The present and future importance of incorporating melt processing and solidification considerations into the sequence of down-stream operations for satisfying product requirements and for developing new competitive market capabilities forms an essential basis for these strategies. The majority of aluminum is consumed in fabricated forms ranging from rolled products to components formed by powder metallurgy. Remelted and cast products represent a rapidly growing manufacturing sector. The evolution of processes for aluminum's multivaried commercial shapes and forms paralleled the industry's success in determined applications and market penetration. The history of the aluminum industry's development of casting, forging, extrusion and rolled products and the significance of process and product developments to the dramatic evolution of aluminum as the metal of the twentieth century is reviewed, and current and projected developments are outlined.

Magnesium Technology 2000: Electrolytic Technology

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

Monday AM — Room: Bayou C
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Ramaswami Neelameggham, Magnesium Corporation of America, Salt Lake City, UT 84116 USA

8:30 AM Invited, Keynote

Magnesium Industry Growth in the 1990 Period: *Robert E. Brown*[1]; [1]Magnesium Monthly Review, 226 Deer Trace Rd., Prattville, AL 36067-3806 USA

The world magnesium industry has experienced a continuing growth of 6 per cent or more per year for over 10 years. Major growth has been in the die casting sector which has grown as much as 14% per year over the last 10 years. New magnesium projects are being announced frequently. In many countries there are large magnesium projects being discussed, studied, designed and constructed. Unfortunately, there is much more discussion than there is construction. A review of the total magnesium supply situation shows a great paradox. The prices of magnesium will have to be lowered to successfully market all of the magnesium production tonnage that is being discussed. Lowered prices makes the economics of a new magnesium plant much less favorable. One of the big problems is process technology. There are literally no processes commercially available that have demonstrated sufficient development to be able to produce magnesium metal economically. The closest technology is the Alcan cell, but it requires a very dry and pure anhydrous magnesium chloride feed. This is the basic area where every project past, present and future has had problems. These problems are still not solved in as this paper is being written.

8:55 AM

Magnesium Electrolysis-A Monopolar Viewpoint: Oddmund Wallevik[2]; *Ketil Amundsen*[1]; André Faucher[3]; Thorvald Mellerud[4]; [1]Hydro Magnesium, Oslo N-0246 Norway; [2]Norsk Hydro ASA, Rsch. Ctr., Porsgrunn N-3901 Norway; [3]Norsk Hydro Canada, Bécancour, Canada; [4]Hydro Magnesium, Brussels, Belgium

Norsk Hydro has produced magnesium in Porsgrunn, Norway, since 1951. The technology including the electrolysis was "inherited" from IG Farben. Hydro has since then continuously developed the magnesium electrolysis, first by improving the IG technology, and then by developing its own "diaphragmless electrolyser" (DLE), now being used for a number of years in Norsk Hydro's plants in Porsgrunn as well as in Bécancour, Québec. A presentation will be made of the Norsk Hydro high-amperage monopolar electrolysis cell. Its performance will be described, as a basis for the conclusion that this type of cell presently is very competitive compared to bipolar cell technologies, although it has a higher energy consumption.

9:20 AM

Investigation on Electrocatalysis for Energy Saving in Magnesium Electrolysis: *Zhong Xie*[1]; Ye-Xiang Liu[1]; [1]Central South University of Technology, Dept. of Nonfer. Metallu., #2 Lushan Nan Rd., Changsha, Hunan 410083 PRC

In the present paper, we report some research results on electrocatalysis for chlorine evolution reaction (CER) in Mg electrolysis. Investigation has been carried out on various oxide electrodes in equimolar NaCl-KCl melts at 700°C. Oxide electrodes were prepared by thermal decomposition procedure on graphite substate. Steady state linear potential sweep and an improved current interruption technique were used to investigate the electrocatalytic activity of transition metal oxides, rare earth oxides and their mixed oxides. Pr6O11, Tb4O7, Tm2O3, RE+Ru and CoxFeyOz (x=0.75,y=2.25,z=4) mixed oxides showed good active for CER, reducing anode overpotential up to 80-110 mv at a current density of 0.6A/cm.sq. compared with graphite electrode. 200-250kWh/T-Mg energy saving could be achieved if above mentioned electrodes being employed in Mg electrolysis. Attempt was made to elucidate the electrocatalysis mechanism.

9:45 AM

Inert Anodes for Magnesium Electrolysis: Jerry F. Moore[1]; *John N. Hryn*[1]; Michael J. Pellin[1]; W. F. Calaway[1]; Kevin Watson[2]; [1]Argonne National Laboratory, Energy Sys. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Noranda Inc., Noranda Tech. Ctr., 240 Boul. Hymus, Pointe-Claire, Quebec H9R1G5 Canada

Magnola Metallurgy Inc. (MMI) will commission a 63,000 tpa primary magnesium production plant in June 2000. The plant will produce magnesium using electrolysis cells with industry standard carbon anodes. Undesirable by-products of the electrolysis process with carbon anodes are chlorinated hydrocarbons (CHCs). These represent a significant environmental concern, which MMI are committed to reducing and eventually eliminating. One possible means to prevent the production of CHCs is to replace the carbon anodes with non-carbon "inert" anodes. The development of a viable inert anode material has been a goal for the magnesium industry for many years. To date, no acceptable material has been found. The major technical hurdle is to develop a material with the ability to withstand chlorination by the evolving chorine gas on the anode surface whilst conducting electrical current efficiently. Argonne National Laboratory (ANL) has identified certain metal alloys that are promising candidate materials for inert anodes. These alloys form self-limiting surface oxide films that are thin enough to allow current to pass, yet thick enough to prevent chlorination of the underlying metal. ANL are investigating the use of these alloys as inert anodes under the joint sponsorship of MMI and the U.S. DOE, Office of Science, Laboratory Technology Research

Program. This paper presents a review of previous inert anode research and an update of the ANL project.

10:10 AM Break

10:20 AM

The Magnola Demonstration Plant: A Valuable Investment in Technology Development and Improvement: *K. Watson*[1]; P. Ficara[2]; M. Charron[2]; J. Peacey[2]; J. Primak[2]; [1]Noranda Inc., Noranda Tech. Ctr., Pointe-Claire, Quebec Canada; [2]Magnola Metallurgy Inc., Magnola Proj. Off., 620 Rene-Levesque W., 10th Fl., Montreal, Quebec H3B1N7 Canada

Noranda will become a major producer of magnesium following the commissioning of its 63,000 tpa Magnola plant in June 2000. The Magnola plant will utilise new process technology to extract magnesium from serpentine mine tailings. The technology is unique and will enable Noranda to become the world's premier primary magnesium producer. This paper presents an historical overview of the Magnola project from the preliminary process development at Noranda Incorporated's Technology Centre through to operation of the pilot demonstration plant in 1996 & 1997 and finally to design of the full scale Magnola plant. In particular, this paper will present some of the major conclusions and improvements derived from the demonstration plant stage and will evaluate their impact on the development of the overall Magnola Process. An update of the current status of the plant construction and pre-commissioning is also provided.

10:45 AM

Magnesium Electrolytic Production Process: G. Shekhovstov[1]; V. Shchegolev[1]; *V. Devyatkin*[1]; V. Tatakin[1]; I. Zabelin[1]; [1]Titanium Institute, 180 Prospect Lenina, Zaporozhue 330035 Ukraine

The Titanium Institute/VAMI proposes two variants of the magnesium electrolytic production process. The first variant is based on a two-stage preparation process of magnesium raw material carnallite (KCl.MgCl2.6H2O) for electrolysis. In the first stage, carnallite is dehydrated in fluidized bed dryers with output of 400 t/day. Operation and control of the drying process is highly automated. The second stage of carnallite dehydration is carried out in electric chlorinators with output of 150-200 t/day. In the chlorinators, carnallite is melted and chlorine treated. Molten carnallite flows to the electrolysis cells. The electrolysis cells are connected in a flow line that operates as one highly productive electrochemical unit. By electrolyte flow, magnesium moves through the electrolysis cells and is accumulated in a separator cell where it is extracted and passed to the casting house for casting magnesium and magnesium alloys into ingots. Electrolysis cells current is about 200-300 kA. Magnesium and magnesium alloys are refined in continuous refining furnaces with the capacity of up to 100 t/day. The process is adopted by magnesium and titanium-magnesium plants of Russia, Kazakhstan, and Ukraine. The best modern projects are realized at the DSM magnesium plant in Israel. The second variation of magnesium electrolysis production process is based on carrying out high dehydration of carnallite in fluidized bed dryers by HCl injection into chambers together with combustion gases. HCl is gained from fuel burning in chlorine gas in magnesium electrolysis cells. Solid highly dehydrated carnallite is charged into cells connected into flow lines having centralized magnesium collection. This process has passed pilot-commercial tests and is ready for industrial realization. The above-mentioned carnallite processing variants can be used for different types of raw material: magnesite, chlorine-magnesium solutions, sea water, dolomite, and carnallite.

11:10 AM

Solid-Oxide Oxygen-Ion-Conducting Membrane (SOM) Technology for Direct Reduction of Magnesium from Its Oxide: *D. E. Woolley*[1]; U. B. Pal[2]; G. B. Kenney[3]; [1]Boston University, Boston, MA USA

Abstract Text Not Available

11:35 AM

Comparison of Fused Cast Alumina Products for Magnesium Chloride Cells: *Alexandre Mauries*[1]; D. A. Whitworth[1]; [1]SEPR, R&D, B.P. #1, Le Pontet, Cedex 84131 France

The molten salt electrolysis of magnesium chloride requires high quality refractories. The reduction of silicon dioxide by magnesium involves the use of high alumina refractories. The sintered refractories fail because of the penetration of the low viscosity salts into the open porosity. The high alumina fused cast refractories appear to be the best solution in this application. Different types of high alumina fused cast products have been prepared and tested in SEPR laboratory. The main properties demanded by the application have been taken into account: low penetration by the molten salts (porosity, cracks, crystal size), chemical stability in contact with MgCl2, high electrical resistivity. Some general trends can be drawn regarding the application of fused cast alumina products in magnesium chloride cells.

Opportunities for Materials & Engineering Research Funding From Government & Industry: Session 1

Sponsored by: TMS, Public & Governmental Affairs Committee, Young Leaders Committee

Program Organizers: Canan U. Hardwicke, General Electric Company, GECRD, Niskayuna, NY 12309 USA; Samuel A. Davis, TIMET, Henderson, NV 89009 USA

Monday AM — Room: Bayou A
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Canan U. Hardwicke, General Electric Company, GECRD, Niskayuna, NY 12309 USA; Samuel A. Davis, TIMET, Henderson, NV 89009 USA

8:30 AM

An Outline of the Federation of Materials Societies (FMS): *John N. Mundy*[1]; [1]Consultant, 10720 Game Preserve Rd., Gaithersburg, MD 20879-3106 USA

FMS is an umbrella organization whose member societies and affiliates represent the professional societies, universities and National Research Council Organizations which are involved with materials science, engineering, and technology. The purpose of FMS is to aid the materials community in obtaining information from and exchanging information with the policy community. An important FMS goal is to help the materials community to arrive at consensus materials policy and to assist it in informing policy makers of materials concerns. TMS is a contributing member of FMS and it is valuable for members of TMS to be aware of how FMS fulfills these tasks.

8:55 AM

An Overview of R&D in the Federal Government with Emphasis on DOE's Materials Research Programs: *Louis Ianniello*[1]; [1]DOE (retired)

Research funding by the Federal Government approximates $80 billion per year, covering basic, applied and technology development, both defense and civilian areas. Materials research is conducted and supported to some extent by almost every agency of the government. The three largest Materials Research funding agencies for external support are the Department of Defense (DOD), the National Science Foundation (NSF), and the Department of Energy (DOE). The talk will help the audience put the Materials Research portion of the Federal budget into perspective and then provide some detailed information on the DOE programs. DOE conducts research at its own laboratories and also funds research proposals submitted in response to solicitations as well as unsolicited proposals. The talk will include information on the various offices that fund materials research and on opportunities for doing research and interacting technically with different DOE programs.

9:20 AM

Materials Opportunities in Energy Efficiency: *Toni Grobstein Marechaux*[1]; [1]U.S. Department of Energy, EE-20, 1000 Independence Ave. S.W., Washington, DC 20585-0121 USA

Energy its production, use, and conservation is a huge part of the world economy, and materials enable much of the new technology needed to improve energy production and to use it more efficiently. The Office of Energy Efficiency and Renewable Energy in the US Department of Energy provides a wide variety of options in funding for research and development, from small grants for the development of inventions to industry cost-shared demonstration projects, and many types of research and development in between. Opportunities for materials funding in the areas of industrial, transportation, power, and building technologies will be discussed.

9:45 AM

Opportunities for Materials Technologies in the Advanced Technology Program: *Clare M. Allocca*[1]; [1]NIST, Adv. Tech. Pgm., 100 Bureau Dr. Stop 4730, Bldg. 101, Rm. A225, Gaithersburg, MD 20899-4730 USA

The NIST Advanced Technology Program (ATP) is a unique partnership between government and private industry to accelerate the development of high-risk technologies that promise significant commercial payoffs and widespread benefits for the economy. The Materials Technology community has already taken advantage of several ATP opportunities, and as a result has moved ahead. Following an overview, this presentation will describe past and potential accomplishments/opportunities in the area of materials technologies, including engineered surfaces and innovative forming techniques. In the area of engineered surfaces, industry has identified high risk, critical technical investments to include: (1) simultaneously improving engineered surface process designs and reducing cost through reduced development time and increased yield and consistency; and (2) developing extremely reliable and predictable surfaces which are integral to the design and operation of a component, as opposed to mere life enhancement. Tools (e.g., process diagnostics and life/performance prediction) and corresponding validation strategies have been identified as key to overcoming these barriers. In the area of innovative forming techniques, industry has identified, as appropriate for ATP, technical barriers to implementation of rapid prototyping/solid freeform fabrication, the aspects of metals (and metal matrix composites) forming which represent a major leap forward rather than an incremental step, and next generation technologies for ceramics and ceramic matrix composites forming. Potential investment areas include predictive tools for process control and scale-up, intelligent tool design, and innovative approaches to design optimization and automation.

10:10 AM Break

10:20 AM

Thoughts on Federal Research Funding in MSE with Specific Examples Related to the National Science Foundation: *Bruce A. MacDonald*[1]; [1]National Science Foundation, Div. of Matls. Rsch., 4201 Wilson Blvd., Rm. 1065, Arlington, VA 22230 USA

There are widespread opportunities for research support by the U.S. Federal Government in materials science and engineering. This can be viewed as an advantage since an investigator is not tied to one particular source for funding. The disadvantage is that there is no single key to gaining federal support since the various funding agencies may have different goals and funding criteria. Clearly the pursuers of funds should be aware of the objectives and proposal requirements of the agency from which they are requesting support. Gaining this knowledge is complicated by the fact that the funding environment is dynamic; therefore, it is incumbent upon the investigator to maintain frequent contact with the agencies from which they want and/or have support. In this discussion I will suggest ways in which this contact can be developed, as well as describing various research initiatives at the NSF.

10:45 AM

Collaborative Research Opportunity for New Faculty: *Reza Abbaschian*[1]; [1]University of Florida, Dept. of Matls. Sci. and Eng., Gainesville, FL 32611-6400 USA

Materials science and engineering research in U.S. universities has been sponsored mainly by the federal government. The mission oriented agencies (mainly DOD and DOE) have provided about 60% of academic research. The agencies, however, have been shifting funds toward applied and systems-oriented research. Many U.S. industries have also eliminated central research laboratories to align their R&D activities more closely with immediate business opportunities. Within academia, the pressure on the faculty remains the same, to educate as many graduate students as they can support. In light of the above, more research in universities is being conducted collaboratively, and in partnership with industry and government laboratories. This provides great opportunity for new faculty to establish their own graduate training and research programs. However, a proper balance between collaborative and individual research must be maintained.

11:10 AM

University/Industry Cooperative Applied Research Initiative: How It Works: *Manoranjan (Mano) Misra*[1]; [1]University of Nevada, Metallu. and Matls. Eng., Mackay School of Mines, Reno, NV 89557 USA

The thrust of this paper is to highlight the successful accomplishments of the Applied Research Initiative program at the University of Nevada, Reno. The industry/university partnership has brought several industries to work with junior and senior faculty in many research projects in the Metallurgical Engineering program. In addition, the program helped in generating large scale funding from the federal agencies. In this talk, several factors including how to develop such infrastructure will be discussed.

Packaging & Soldering Technologies for Electronic Interconnects: Soldering and Packaging Technologies

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

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

Monday AM — Room: Lincoln D
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: G. Ghosh, Northwestern University, Dept. of Mats. Sci., Evanston, IL 60208-3108 USA; P. T. Vianco, Sandia National Laboratories, Matls. Joing Dept., Albuquerque, NM 87185-1411 NM USA

8:30 AM Opening Remarks

8:35 AM Invited

Solder Reaction on Electroless Ni(P) in Low Cost Flip Chip Technology: *K. N. Tu*[1]; [1]University of California, Dept. of Mat. Sci. Eng., Los Angeles, CA 90095-1595 USA

The wetting reaction between eutectic SnPb solder and electroless-Ni(P) under-bump-metallization has been studies by SEM, TEM, EDX and e-probe microanalysis. Two findings are interesting. The first is the enhanced crystallization of the electroless Ni(P), which has an amorphous structure in the as-plated state. The second is the lateral penetration of solder along the interface between the electroless Ni(P) and the dielectric SiON substrate. The crystallization leads to the formation of Ni_3P and Ni_3Sn_4 compounds. The penetration is accompanied by the formation of Ni_3Sn_4 compound. The kinetics of the crystallization and penetration will be presented.

8:55 AM Invited

Evolution of Interfacial Morphology during Reaction of Copper and Electroless Nickel with Eutectic Pb-Sn Solder: A. S. Zuruzi[1]; *S. K. Lahiri*[1]; [1]Institute of Materials Research and Engineering, 10 Kent Ridge Crescent 119 Singapore

Interfacial morphology of the intermetallics formed during the reflow operation has an important effect on the wettability of the underbump metallurgy and reliability of the solder joints. Evolution of the interfacial morphology during reflow is therefore of considerable interest for scientific as well as technological reasons, particularly from the viewpoint of reworking of assembled components. With the present trend towards smaller and lighter electronic components, there is currently a need in the microelectronics industry for direct attachment of chip to organic substrates using the flip chip bonding technique. However, the use of organic materials requires lower reflow temperatures during the assembly process than that needed for reflowing conventional high Pb solder on chip terminal pads. Use of eutectic Pb-Sn solder with electroless nickel underbump metallurgy, instead of the usual copper based metallurgy, is one of the methods which has recently been the subject of investigation by a number of researchers for implementing a low temperature reflow process[1,2]. This paper will address and compare the evolution of the interfacial morphology during reaction of eutectic Pb-Sn solder with electroless nickel and copper. 1. J. Kloeser, K. Heinricht, K. Kutzner, E. Jung, A. Ostman, E. Zakel, and H. Reichl, Proc. ECTC, pp. 254-264 (1997). 2. J.W. Wang, P.G. Kim, K.N. Tu, D.R. Frear, and P. Thomson, J. Appl. Phys., 85, 8456-8463 (1999).

9:15 AM Invited

Wetting of Low Melting Point Alloys on Metal Substrates: *Timothy J. Singler*[1]; Stephan J. Meschter[1]; [1]SUNY Binghamton, Mech. Eng., Binghamton, NY 13902-6000 USA

The wetting dynamics of low melting point alloys on metal substrates is assessed using drop spreading. We observe a primary wetting regime characteristic of many inert liquid systems and a secondary spreading regime characteristic of product-forming reactive systems. We study the influence of temperature, and discuss its effects on several interesting spreading phenomena including the appearance of transient solid phases. We explore the role of surface coatings, particularly their effects on wetting kinetics and contact line morphology.

9:35 AM

Application of Soldering in Partial Melting Zone to Grid Area Package Using Pb Free Hyper-Eutectic Solders: *Jun Seok Ha*[1]; Jae Yong Park[1]; Jae Pil Jung[2]; Choon Sik Kang[1]; [1]Seoul National University, Matls. Sci. & Eng., Shillim-dong, Kwanak-gu, Seoul 151-742 South Korea; [2]University of Seoul, Matls. Sci. & Eng., Jeonnong-dong, Dongdaemun-gu, Seoul South Korea

Unlike the conventional soldering process, the partial melting soldering method is performed above the eutectic temperature and between the eutectic point and the liquidus line. Because it is carried out in the mushy zone, the liquid and the solid phases co-exist. This method will enable us to use other alternative solders than the ones with eutectic composition and thus accelerate development and application of new lead-free solders by offering us a broader range of alternatives. In this study we applied lead- free hyper-eutectic solders (Sn-Bi Sn-In and Sn-Ag) to the partial melting soldering method and investigated the possibility of bonding, metallurgical characteristics and microstructural evolution. A grid array-type substrate was fabricated for soldering in drying oven and conventional reflow soldering machine with hot-air reflow type. Various solder compositions were used in the liquid-solid interface region and experiments were carried out in several temperature ranges. To determine wettability between partial melted solder and Cu substrate, wetting analysis test was conducted at each composition-temperature variable set, and also the test results were compared with the case of full melted solder. After soldering, Scanning Electron Microscope (SEM was used for observation of microstructure and its evolution during aging. To check the soldered joint strength, shear and tensile tests were performed.

9:55 AM

The Possibility of Soldering in Partial Melting Zone Using Hyper-Eutectic Sn-Pb Alloys: *Jae Yong Park*[1]; Jun Seok Ha[1]; Jae Pil Jung[2]; Choon Sik Kang[1]; [1]Seoul National University, Matls. Sci. & Eng., Shillim-dong, Kwanak-gu, Seoul 151-742 South Korea; [2]University of Seoul, Matls. Sci. & Eng., Jeonnong-dong, Dongdaemun-gu, Seoul South Korea

Conventional soldering technology is performed above the full melting temperature region of low melting point solders; the massive trend toward Pb-free solders is not free from this type of temperature restraint in terms of the choice of usable solders. The temperature limit narrows down the choice of usable alternatives to Sn-Pb solders to tin-based alloys with eutectic or near eutectic composition. Off-eutectic alloys, however, with a high temperature liquidus line can be also used as solders in the partial melting temperature zone. The liquid state and the solid state coexist in the partial melting temperature zone, and the portion of liquid state is determined by the lever rule in the phase diagram. To investigate the possibility of soldering in the partial melting temperature zone, the hyper eutectic Sn-Pb alloys and Cu plates were interconnected in the temperature range between the eutectic temperature and liquidus temperature. Drying oven and conventional reflow soldering machine with hot-air reflow type were used. To verify the joint state, a microstructural observation was carried out using Scanning Electron Microscope (SEM). Shear test was conducted to check the strength of the soldered joint interconnected in the partial melting temperature zone. The test results showed a sustainable strength of joint between hyper-eutectic solders and Cu substrate interconnected in the partial melting temperature zone.

10:15 AM Break

10:30 AM Invited

Development of Fluxes for Lead-Free Solders: *Semyon Vaynman*[1]; Morris E. Fine[1]; [1]Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL 60208 USA

New lead-free solders, that contain zinc are promising candidates to replace near-eutectic tin-lead solders, because these solders have lower melting temperatures than those based on the tin-silver eutectic, posses good mechanical and fatigue properties and are less expensive than other alternatives to lead containing solders. However, the contact angle on copper for Sn-Zn solders is rather high when utilized with fluxes used for Sn-Pb solders. A novel approach for flux development to improve wetting of copper surface by tin-zinc eutecticis uses tin containing organic compounds as an additive. These metallo-organics decompose at soldering temperatures and produce metallic tin on the surfaces to be soldered. This process improves wetting of copper surface by molten tin-zinc eutectic.

10:50 AM

Reliability Investigation of Printed Wiring Boards Processed with Water Soluble Flux Constituents: *W. Jud Ready*[1]; Laura J. Turbini[1]; [1]Georgia Institute of Technology, Matls. Sci. and Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

An investigation was conducted to evaluate the effect on reliability of water soluble flux and fusing fluid constituents used in the manufacture of electronic product. The purpose of this program was to determine the mean-time-to-failure (MTTF) of FR-4 substrates processed with three different water soluble flux formulations. The fluxes incorporated hydrochloric acid, hydrobromic acid, a polyglycol and a polyether. In order to determine the MTTF for conductive anodic filament (CAF) growth a test coupon with variable hole-to-hole spacings was used. The variables being studied were (1) flux type, (2) conductor spacing and (3) operating voltage. Quantification of the effect of these variable is determined through a series of MTTF tests. In addition to the electrical measurements, optical and scanning electron microscopy (SEM) is used with energy dispersive X-ray spectroscopy (EDS) to determine the chemical nature of the CAF.

11:10 AM

Effect of Plasma Cleaning on the Flip-Chip Bonding Properties of Si-Wafer/Bumps/Glass System: *Soon Min Hong*[1]; Chang Bae Park[2]; Jae Yong Park[1]; Jae Pil Jung[2]; Choon Sik Kang[1]; [1]Seoul National University, Divi. of Matls. Sci. and Eng., Shillim-dong, Kwanak-gu, Seoul 151-742 Korea; [2]University of Seoul, Matls. Sci. & Eng., Jeonnong-dong, Dongdaemun-gu, Seoul, South Korea

The flip chip provides the highest packaging density and performance and the lowest packing profile among other assembly methods. As the packaging density grows high, however, the cleaning of flux used in conventional process becomes increasingly difficult. The flux residue can seriously affect the reliability and performance of flip chip assemblies by corrosion. In addition, the chemical solvent for flux

cleaning process can also cause the environmental problem which is a world-wide concern in recent years. The purpose of this research is to evaluate the fluxless flip chip bonding properties between Si wafers and Au-coated glass substrates in optical recording media application using Sn-Pb, Sn-Ag, Sn-Bi micro solder bumps. The solder bumps were electroplated on the UBM(Cr/Cu)-deposited Si wafer and reflowed. The pitch of the bumps was 50-150¥ ìm. Instead of flux, we used the Ar plasma cleaning to remove the oxides and other contaminants of solder bumps and glass substrates before flip chip bonding. The wettability of the Au-coated glass and UBM-coated Si wafer were estimated by meniscograph method and area of spread method. The mechanical properties of the joint were examined by micro defect analysis and micro tensile test. The bonding properties were compared with those of the bonding process using flux. The cross section of the solder joint and the intermetallic compounds between solder and coating materials were analyzed by optical microscopy and SEM. The effect of flip chip process parameters, such as temperature profile, conveyor speed and atmosphere, on the bonding properties were also discussed.

11:30 AM

Characteristics of the Sn-Pb Eutectic Solder Bump Formed via Fluxless Laser Reflow Soldering: Jong-Hyun Lee[1]; Jong-Tae Moon[2]; Yong-Ho Lee[1]; *Yong-Seog Kim*[1]; [1]Hong Ik University, Metallu. and Matls. Sci., Sangsu-dong Mapo-Cu, Seoul Korea; [2]Hyundai Electronics Company, Device and Semiconductor Rsch. Div., Ichon 467-701 Korea

With concerns on the environmental contamination and the reliability of the electronic devices, many attempts were made to develop a fluxless reflow soldering process in electronic packaging. In this study, the fluxless reflow soldering was conducted via heating the solder bump using CO_2 laser under controlled atmosphere. A solder disk was placed on the pre-tinned contact pad of the Si wafer and heated with the laser for reflow bumping. The effects of heat input, reflow soldering atmosphere, solder compositions on the mechanical properties and thermal stability of the solder bump were investigated. The heat input to a solder bump was changed from 20 to 60 J and the soldering atmosphere argon, helium, nitrogen, and air. The solders used were Pb-Sn eutectic solder, Pb-Sn composite solder reinforced by in-situ Cu6Sn5 dispersoids, and Sn-3.5%Ag. The reflow soldering under argon helium, nitrogen atmosphere resulted in spherical reflowed bumps. Microstructural observation of the reflowed bump showed a very thin intermetallic layer formed at the solder/contact pad interface. Higher heat inputs resulted in an excessive dissolution of the contact pad metals and lower heat inputs in an insufficient reflow. Shear strengths of the reflowed solder joints measured were equal to those of the reflowed joints formed in a furnace. A study on the mechanisms of the fluxless reflow soldering via the laser heating were conducted by measuring the oxygen concentration distribution in the solder bump by Auger Electron Spectroscopy as well as by estimating the thermal stress developed during the laser heating by thermal modeling of the solder bump.

11:50 AM Invited

Measurements of Metal/Polymer Adhesion Strengths in Microelectronic Packaging: *Jin Yu*[1]; [1]Korea Advanced Institute of Science and Technology, 3373-1 Kusong-dong Yusing-ku, Taejon, Korea

In plastic packages, residual stresses arising from thermal mismatches or pressures exerted by vaporized moisture often lead to the delamination of metal/polymer interfaces. Modifications of polymer surfaces by rf plasma and oxidation treatments of metal surfaces before joining dissimilar materials are commonly used to enhance the adhesion strength of the interface, which is measured by a plethora of techniques. In the present analysis, adhesion strength of Cu/Polyimide and Cu-based leadframe/epoxy were measured using the peel test, pull-out test, and several fracture mechanics tests, and effects of the plasma and oxidation treatments and near the crack tip stress states were investigated. Then, correlations between the peel strength, pull strength and the interface fracture toughness were presented, and the relations among the peel strength, interface fracture toughness and the work of adhesion were discussed based on the X-ray measurements of plastic dissipation and the theoretical analysis.

Pressure Technology Applications in the Hydrometallurgy of Copper, Nickel, Cobalt and Precious Metals: Process Design and Engineering Considerations in High Pressure Hydrometallurgy

Sponsored by: Extraction & Processing Division, Copper, Nickel, Cobalt Committee

Program Organizers: James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

Monday AM — Room: Lincoln C
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Jussi Asteljoki, Outokumpu Oy, Corporate Res. & Dev., Espoo 02201 Finland; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

8:30 AM

Process and Engineering Considerations in the Pressure Decopperizing of Copper Refinery Slimes: *James E. Hoffmann*[1]; [1]James E. Hoffmann and Associates Company, P.O. Box 420545, Houston, TX 77242-0545 USA

Oxidative pressure leaching is gradually supplanting all other techniques for the decopperizing of copper refinery slimes. The reasons for this include: much more rapid reactions, more compact equipment, and perhaps most important, a much lower final copper concentration in the decopperized slimes. This paper will first discuss the reactions occurring during decopperizing, the acidity and temperatures encountered and the techniques employed for discharging non condensable gasses. A spreadsheet is provided which allows adjusting the operating conditions depending upon raw (undecopperized) slimes composition, solids concentration, and lixiviant composition. Typical process flowsheets and process equipment flowsheets are provided.

9:00 AM

Optimizing Gas Mass Transfer in Autoclaves: *Peter Forschner*[1]; Stefan Land[1]; Ronald Klepper[2]; [1]EKATO Ruhr-und, Mischtechnik GmbH, Schopfheim, Germany; [2]EKATO Corporation, Salt Lake City, UT USA

In hydrometallurgy, pure gases like oxygen and hydrogen are used for oxidation and reduction. The gases are expensive and therefore should be used efficiently. Non reacted gas can be recycled into the liquid. Historically vortex gassing has been used, i.e. a mixing impeller close to the liquid surface entrains gas from the headspace into the liquid through a gas vortex. Novel and more efficient methods are surface gassing with up pumping impellers and self-inducing gassing impellers. These alternative impeller systems are well established and common in the chemical industry, but have not been used until recently in hydrometallurgy. Gassing impellers act as internal compressors and increase the interfacial surface for a much higher mass transfer compared to alternate methods mentioned. In the paper results from lab and pilot tests and data from operated plants will be presented. The reactions with gases normally take place under moderate to high pressure, therefore a safe enclosure with mechanical seals and the safety “philosophy” is important. Experienced life and replacement cycles will be reported.

9:30 AM

Laboratory Autoclaves for Hydrometallurgical Research: *Fathi Habashi*[1]; [1]Laval University, Dept. of Min. and Metallu., Quebec City G1K7P4 Canada

Laboratory autoclaves for hydrometallurgical investigations are available in a variety of sizes, models, and materials of constructions. They vary in sizes from 25 ml to 2 liters for laboratory studies and 5

to 50 gallons for pilot plant work. They are essential tools for studying aqueous oxidation of sulfide concentrates, dissolution of oxide minerals at high temperature and pressure and hydrothermal precipitation reactions. The maximum pressure and temperature at which any pressure vessel can be used will depend upon the design of the vessel and the materials used in its construction. Since all materials lose strength at elevated temperatures, any pressure rating must be stated in terms of the temperature at which it applies. A review of existing models and their accessories will be given.

10:00 AM Break

10:15 AM

Design Considerations in Autoclaving: *Ir. Herman Pieterse*[1]; [1]Pieterse Consulting Inc., 6321 N. Calle Campeche, Tucson, AZ 85750 USA

The hydrometallurgical route, due to the fact that it often provides increased recovery, reduced air and water pollution, and lower capital costs relative to roasting and smelting, is becoming the preferred route for many metals. The design of autoclave circuits for various metals are discussed. Emphasis is placed on kinetics, energy balance, heating, cooling, retention, mixing, mass transfer, letdown, pumping, materials of construction, equipment selection. The leaching of copper, as one of the few applications where autoclaving has not been widely applied is highlighted.

10:45 AM

Titanium Clad, High Pressure Acid Leach Autoclaves for Nickel Laterite Ore Processing: *George A. Young*[1]; [1]Dynamic Materials, Metallu. Dept.

High pressure, acid leach autoclaves have been recently installed in Australia to process laterite ores of nickel. As a preferred design over brick/lead lining all three projects have used titanium clad (integral lined), carbon steel pressure vessel autoclaves. The design features horizontal, chambered vessels with agitation. Based on various factors including ore chemistry all three projects specified different grades of titanium, i.e., gd. 1, gd. 11 and grade 17. This paper will review some of those grade selections decisions disclosed by the owners. Additionally, performance of the titanium grades may be difficult to determine with one year or less service but some autoclave internal inspections have been conducted. Comments from the operators on autoclave and material performance will be included. Dynamic Materials, Inc., Lafayette, CO supplied the titanium-clad steel for the autoclave vessels for two of three projects, Bulong and Murrin-Murrin. ASC-E, Adelaide Australia fabricated the vessels for all three projects including Cawse. Several other projects are awaiting final approval to proceed.

Surface Engineering in Materials Science I: Coatings/Films Synthesis and Processes (SP)-I

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

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

Monday AM — Room: Canal B
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: I. Manna, IIT, Metallu. and Matls. Sci. and Eng., Kharagpur, WB 721302 India; Brajendra Mishra, Colorado School of Mines, Adv. Coatings and Surface Eng. Lab. Dept., Golden, CO 80401-1887 USA

8:30 AM

Oxidation-Resistant Coatings for Molybdenum Electrodes: *Sury Challapalli*[1]; Earl Hixson[1]; John Moore[1]; [1]Colorado School of Mines, Adv. Coati. and Surf. Eng. Lab., Dept. of Metallu. and Matls. Eng., Golden, CO 80401-1887 USA

The high-temperature oxidation resistance of molybdenum can be significantly improved by coating it with MoSi2. But, at the high temperature of operation, silicon from MoSi2 diffuses into the molybdenum substrate and the oxidation resistance of the system deteriorates. Further, because of the CTE mismatch between Mo and MoSi2, the composite breaks down and spalls on thermal cycling. To alleviate the problem of the CTE mismatch, the CTE of MoSi2 is matched with that of Mo by the addition of 50 wt% SiC. But, the problem of silicon (and now additionally carbon) diffusion into the molybdenum substrate persists, changing the chemistry of the overlayer and deteriorating the oxidation resistance of the composite. Incorporating a diffusion barrier layer between the Mo substrate and the MoSi2+SiC composite layer on the top solved this problem. The newly developed amorphous diffusion barrier layer prevents diffusion of both carbon and silicon into the substrate. A number of problems still need to be resolved with respect to the diffusion barrier layer. These concern the chemistry, thermal stability, crystallization behavior, and cyclic oxidation resistance. Finite element modeling studies are also being undertaken to determine the optimal thickness of the individual layers, and the compositional gradient of the MoSi2+SiC composite to provide a tolerable residual stress level and also for optimal distribution of that stress in the overall coating system. These results will be utilized to optimize the diffusion barrier layer thickness. The present talk will review the recent results obtained and highlight the future investigations. Supported by NSF under DMR Award # 9730775.

8:50 AM Invited

Synthesis of Nanocrystalline Inconel 625 Powders by Cryomilling: *Degang Cheng*[1]; Rudy Rodriguez[1]; Mike Ice[1]; E. J. Lavernia[1]; [1]University of California Irvine, Dept. of Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697-2575 USA

The present paper report on a successful synthesis of nanocrystalline Inconel 625 powders by cryogenic high-energy ball milling (cryomilling). Commercially available Inconel 625 powders (Diamalloy 1005 AMDRY 625) is milled in liquid nitrogen for 8 hours. The characteris-

tics of the milled powders, i.e., morphology, self-agglomeration, powder size, grain size and structure evolution during milling, were analyzed using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

9:15 AM Invited

Film Formation on Metals and Alloys by Thermal, Electrochemical and Plasma Oxidation: *D. L. Cocke*[1]; S. Promreuk[1]; D. G. Naugle[2]; R. B. Schennach[1]; [1]Lamar University, Gill Chair of Chem. and Chem. Eng., P.O. Box 10022, Beaumont, TX 77710 USA; [2]Texas A&M University, Dept. of Physics, College Station, TX 77840 USA

Interfacial oxidation is an established approach to produce surface thin films for catalysts, corrosion and ware protective coatings, and electronic structures. The three main methods, thermal, anodic and plasma, for oxidation of metallic substrates still lack an adequate fundamental physical-chemical models, that can allow film design, particularly on alloys. Oxidation of alloys produce multicomponent oxides with quite different structures depending on the conditions and the methods of oxidation. The three methods will be discussed in terms of physical/chemical parameters that influence the chemical nature and structure of the resulting oxides. The electrochemical processes that occur during the materials reaction with a chosen environment will be used to discuss the physical and chemical mechanisms involved. Intrinsic and extrinsic electric fields will be shown to influence the chemical and structural nature of the resulting oxide structures. Surface analysis results [XPS (X-ray Photoelectron Spectroscopy) and ISS (Ion Scattering Spectrometry)] from the three oxidation methods applied to metals and their binary and ternary alloys will be discussed. The resulting structures on the ternary and binary alloys will be discussed in terms of an evolving electrochemical model.

9:40 AM

Study of Operating Conditions on Electroless Plating of Nickel for MEMS Application: *Jin Huh*[1]; Jae-Ho Lee[1]; [1]Hong Ik University, Dept. of Metallu. Eng. and Matls. Sci., Seoul 121-791 Korea

Electroless depositions of nickel were conducted in different bath conditions to find optimum conditions of electroless nickel plating for fabrication of micro structures in MEMS applications. In LIGA or LIGA-like process, the operating temperature and pH is restricted and optimum operating conditions were different from conventional electroless deposition of nickel. And then to find optimum conditions, the effects and selectivity of activation method on several substrates were investigated. The effects of nickel salt concentration, reducing agent, complexing agent and inhibitor on deposition rate was investigated. The effect of pH on deposition rate and content of phosphorous in deposited nickel was also observed. In optimum operating bath condition, deposition rate was 7?/hr at 60? and pH 10.0. The rate was decreased with stabilizer concentration. And then micro structures were fabricated with optimum bath condition.

10:00 AM

Electron Beam Enhancement of Composite Coatings: *Vadim J. Jabotinski*[1]; Francis H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., Moscow, ID 8384-3026 USA

Electron beam processing offers great technical and economic capabilities of improvement of composite coatings. Heating combined with radiation treatment offering by the electron beam processing allows novel changes and modification in coating materials. This paper will consider fundamentals and applications of the electron radiation in processing traditional and advanced coating materials including high-temperature superalloy-, intermetallic-, and ceramic-matrix composites applied by conventional techniques such as thermal spraying, high velocity oxygen fuel, plasma, laser, and physical and chemical vapor deposition. The specific effects leading to increase in the coating lifetime and wear, heat, and corrosion resistance will be identified. Possible mechanisms for increasing the toughness, adhesion, bond strength and reducing porosity and cracks will be discussed.

10:20 AM Break

10:35 AM

Modeling of Combustion Flame Assisted Chemical Vapor Deposition of Diamond Thin Films: *J. Kapat*[1]; K. Elshot[1]; [1]University of Central Florida, Dept. of Mech., Matls. & Aero. Eng., Orlando, FL 32816-2450 USA

Combustion flame assisted chemical vapor deposition (CFCVD) is of interest to researchers because of its simplicity. The main advantages of this method over other CVD methods include higher growth rates, low cost, and the potential to deposit a large area. In this study, a numerical model has been developed for analyzing chemically reacting flow, mass and heat transport and reactions in CFCVD of a diamond thin film with a laminar, premixed C2H2/O2 flame. This model is used to numerically investigate the effect of different process parameters on the growth rate of the thin film. In this model, the flame is held perpendicular to a Si substrate, where the film is deposited. The temperature of the substrate is controlled independent of the flame temperature as the substrate is attached to an externally cooled copper substrate. As would be discussed later, the substrate is placed within the feather region of the flame for optimum diamond deposition. Chemical model discriminates between graphitic and diamond phases through the use of different surface reactions so as to model the differential etch and growth rates of these two phases. The focus of this paper is to present the effect of inlet gas composition and substrate to flame nozzle distance on thin film deposition rate.

10:55 AM

Process Parameter Selection Rules and Direction of Maximum Stress for Laser-Deposited Coatings: *A. Kar*[1]; Franz-Josef Kahlen[1]; [1]University of Central Florida, Laser-Aided Manu., Matls. and Micro-Process. Lab., Schl. of Optics and Ctr. for Rsch. and Edu. in Optics and Lasers, Mech., Matls., and Aero. Eng. Dept., Orlando, FL 32816 USA

This paper presents a set of design rules for a laser-aided powder deposition process. A high power CO2 laser is used to melt the powder and deposit a coating on a substrate. Dimensionless numbers characterizing this powder deposition process are identified using Buckingham's P-Theorem. These dimensionless numbers are used to identify a range of values for the process parameters, such as the laser beam power, spot diameter, substrate translation speed and powder flow rate, to achieve good quality coatings for different coating materials. The yield and ultimate strengths are examined for stainless steel 304 (SS 304) coatings for three different processing conditions. These stresses are related to the dimensionless similarity parameters through the operating conditions and physical dimensions of the deposit. Experiments conducted for SS 304 shows that the yield strengths are close to the value of wrought material. Ultimate strengths are within 80% of the corresponding values for wrought SS 304. The yield strength is found maximum in a given direction and this direction is oriented very close to the direction of material solidification. A mathematical model is derived to calculate the residual stresses created during solidification, accounting for directionally preferred solidification.

11:15 AM

Time-Dependent Relationships between Hf Dopant Incorporation and HfCl4 Precursor Concentration during CVD-NiAl Diffusion Coating Growth: *G. Y. Kim*[1]; Limin He[1]; Justin D. Meyer[1]; W. Y. Lee[1]; [1]Stevens Institute of Technology, Dept. of Chem., Biochem. and Matls. Eng., Castle Pt. on Hudson, Hoboken, NJ 07030 USA

The incorporation behavior of Hf as a beneficial dopant during aluminizing of a single crystal Ni alloy has been studied using a laboratory-scale CVD reactor which can be mathematically modeled while emulating the actual manufacturing environment. The effects of proactively varying the gas phase concentration of the dopant precursor (HfCl4), as a function of aluminizing time, on the concentration and distribution of Hf in the NiAl coating matrix were examined to understand the dynamic nature of the Hf doping process. Also, the process parameters and reactor conditions which led to the formation of Hf-rich particles and experimental irreproducibility were identified. These results will be discussed in the context of increasing the scale adhesion behavior of the Hf-doped NiAl coating, through model-based process optimization, for thermal barrier coating applications.

Teaching Electronic, Magnetic and Optical Materials: A Symposium in Memory of Professor Gregory E. Stillman: Session I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Education Committee
Program Organizer: Mark A. Palmer, Virginia Commonwealth University, Richmond, VA 23284-3015 USA

Monday AM — Room: Bayou D
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Mark Palmer, Virginia Commonwealth University, Richmond, VA 23284-3015 USA

8:30 AM Opening Remarks
Mark Palmer, Session Chair;
Robert Stull, Chair EMPMD

8:40 AM
A Brief Summary of Professor Stillman's Contributions: John Parsey

8:45 AM Keynote
Bringing Electronic Materials to the Forefront of Engineering Education: *Emily L. Allen*[1]; [1]San Jose State University, Dept. of Chem. and Matls. Eng.

The design, selection, and processing of materials has always been critical to the practice of all engineering disciplines. As the study of traditional metallurgy transformed into the discipline of materials science and engineering, a marriage with solid state physics and chemistry occurred which brought new types of materials into being and allowed the unification of the discipline under the themes of structure, properties and processing. With the rise of the electronic age, the importance of electronic, photonic and magnetic materials has skyrocketed in economic importance as well as in scientific and engineering advances. MS&E curricula, and the service courses we offer to other programs, have not always kept pace with the increased importance of electronic materials. At San Jose State, we have initiated new courses and programs in electronic materials, utilizing the appropriate structure, properties, and processing approaches through laboratory experience. I will describe our electronic materials courses for Materials Engineering majors as well as our service courses for Electrical, Computer and Chemical Engineering majors. Our new degree in Microelectronics Process Engineering will be highlighted as well. I will stress the learning styles-based pedagogical methods used to enhance the learning environments of various courses.

9:30 AM Keynote
Applets and Dynamic Multimedia Objects for Teaching and Learning: *Chu R. Wie*[1]; [1]State University of New York, Buffalo, NY USA

Professor Chu is the Director for the Center for Active Learning of Microelectronics and Photonics at SUNY Buffalo, where he has developed "Educational Java Applets in Solid State Materials," he is also the Chair of the 2000 International Conference On Simulation and Multimedia in Engineering Education Conference which will bring together experts and colleagues who are interested in the fast growing area of modeling (both analytical and computational), simulation and visualization (and their comparison with experiments and observations), and simulation and multimedia-based engineering education. He will discuss his experience in developing these modules, using them in the classroom, and the activities of others as presented at the conference.

10:15 AM Break

10:30 AM
Microtechnology Education: Semiconductor and Mems Processing for Undergraduate and Graduate Students at the University of Washington: *T. G. Stoebe*[1]; T. P. Pearsall[1]; J. W. Rogers[2]; R. B. Darling[3]; M. Afromowitz[3]; P. Yager[4]; [1]University of Washington, Matls. Sci. and Eng., Seattle, WA 98195-2120 USA; [2]University of Washington, Chem. Eng. Dept., Seattle, WA 98195-2120 USA; [3]University of Washington, Elect. Eng. Dept., Seattle, WA 98195-2120 USA; [4]University of Washington, Bioeng. Dept., Seattle, WA 98195-2120 USA

An NSF-sponsored Combined Research and Curriculum Development project has allowed a team of faculty members at University of Washington to develop a series of courses and laboratories for students interested in microtechnology. A sophomore class introduces students to electronic materials properties and processing; while a junior laboratory provides an introduction to clean room processes and equipment. A senior level class on semiconductor processing includes a laboratory in which the students make a MOSFET device. Graduate level courses include an introduction and laboratory class in MEMS and a process integration class. Details on course curricula and laboratory development will be presented with lessons learned and advice for the development of similar programs. This program is supported by the National Science Foundation.

11:00 AM Invited
Introduction of Electronic Materials to Undergraduate Students in Materials Engineering: *Matthias Militzer*[1]; [1]The University of British Columbia, Dept. of Met. and Matls. Eng., Vancouver, BC V6T1Z4 Canada

Electronic materials are taught to undergraduate students in materials engineering at the University of British Columbia as a two credit elective course in the fourth year. Since the students have in general no background in quantum physics, the course is based on a predominantly descriptive approach to the subject with the objective being to develop an understanding of basic concepts of specifications and making of semiconductor materials and devices. Semiconductor materials are introduced in relation to other materials (metals, ceramics, etc.) which are stronger emphasized in the current curriculum. It is delineated why silicon is the material of choice. To motivate learning the basics of electronic materials, selected optical and electronic devices (transistors, photodetectors, etc.) are introduced throughout the course. An integral part of the course is to incorporate research aspects by a site visit to laboratory facilities (molecular beam epitaxy, clear room, etc.) and presenting selected results of research conducted by the instructor or guest lecturers (e.g. on misfit dislocations). Further, assignment work is included to perform simple calculations of materials aspects in the electronic industry.

11:30 AM Invited
Materials Science Education for the Information Age: *Sharmila M. Mukhopadhyay*[1]; [1]Wright State University, Dept. of Mech. and Matls. Eng., Dayton, OH 45435 USA

Materials science, though regarded as an interdisciplinary field, has historically evolved from metallurgy and this influence is very clear in the traditional "materials" courses offered in most places. The typical focus is normally on the interconnected chain linking processing-structure-properties-performance, each link stressing more on mechanical & structural aspects than on any other. In this presentation, some ideas of possible additions and alterations to the existing curricula will be presented. These examples range from freshmen level experiments across the entire engineering curriculum to focused graduate (or upper-level undergraduate) courses such as "Electroceramics," "Electric Materials," etc. that the author has introduced. Student responsiveness, degrees of success and special challenges will be discussed on a case by case basis.

Ultrafine Grained Materials: Fundamentals and Process Mechanisms: I

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

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

Monday AM — Room: Polk A/B
March 13, 2000 — Location: Opryland Convention Center

Session Chair: S. Lee Semiatin, Air Force Research Laboratory, Wright-Patterson Air Force Base, OH 45433-7817 USA

8:30 AM Introductory Remarks

8:35 AM Invited
Grain Boundaries of Nanophase Alloys Prepared by Mechanical Attrition: *Brent Fultz*[1]; Heather N. Frase[1]; [1]California Institute of Technology, Div. Eng. Appl. Sci., Mail 138-78, 1201 E. Calif. Blvd., Pasadena, CA 91125 USA

We report results on the structures of grain boundaries in nanocrystalline materials prepared by high energy ball milling. Results are reported on average widths of grain boundaries, as determined from a combination of Mossbauer spectrometry measurements in conjunction with grain size measurements by x-ray lineshape analysis and TEM dark field imaging. More recently we have used small angle neutron scattering to measure the densities of atoms and magnetic moments within grain boundaries of nanocrystalline fcc Ni_3Fe. Both densities are suppressed within the grain boundaries in comparison to bulk material. From the nuclear scattering measured by SANS, we find a distribution for the atom density in the grain boundary. Curiously, by quenching nanocrystalline samples to low temperature we can alter somewhat the grain boundary density. Quenching causes a change in the atomic density of the grain boundaries, indicating a more discontinuous transition in density between grain boundary and bulk material.

9:00 AM Invited
Localized CVD and the Ultrafine Grain Structure: *Harris L. Marcus*[1]; Shay Harrison[1]; Leon Shaw[1]; James E. Crocker[1]; Lianchao Sun[1]; [1]University of Connecticut, Instit. of Matls. Sci., Storrs, CT 06269-3136 USA

In high rate localized chemical vapor deposition using a laser beam to thermally decompose gas precursors the resulting grain size ranges from the near amorphous to a continuum of ultrafine grain sizes depending on processing parameters. This paper will describe the nature of the grain sizes for various ceramic materials as a function of processing and postprocessing conditions. The grain size characterizations were performed using Raman spectroscopy, NMR, TEM, X-ray and other analytic approaches. The results will be described in terms of the various characterization approaches and related to modeling of the processing variables.

9:25 AM Invited
Chemical Vapor Synthesis of Nanostructured Powders and Their Properties: *Horst W. Hahn*[1]; [1]Darmstadt University of Technology, Matls. Sci. Dept., Thin Films Div., Petersenstr. 23, Darmstadt 64287 Germany

Chemical Vapor Synthesis and Low Pressure Flame Synthesis offer exciting opportunities for the preparation of nanocrystalline ceramic powders with excellent control of the particle size, size distribution, degree of agglomeration, morphology and elemental distribution in the individual nanoparticles. In addition, porous and dense nanocrystalline coatings with functional gradients can be obtained starting from the same metalorganic precursors. The design opportunities are similar to those common in CVD processing of thin films. The synthesis method will be described including a model for the growth of primary particles and agglomerates. The resulting properties of nanocrystalline doped, two phase and coated ceramic composites will be discussed.

9:50 AM
Synthesis and Characterization of Mechanically Amorphized and Shock Densified Nanocrystalline NiTi Alloy: *Xiao Xu*[1]; Naresh N. Thadhani[1]; [1]Georgia Institute of Technology, School of Matls. Sci. and Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

In this study, mechanical amorphization (MA) and shock compaction (SC) experiments were performed on pre-alloyed Nitinol powders as well as elemental Ni and Ti powder mixture to prepare bulk nanocrystalline alloy compacts. The MA experiments were conducted using a SPEX 8000 mixer/mill. The pre-alloyed powders were ball-milled in an Ar atmosphere and the elemental powders were alloyed using Hexane. The SC experiments were performed using a single-stage gas gun at peak pressure of 5-9 GPa. The pre-alloyed compact showed crystallization to B2 phase with a nano-size microstructure. In contrast, amorphous microstructure was retained in the shocked compact of elemental mixture, which crystallized to nanocrystalline NiTi B2 phase during post-shock thermal treatment. In this paper, we will present the results of shock densification and microstructural characteristics of the nano-structure formed in the bulk NiTi compacts. Research funded by ARO under Grant No.DAAG55-97-1-0163.

10:10 AM
Synthesis and Characterization of Ultrafine Ti Powders: *S. Amarchand*[1]; T. R. Ramamohan[1]; P. Ramakrishnan[1]; [1]Indian Institute of Technology, Dept. of Metallu. Eng. and Matls. Sci., Bombay 400076 India

The present work deals with a novel chemical solution synthesis route, for the preparation of nanosize Ti powders, from Titanium dioxide. Titanium dioxide is allowed to form a complex, titanium catecholate precursor, in presence of ammonium sulphate and concentrated sulphuric acid. The complex is filtered, washed with cold isopropyl alcohol and dried. Hydrogenation of titanium catacholate is carried out for different durations at various temperatures. Titanium hydride is prepared by heating the titanium catacholate precursor at 800°C in a furnace of 8E-6 torr vacuum and dehydrogenated in vacuum to get fine Ti powders. The characteristics evaluated by XRD, SEM and TEM indicated that the powders are pure Titanium with crystallite size in the range 45-60 nm.

10:30 AM Break

10:40 AM Invited
Magnetization Reversal in Nanocomposite Exchange Spring Magnets Observed Directly with a Magneto-Optical Indicator Film: *Robert D. Shull*[1]; Alexander J. Shapiro[1]; Henrietta J. Brown[1]; Valerian I. Nikitenko[2]; Vladimir S. Gornakov[2]; J. Samuel Jiang[3]; A. Inomata[3]; C. H. Sowers[3]; Samuel D. Bader[3]; [1]NIST, Metallu. Div., 100 Bureau Dr., MS 8552, Gaithersburg, MD 20899-8552 USA; [2]Institute of Solid State Physics, Russian Acad. of Sci., Chernogolovka, Russia; [3]Argonne National Laboratory, MSD 223, 9700 S. Cass Ave., Argonne, IL 60439-4845 USA

Epitaxial Sm-Co (350 Å)/Fe(500 Å) bilayer films were grown on Cr (200 Å) buffered MgO (100) substrate by sputtering. Magnetic hysteresis loops measured in a SQUID magnetometer showed characteristic exchange-spring behavior where the reversal of the soft Fe layer is pinned at the interface by the SmCo hard layer. For the first time in such a material, the remagnetization process was observed directly using the magneto-optic indicator film (MOIF) technique. In order to

investigate the magnetic spin rotation process inside the bilayer during remagnetization, a 0.3 mm hole was made in the sample, and the magnetostatic field (H_{ms}) around the hole was visualized through the intensity changes of the double Faraday effect in a transparent indicator film with in-plane anisotropy. Black and white contrast on opposite sides of the microhole was observed, indicating the direction of magnetization in the sample around the hole. We followed the line of contrast symmetry and analyzed the spin rotation process in the soft ferromagnetic component during remagnetization. This was compared to the macroscopic magnetization as determined by a vibrating sample magnetometer (VSM). Findings include direct observation of spin rotation in opposite directions for field applications slightly off either side of the easy axis of magnetization during conventional field reversal. When the field was aligned with the easy axis, no uniform spin rotation was observed. During rotational hysteresis, a unique spin behavior was observed: spin rotation was discovered to change sign without an accompanying change in the sign of the field rotation. The reasons for these unusual remagnetization observations will be discussed.

11:05 AM

The Relationship between the Reaction Conditions and the Characteristics of the Metal-Bearing Ferrites Produced at Ambient Temperature: *Oscar Juan Perales Perez*[1]; Yoshiaki Umetsu[2]; Atsuo Kasuya[1]; Kazuyuki Tohji[3]; [1]Tohoku University, Ctr. for Interdis. Rsch., Aramaki aza Aoba, Aoba-ku, Sendai 980-8578 Japan; [2]Tohoku University, Instit. for Adv. Matls. Process., Katahira 2-1-1, Aoba-ku, Sendai 980-8577 Japan; [3]Tohoku University, Dept. of Geosci. and Tech., Aramaki aza, Aoba-ku, Sendai 980-8578 Japan

Magnetite and various metal-bearing ferrites were produced directly from aqueous solutions at 25°C by simultaneous control of the oxidizing conditions for co-existing Fe(II) ion and pH. In this presentation, the correspondence between the reaction conditions of formation of the ferrite in sulfate medium and its structural and magnetic characteristics will be discussed. The formation of a Zn-bearing ferrite was selected as a first case study. The precipitates were characterized by XRD, EPMA, FT-IR, HRTEM and magnetic hysteresis loop measurement. Furthermore, the analysis of the local structure of Fe and Zn atoms by Extended X-ray Absorption Fine Structure Spectroscopy (EXAFS) was also undertaken. It was found that the crystallinity, the de-hydration of the intermediate compound and the diminution of the sulfate content in the nano-sized ferrites (average crystallite size 10 nm) could be promoted by: (i) increasing the Fe/Zn mole ratio in the precipitates, (ii) a suitable duration of the aeration of the ferrite precursor suspension under alkaline conditions or, (iii) by aging of the precipitates at 25°C. In turn, EXAFS revealed that the ambient-temperature Zn-bearing ferrite exhibited a similar structure than the ceramic Zn ferrite produced at temperatures above 1000°C and Zn atoms were fully incorporated into the ferrite framework occupying the tetrahedral sites. The mentioned effects were attributed to the suitable progress of the oxidation-hydrolysis reactions of Fe(II) species and the loss of water from the intermediate compound during the formation of the ferrite structure incorporating Zn atoms, which also explained the observed enhancement in the saturation magnetization of the precipitates. The above results enable our proposal to be considered not only as a novel route to synthesize magnetic materials (ferrites) at ambient temperature but also a promising alternative to remove metal ions and produce re-usable precipitates in the treatment of large volumes of polluted effluents.

11:25 AM

Thermal Mechanisms of Grain Refinement in Lath Martensitic Steels: *John William Morris*[1]; Zhen Guo[1]; [1]University of California, Dept. of Matls. Sci., Berkeley, CA 94720 USA

The effective grain size of a martensitic steel with a dislocated lath substructure is the pertinent coherence length. For intergranular fracture this is the prior austenite grain size, for transgranular cleavage, the packet size in the (100) plane, and for slip-dominated processes, the packet size in (110). To achieve exceptional properties, it is important to refine the effective grain size into the submicron range, and it is desirable to do this with thermal processes that can be applied to plate product. Three mechanisms are available. In increasing order of effectiveness they are: alternate thermal cycling to refine the prior austenite grain size and the packet size, intercritical tempering to disrupt lath alignment by interposing thermally stable austenite, and rapid thermal cycles that disrupt packet alignment. The mechanisms of refinement to ultrafine grain size will be discussed. Successful examples of each grain refinement mechanism will be given.

11:45 AM

Kinetics of Glass Formation and Nanocrystallization in Al-RE-(TM) Alloys: *Robert I. Wu*[1]; Gerhard Wilde[1]; John H. Perepezko[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. and Eng., 1509 University Ave., Madison, WI 53706 USA

Aluminum-rich amorphous alloys have attracted considerable attention in recent years for their superior mechanical properties compared with conventional Al-based alloys. Often, the occurrence of a precursor crystallization reaction during heating of rapidly solidified amorphous samples leads to the formation of microstructures with finely dispersed nanocrystalline Al in an amorphous phase and offers effective dispersion strengthening. While many amorphous alloys are produced by rapid solidification processing, mechanical intermixing also provides an alternative route to synthesize metallic glasses. In the present study, characterization results on the Al-based metallic glasses by TEM and modulated-temperature calorimetry (DDSC) indicate that the glassy state has actually been attained by rapid melt-quenching as well as by deformation mixing. Annealing studies on the glassy samples produced by different processing pathways indicate a completely different nanocrystallization behavior and suggest that the quenched-in nuclei originate from the rapid melt-quenching process. Moreover, glass formation appears to be controlled by the suppression of growth of nuclei formed during rapid melt quenching for Al-based systems. With the aim of enhancing the mechanical properties of Al-based nanocrystalline materials, efforts have been focused upon increasing the particle density of nanocrystals by probing the crystallization kinetics of the primary phase. A key issue in the controlled synthesis of nanocrystalline Al microstructures is the capacity to control the nucleation density that appears to be linked to quenched-in, pre-existing clusters. Numerical modeling based upon the size distribution of the primary nanocrystals in partially crystallized $Al_{88}Y_7Fe_5$ samples has been applied to analyze the nucleation kinetics during nanocrystallization in these metallic glasses. In addition, incorporation of insoluble elements (e.g. Pb) during RSP has successfully yielded an increase in the nanocrystal particle density developed during primary crystallization. The discovery has provided new microstructural control in the study of heterogeneous nucleation kinetics in Al-based metallic glasses. The support of the ARO (DAAG55-97-1-0261) is gratefully acknowledged.

AIME/TMS KEYNOTE ADDRESS

Monday, March 13, 2000
11:30 AM - 1:00 PM
Room: Presidential Ballroom

FUTUREVIEW: A Look Ahead
Speaker: Daniel Burrus, Burrus Research Associates, Inc.

Thanks to recent innovations in science and technology we are at the gateway to a renaissance in materials technology, in terms of not only developing new applications, but also in terms of the industry itself. Although we are at a time of tremendous opportunity, we are at the same time faced with problems of equal magnitude. This presentation will provide you with valuable insight into how best to capitalize on present and future opportunities, while artfully minimizing problems along the way.

Advanced Technologies for Superalloy Affordability: Superalloy Modeling-Processing, Microstructure and Property

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: K. M. Chang, West Virginia University, Mechanical & Aerospace Engineering, Morgantown, WV 26506 USA; K. R. Bain, GE Aircraft Engines, Cincinnati, OH 45215 USA; D. Furrer, Ladish Company, Cudahy, WI 53110 USA; S. K. Srivastava, Haynes International, Kokomo, IN 46904 USA

Monday PM — Room: Canal C
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Krishna Srivastava, Haynes International, Kokomo, IN 46904 USA; Michael Fitzpatrick, Solar Turbines Inc., Adv. Tech. Matls. and Process., San Diego, CA 92186 USA

2:00 PM Invited

Application of Calphad Methods to Ni-Based Superalloys: *Nigel Saunders*[1]; [1]Thermotech Limited, Surrey Tech. Ctr., The Surrey Rsch. Park, Guildford, Surrey GU25YG UK

Thermodynamic calculations using the CALPHAD route are becoming increasingly used for practical purposes [1]. This paper will describe a number of applications of the methodology to Ni-based superalloys, with particular emphasis on issues associated with affordability. These applications will include its use in helping to define composition specifications, monitoring of sigma formation, critical temperatures and heat treatment windows during processing, more efficient and speedy alloy design and calculation of critical input parameters for use in process modeling. [1] N.Saunders and A.P.Miodownik "CALPHAD-a Comprehensive Guide" (Elsevier Science, Oxford, 1998).

2:25 PM Invited

Phase Compositions in Alloy 718: A Comparison between APT/APFIM Measurements and Thermodynamic Predictions: *M. K. Miller*[1]; S. S. Babu[1]; [1]Oak Ridge National Laboratory, Mets. and Cer. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6376 USA

In order to improve the properties of nickel-based superalloys, it is essential to determine the partitioning behavior of the solute additions within the microstructure. The techniques of atom probe field ion microscopy (APFIM) and atom probe tomography (APT) permit the solute partitioning and compositions of the coexisting phases to be determined with near atomic resolution in these complex engineering alloys. In Alloy 718, these techniques have revealed that the secondary precipitates have a dual nature and are combination of the DO_{22}-ordered $Ni_3(Nb,Ti)$ ' phase and the $L1_2$-ordered $Ni_3(Al,Ti,Nb)$ g' phase. The experimentally determined compositions of these phases will be compared to thermodynamic predictions. This research was sponsored by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. and through the SHARE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities.

2:50 PM

Capitalizing on Computational Tools in Alloy and Process Development: *Michael G. Fahrmann*[1]; G. D. Smith[1]; [1]Special Metals Corporation, 3200 Riverside Dr., Huntington, WV 25705 USA

Recently computational tools such as Thermo-Calc have been made increasingly use of to expedite alloy development and process improvement, thus cutting cost/time cycles of experimental trials. Three concrete examples are presented illustrating the capabilities of the current modeling software: (1) the development of a new weld wire for automotive exhaust systems, (2) the development of a new alloy for superheater tubing in advanced power plants, (3) the improvement of heat treating processes of high performance alloys. It is emphasized, however, that Thermo-Calc requires from the user also a sound knowledge of the physical metallurgy of the system in order to ensure effective usage of this tool.

3:10 PM

Precipitation of γ' from γ during the Weld Thermal Cycle: Recent Results of APFIM Characterization and Modeling: *S. S. Babu*[1]; S. A. David[1]; J. M. Vitek[1]; M. K. Miller[1]; [1]Oak Ridge National Laboratory, 1 Bethel Valley Rd., Oak Ridge, TN 37831 USA

Welding plays an important role in economical reuse and reclamation of used and failed nickel-base superalloy blades. It has been shown that it is possible to weld these alloys with high-energy processes such as electron beam and laser welding. The microstructure development during weld thermal cycles plays a vital role in the weldability and the service properties. In this paper, the precipitation of γ' phase during simulated weld cooling conditions was studied with transmission electron microscopy and atom probe field ion microscopy and atom probe tomography. The results indicate non-equilibrium alloying elements partitioning between γ and γ' phases. The morphology of γ' precipitates was irregular compared to that of cuboidal shape in the heat-treated condition. The experimental results will be compared with thermodynamic and kinetic calculations. This research is supported by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

3:30 PM

Solidification Diagram of Ni-Cr-Fe-Nb System: *Wei Chen*[1]; Wanhong Yang[1]; Keh-Minn Chang[1]; Sarwan K. Mannan[2]; John J. deBarbadillo[2]; [1]West Virginia University, Dept. of Mech. and Aero. Eng., P.O. Box 6106, Morgantown, WV 26506-6106 USA; [2]Special Metals Corporation, 3200 Riverside Dr., Huntington, WV 25705 USA

Macroscopic solidification segregation is one of the key issues in the production of high quality premium ingots of Ni-base superalloys, such as alloys 718, 706 and 625. These commercial alloys contain certain amounts of Nb, which is associated with the formation of eutectic phases like Laves and Ni_3Nb. Quarternary Ni-Cr-Fe-Nb alloy system is selected as the model alloy system to study the fundamentals solidification behaviour of multicomponent industry alloys. Several experimental alloys consisting of various combinations of Cr, Fe and Nb were prepared. The elemental segregation profiles and terminal solidification phases were determined by DTA and SEM-EDS analysis. Two types of eutectic products, Ni_3Nb and Laves, exist in the interdendritic region. A computational thermodynamic software, Thermo-Calc, was employed to calculate the solidification constitution diagram of Ni-Cr-Fe-Nb system. The calculated phase boundaries were verified using experimental results. A simplified thermodynamic database was developed to predict the solidification path and elemental partitioning of the model alloys. The obtained diagram can help to understand the solidification segregation of Nb-containing superalloys with complex chemical compositions.

3:50 PM Break

4:05 PM Invited

Oxygen Enhanced Crack Growth in Nickel-Based P/M Superalloys: *Robert P. Wei*[1]; [1]Lehigh University, Dept. of Mech. Eng. and Mech., 7 Asa Dr., Bethlehem, PA 18015 USA

As a part of a study to elucidate the role of niobium on crack growth in oxygen at high temperatures, three powder metallurgy (P/M) alloys, with nominal composition similar to alloy IN-100, but with 0, 2.5 and

5 wt. pct. niobium, were investigated. These alloys are γ strengthen and were designed to suppress the formation of γ' precipitates. The volume fraction of precipitates in the alloys is comparable at about 53 pct. The alloys were tested in high-purity oxygen and argon, under sustained-loading, at 873, 923 and 973 K. Companion microstructural and surface chemistry studies were also conducted. In this paper, the data on the kinetics of crack growth will be summarized and compared against those for Inconel 718. The mechanisms for crack growth and the role of niobium and of other elements will be discussed in terms of the ensemble of results.

4:30 PM

Temperature Evolution during High-Cycle Fatigue in ULTIMET® Superalloys: *L. Jiang*[1]; P. K. Liaw[1]; C. R. Brooks[1]; H. Wang[2]; D. L. Klarstrom[3]; [1]University of Tennessee, Dept. of Matls. Sci. and Eng., 427-B Dougherty Eng. Bldg., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, 1 Bethel Valley Rd., Oak Ridge, TN 37831-6064 USA; [3]Hayes International Inc., 1020 W. Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA

The infrared (IR) thermography, as a nondestructive technique, was used to investigate the fatigue damage of ULTIMET® alloy, a cobalt-based superalloy. The cumulative fatigue damaging process was characterized by the temperature changes during high-cycle fatigue. A noncontact, high-speed, and high-resolution IR thermoimaging system was capable of monitoring the temperature oscillation corresponding to the change of the mechanical state during high-cycle fatigue testing. A thermodynamic model is presented for predicting the thermal-mechanical response. The predicted temperature response was found to be in good agreement with the experimental results.

4:50 PM

The Effects of Heat Treatment on Time-Dependent Fatigue Crack Propagation in a Low Thermal Expansion Superalloy INCONEL 783: *Longzhou Ma*[1]; Keh-Minn Chang[1]; Sarwan K. Mannan[2]; [1]West Virginia University, Mech. and Aerospace Eng. Dept., P.O. Box 6106, Eng. Sci. Bldg., Morgantown, WV 26506-6106 USA; [2]Special Metals Corporation, 3200 Riverside Dr., Huntington, WV 25705 USA

Recently developed alloy 783 (nominal composition of Ni-34Co-26Fe-5.4Al-3Nb-3Cr) is precipitation strengthened by Ni_3Al-type β and NiAl-type γ phases. Due to its low coefficient of thermal expansion, high strength, and good oxidation resistance, alloy 783 is used for casings and bolting applications in gas turbines. Commercial alloy 783 plate was rolled in the laboratory to 50% reduction at 870°C. A part of the plate was direct aged and the other part was annealed at 1121°C and aged. This was followed by fatigue crack propagation of the aged materials at 300°C, 450°C, 600°C, and at room temperature. Tests were carried out at 10 Hz and 1/3 Hz using sinusoidal loading and also under a 100-second hold time using trapezoidal waveform. Interestingly, the crack growth rates of direct aged and annealed plus aged materials were not significantly different.

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Effects of Heat Treatments and Thermomechanical Processing on the Beta and Gamma Phases in Inconel 783 Alloy: *Mohindar S. Seehra*[1]; A. Manivannan[1]; Codrin Cionca[1]; L. Ma[1]; Keh-Minn Chang[1]; [1]West Virginia University, Morgantown, WV 26506 USA

Inconel 783 is a low CTE alloy in which the role of β phase is important but not well understood. Here, changes in the β and γ phases in alloy 783 upon heat treatments and thermomechanical processing (TMP) are studied using x-ray diffraction (XRD), thermogravimetric analysis (TGA) in a magnetic field and microstructural observations. TMP included: HR0 (hot rolled to ¾ inch at 2000°F); HR1 (HR0 rolled 50% at 1600°F); HR2 (HR0 rolled 50% at 1850°F) and HR3 (HR0 rolled 50% at 2100°F). Heat treatments included normal treatment (NT) and direct aging (DA). HR0DA contains only the -phase and no magnetic transition (T_c) above 25°C. Samples HR1DA, HR2DA and HR3DA have β and γ phases and two T_c's near 390°C and 490°C. For HR1NT and HR2NT, β phase is weak and T_c at 390°C is lowered to 352°C. These results will be discussed relative to thermodynamic calculations on phase formation and phase chemistry.

Aluminum Reduction Technology: Cell Development/Operation

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

Monday PM — Room: Sewanee
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Alton Tabereaux, Reynolds Metals Company, Corp. Rsrch. and Dev., Muscle Shoals, AL 35661-1625 USA

2:00 PM Invited

Development of High Amperage Prebaked Cells in China: S. Z. Feng[2]; Z. X. Leng[2]; *Yiren Gan*[1] [1]Zhengzhou Light Metal Research Institute, Shangjie, Zhengzhou China; [2]Guizhou Aluminium Plant, Guiyang, Guizhou China

Aluminium production capacity in China is over 2.5 million tonnes/annum, but the main type of pots is Soderberg pot. In order to change the backward situation, a development program for high amperage point-fed prebaked pot has been carried out since the latter part of the 1970's. A laboratory has been set up to carry out basic research work on electro-magnetic-hydrodynamics, thermal balance and other parameters in aluminium pots. It includes modeling tests, measuring methods in laboratory and industrial pots, development of mathematical modeling and calculation program. After testing and verifying the results in industrial pots, new types of 186kA and 280kA pots have been developed and tested successfully in pilot plants. Based on the test results, 2 lines of 186kA pots have been set up and are in production, another 2 lines are in construction and 2 lines of 280kA pots are planned for construction. This technology will be used in new aluminium plants in China. A 320kA testing line will be put into production soon.

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Danjiangkou-A Step Forward: *Hugh McLaughlin*[1]; Guo Xing[2]; [1]VAW Aluminium-Technologie GmbH, P.O. Box 101554, Neuss D-41415 Germany; [2]Danjiangkou Aluminum Industry Company, Hubei Province, China

VAW's former Toeging 1 smelter in Germany has been purchased by the Danjiangkou Aluminium Industry Company, Ltd. and successfully relocated to China. The startup of these facilities during 1999, supported by VAW-ATG, has upgraded and expanded the existing Soderberg plant. Today it is one centered around a 100 pot, highly efficient, end to end prebake, complete with a modern computer control system. This low cost modular design was producing aluminum only 14 months after start of construction. This 114.5 kA potline operated consistently at over 94% current efficiency with a power consumption of 13.4 kWh/kg during its former life. Its sale to China, due to power constraints in Germany, and its efficient reassembly and startup reflect favorably on the design and operational stability of this cell. The pots are equipped with center hoppers for alumina, aluminium fluoride, and crushed bath. Crust breakers and point feeders are incorporated in the hoppers. The VAW technology featuring pot design and hardware as well as the ELAS pot control system bring an era of new life to the aluminium smelting industry in central China. This paper presents some details around this project and emphasises the extreme interest within China to upgrade their aluminium smelting facilities.

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Satisfying Financial Institutions for Major Capital Projects: *Jan Heintzen*[2]; *Robert Paul Harrison*[1]; [1]Hatch, Light Metals Grp., 5 Place Ville-Marie, #200, Montreal, Quebec H3Z3R9 Canada; [2]Beddows

& Company, Aluminum Projects, 5 Place Ville-Marie, Montreal, Quebec H3B2G2 Canada

Major capital investments in the aluminum sector require significant financing, often in the hundreds of millions of dollars. Lenders are understandably conservative about transactions of this magnitude, and impose not only contractual and financial obligations but technical ones as well. A project must be technologically sound, properly staffed and managed, and environmentally acceptable-to the satisfaction of the banks-before they will disburse the required capital. With permission from the Billiton Group, two recent examples are presented to illustrate these concepts: the construction of the Hillside smelter in South Africa, and the acquisition of Worsley Alumina in Australia.

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The Effect of Anode Spike Formation on Operational Performance: *Bernd Rolofs*[1]; Neal Wai-Poi[1]; [1]Hoogovens Aluminium Huttenwerk GmbH, Postfach 101154, Voerde D46549 Germany

Since the start-up of the Voerde smelter in 1970, several upgrades and retro-fits of the existing P69 technology have been completed. These include, the introduction of bigger anodes and the implementation of Celtrol2 computer process control system. These and other modifications enabled the Voerde smelter to make significant improvements in cell performance and operations, resulting in e.g.94+% current efficiency and an average cell life of 2600 days. However, since the conversion from wet to dry scrubbing in 1992, the smelter has experienced a persistent problem with carbon dust levels. As a consequence, the anode spike frequency has increased significantly. The spiking has had a negative impact on current efficiency, typically a 1.6% loss and resulted in a greater variation in operational parameters. Therefore, several trials have been conducted to investigate the origin of the carbon dust, the impact on cell operations and the influence of operating practices on spiking.

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Aspects of Field Studies on Hall-Heroult Cells: *Nolan E. Richards*[1]; [1]Richards Consulting, 117 Kingswood Dr., Florence, AL 35630 USA

Whenever a meaningful database for the performance of a cell representative of a potline is needed, a comprehensive characterization of such a cell should be considered. Such a characterization is even more desirable when modifications to any aspect of the cell, e.g., bath chemistry, anode area, line current, thermal insulation, protocol for alumina additions are planned. Comprehensive field studies are also important in ensuring that the performance of a cell representative of a greenfield potline falls within the prescribed targets. Recommendations are given for an array of in situ field measurements and methods for conducting them. Typical results and sensitivities are presented. Examples of the data obtained from a selection of cells are shown and discussion offered of the synergism and interpretation of some results.

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Advanced Solid Bath Transport at DUBAL: *W. A.R. Al-Sayed*[1]; J. Ifju[2]; I. Del Porto[2]; M. Moni[2]; [1]Dubai Aluminium Company, P.O. Box 3627, Dubai United Arab Emirates; [2]Techmo Car Spa, Via R. Colpi, Limena(PD) 35100 Italy

Recycling the materials extracted from the cells and removed temporarily from the smelting process due to periodic operation of the prebaked anodes is compulsory in order to maintain high efficiency and performance of the primary aluminium smelting. Utilization of the solid bath requires a series of operations including its handling, processing and transport which has to be fulfilled in a safest way and lowest possible losses. A tailor-made and specially designed vehicle has been developed for the Dubai Aluminium Company Ltd. in close cooperation with the operation experts aimed at efficient handling, fast and safe transport taking into account environment protection and good working conditions for the operators. This paper describes the work done and economical effect in relation to the use of vehicles with innovative concept of design named Easy Truck.

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Design of Shunt Rheostat for Coke-Bed Preheating of Aluminium Reduction Cells: S. A. Mohamed[1]; *F. M. Ahmed*[1]; [1]Aluminium Company of Egypt, Rsrch. and Dev., Naga-Hammadi, Egypt

Coke-bed preheating is one of the most popular methods to preheat aluminium reduction cells. The preheating by this method can be achieved with partial or full line current. To achieve preheating without full line current, shunt resistance is employed. The design procedure of shunt resistance includes, selection of material, collection of preheating and cell design data, thermoelectric calculations in addition to the assembly and detail drawings. In this paper, the steps taken to design two different groups of shunt resistance fox preheating of 200kA end to end, prebaked anode cell is explained. The results of applying the different designs in preheating of aluminium cells are discussed.

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Reduction Cell Technology Development at DUBAL through 20 Years: A. J.M. Kalban[1]; *Y. A.M. Al Farsi*[1]; A. S.S. Binbrek[1]; [1]Dubai Aluminium Company Limited, P.O. Box 3627, Dubai United Arab Emirates

The Dubai Aluminium Company Limited (DUBAL) smelter commenced operations in 1979 with 3 potlines comprising 360 prebaked cells at 150 kA and producing 135,000 tonnes of aluminium annually. By 1990, a series of continuous, innovative improvements to the cell components and operational practices radically transformed the original cell technology to operate at 180 kA. An expansion of 139 cells in Potline 4 resulted in a total of 499 cells producing ~ 250,000 tonnes aluminium annually. By 1996, having developed, jointly with Comalco, and tested five prototype CD-200 reduction cells at 200 kA, DUBAL installed 240 cells in Potline 5. Total cells thereby increased to 739 and annual production to 375,000 tonnes. In 1999, another 240 CD200 cells were commissioned in Potline 6 increasing the cell population to 984 cells and annual production to 536,000 tonnes of high quality aluminium whilst generating 1400 MW power through natural gas-fueled turbines. This paper tracks 20 years of DUBAL's continuous advancement in reduction cell technology, both in terms of improvements and development.

Automotive Alloys 2000: Fundamental

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Subodh K. Das, University of Kentucky, College of Engineering, Center for Aluminum Technology, Lexington, KY 40506-0043 USA

Monday PM — Room: Knoxville A
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Subodh K. Das, University of Kentucky, College of Eng., Lexington, KY 40232 USA

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Precipitation of Iron Containing Phases in 319 Type Alloys during Solidification: *Jacob W. Zindel*[1]; Larry A. Godlewski[1]; William T. Donlon[1]; [1]Ford Motor Company, Ford Rsch. Lab., P.O. Box 2053, Dearborn, MI 48121 USA

Al-Si-Cu-Mg alloys, commonly referred to as 319, are widely used in the production of cast aluminum automobile powertrain components. Secondary alloys such as these contain iron as an impurity as a result of contamination in the recycling stream. During solidification, iron containing intermetallic compounds form which have been attributed to increasing the propensity for microporosity formation, decreasing ductility, and reducing fatigue strength of castings. In an effort to understand how to control the formation of these phases, work has been conducted to characterize their nucleation and growth. This study consisted of interrupting the solidification of 319 alloy samples with different Fe concentrations by quenching them into an iced-brine bath. Samples with 0.40, 0.65, and 0.95 wt.% Fe were quenched at

various solid fractions to determine when the various iron compounds precipitate. Typically, two Fe-containing phases are observed in these alloys, a cubic phase, alpha, with the approximate stoichiometry of Al15(Fe,Mn)3Si2 and a monoclinic phase, beta, with the approximate stoichiometry of Al5FeSi. In the 0.4 wt.% Fe alloy, alpha appeared to have precipitated after the nucleation of the Al-Si eutectic. No other iron compounds were observed in this alloy. In the 0.65 and 0.95 wt.% Fe alloys, a tetragonal phase, delta (Al4Si2Fe), was observed to precipitate near the liquidus followed by alpha and beta precipitation at a temperature above the Al-Si eutectic reaction. The temperature of the thermal arrest due to Fe compound precipitation increased with increasing Fe concentration.

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Improving Stress Corrosion Cracking in Al-Mg Alloys: *John S. Vetrano*[1]; M. J. Danielson[1]; D. R. Baer[1]; R. H. Jones[1]; [1]Pacific Northwest National Laboratory, MSIN P8-16, P.O. Box 999, Richland, WA 99352 USA

The use of Al-Mg alloys with Mg levels of greater than 3.5 wt.% for automotive structural components is hampered by their susceptibility to stress corrosion cracking (SCC) in salt water. Segregation of Mg to grain boundaries causing precipitation of the Al3Mg2 (b) phase during low-temperature thermal exposure (sensitization) has been shown to be the primary cause of SCC in these alloys. We have utilized alloying additions and tailored heat treatments to lessen the SCC susceptibility in Al alloys containing up to 7% Mg following sensitization heat treatments at 175°C. Alloying additions studied include Zn, Mn and Sc, at levels allowing the study of solid solution and precipitate effects. Both Mn additions and a slower cooling rate from the initial annealing prior to sensitization treatment reduced the SCC susceptibility as measured by the ASTM G-67 weight loss tests. Allowing the samples to cool in the furnace reduced the SCC susceptibility compared to those samples that were water quenched after annealing. Additionally, the addition of 0.09 wt.% Cu reduced the SCC susceptibility although evaluation is still in progress. The effects of alloying additions and thermal treatment on the SCC behavior of Al-7 wt.% Mg alloys are thought to be due to a change in grain boundary b phase precipitation. Work supported by the Materials Division, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC06-76RLO 1830.

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The Precipitation Sequence in 6111 Aluminum Alloy: Weifang Miao[1]; *David E. Laughlin*[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The hardening of 6111 aluminum alloy is realized through the precipitation of various metastable precipitates, therefore a more in depth understanding of the precipitation sequence is very important to achieve the full strengthening potential of the alloy. While the focus of previous investigations of the precipitation hardening behavior in Al alloy 6111 has been on the aging response, many details of the precipitation process remain unclear. In this paper, the precipitation sequence in Cu-containing Al alloy 6111 has been studied by means of differential scanning calorimetry and transmission electron microscopy. It was found that in contrast to the precipitation sequence in Al-Mg-Si alloys, which is usually GP zones—needle-like $_{\beta}$"—rod-like $_{\beta}$'—$_{\beta}$ platelets, the precipitation sequence in the 6111 alloy is believed to be: GP zones—needle-like $_{\beta}$"—lath-like Q'—Q. Financial support from Ford Motor Company is gratefully acknowledged.

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Effect of Different Natural Aging Times on the Precipitation Behaviour of AA6111: *Shahrzad Esmaeili*[1]; Warren James Poole[1]; David J. Lloyd[2]; [1]University of British Columbia, Dept. of Metals and Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T1Z4 Canada; [2]Alcan International, Kingston Rsch. and Dev. Ctr., P.O. Box 8400, Kingston, ON K7L5L9 Canada

It has been shown that natural aging after solution treatment negatively affects the subsequent artificial aging response for 6000 series aluminum alloys. In this work, the effect of different natural aging times has been examined for the automotive alloy AA6111. The level of natural aging time was varied from 0 to 4 months and was then followed by artificially aging at 180°C. The changes in microstructure were characterized using tensile tests, electrical resistivity measurements, differential scanning calorimetry (DSC) and transmission electron microscopy (TEM). Electrical resistivity measurements were found to be very sensitive to the heat treatment procedure and can be used to provide information on the formation of solute clusters and their subsequent dissolution during artificial aging. It was also found useful to conduct DSC traces after different levels of artificial aging to follow the sequence and kinetics of precipitation. The results from this work confirmed that natural aging significantly reduced the strength at short artificial aging times. As the level of natural aging was increased, this effect was increased. However, for natural aging times greater than approximately 7 days, a further increase in natural aging time had a much smaller effect. It is thought that the formation of solute clusters during natural aging reduces the solute available for precipitation at higher temperatures. It appears that dissolution of clusters in order to free up solute for precipitation is the rate controlling process during the initial stages of artificial aging.

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The High-Cycle Fatigue and Fracture Response of Al-Cu-Mg Alloy 2524: *T. S. Srivatsan*[1]; D. Kolar[1]; P. Magnusen[2]; [1]The University of Akron, Dept. of Mech. Eng., Akron, OH 44325-3903 USA; [2]Aluminum Company of America, Alcoa Tech. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA

The design of structural components for the newer generation of civilian and military aircraft demands satisfactory performance from the material under conditions of cyclic stress amplitude and strain amplitude control, and an extended service life. In this connection, a study has been made to understand the influence of test temperature on cyclic stress amplitude response characteristics and fatigue life of aluminum alloy 2524. Test specimens of the alloy were cyclically deformed over a range of stress amplitudes at both ambient and elevated temperatures. In this presentation, the stress response characteristics and resultant fatigue properties of the alloy will be highlighted in light of the competing and mutually interactive influences of cyclic stress amplitude, intrinsic microstructural effects, matrix deformation characteristics and final fracture behavior. Research supported by State of Ohio: Board of Regents (Columbus, OH, USA), and The University of Akron (Akron, OH, USA).

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Fatigue Performance of Mechanically Surface Treated Aluminum and Magnesium Alloys: *Lothar Wagner*[1]; Matthias Hilpert[1]; [1]Technical University of Brandenburg at Cottbus, Chair of Matls. Tech. and Physical Metallu., P.O. Box 101344, Cottbus D-03013 Germany

The effect of shot peening and roller-burnishing on the HCF behavior of two widely used automotive light-weight alloys (6061 Al and the magnesium alloy AZ 31) was studied. For shot peening and roller-burnishing, the main process parameters Almen intensity and rolling force, respectively were widely varied to optimize fatigue performance. The electrolytically polished condition served as a reference. Fatigue tests were performed in rotating beam loading (R=-1) in air and in 3.5% aqueous NaCl solution at frequencies of about 60 Hz. It was found that the response of the magnesium alloy to shot peening depended strongly on Almen intensity, i.e., pronounced lifetime improvements were observed only in a range of very low Almen intensity. Higher intensities led to marked overpeening effects. In contrast, the aluminum alloy showed no loss in lifetime with increasing Almen intensity. Since both alloys responded with a similar lifetime improvement to increasing rolling forces in roller-burnishing (which generally leads to a smooth surface finish) it is argued that the shot peening-induced high surface roughness and microcracks are the main reason for the marked sensitivity of the magnesium alloy to Almen intensity in shot peening.

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Comparison of Microstructures between the As Cast and Rapidly Solidified A 390 Alloy: *E. G. Baburaj*[1]; R. Fielding[1]; K. Prisbrey[2]; J. Hill[3]; R. Oswald[3]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Processes, 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]University of Idaho, Dept. of Metallu. and Mining Eng., Moscow, ID 83844-3024 USA; [3]LA Aluminum Casting, W. 1905 Miles Ave., Hyden Lake, ID 83835 USA

The hyper eutectic Al-Si base alloy A 390 (Al-17.5Si-4.5Cu-0.5Fe-0.1Mn-0.55Mg-0.1Zn) is a better alternative to the widely used A356 (Al-7Si-0.2Cu-0.2Fe-0.1Mn-0.3-Mg0.1Zn-0.2Ti) due to its superior properties which include good liquid alloy fluidity, small solidification range, high strength and rigidity, good thermal conductivity, low thermal expansion coefficient and good resistance to abrasion, wear and corrosion. However A390 finds limited use due to poor ductility of the cast structure which usually consists of a eutectic matrix with a distribution of blocky primary Si particles. Modification of the alloy, through addition of P, Na and Sr, is known to reduce the primary Si particle size and thereby improve the ductility. Application of rapid solidification processing of the alloy can further reduce the particle size of silicon. The present paper is a comparison of the effects of trace elements and cooling rate on the microstructure of the alloy.

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The Effect of Porosity on the Fatigue Properties in a Cast 319 Al Alloy: *James M. Boileau*[1]; John E. Allison[1]; [1]Ford Motor Company, Scientific Rsch. Labs., MD 3182 SRL, P.O. Box 2053, Dearborn, MI 48121-2053 USA

As the automotive industry increases its use of cast aluminum components, the need for more detailed information relating the effect of casting practice on fatigue behavior also increases. One of the key factors influencing the fatigue of cast aluminum is porosity. Therefore, a study characterizing the influence of solidification time on the microstructure and fatigue properties in a cast 319 Al alloy was conducted. Multiple fatigue tests were conducted on a cast 319 Al alloy (T6 and T7 heat-treatments) at selected stress levels so that valid statistical comparisons could be made. Extensive metallographic and fractographic characterization was performed to understand the influence of pore size and distribution on fatigue life. In general, microporosity was associated with all of the fatigue failures and was located at or near the specimen surface. Also, as solidification time increased, the average initiating pore diameter increased and the number of samples having multiple initiating sites tended to increase. Multiple initiating sites were observed in several samples and were observed to have an effect on the fatigue. Quantitative measurements of microporosity found that conventional metallographic techniques substantially underreport the maximum pore size present in the W319 alloy.

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Age Softening of AA5182 Alloy: *Jian Chen*[1]; James G. Morris[1]; [1]University of Kentucky, Chem. and Matls. Eng. Dept., Anderson Hall 177, Lexington, KY 40503 USA

The age softening phenomenon of AA5182 alloy was studied. The effect of different degrees of cold rolling, aging temperature, homogenisation temperature on the age softening behavior were measured. It was found that no observable changes in crystallographic texture, grain size, particle size and distribution and even in dislocation structure occur during the age softening process, but the electricity resistivity decreases continuously. The age softening process is a thermally activated and it accelerates with increasing aging temperature. The activation energy of the age softening process was measured. The mechanism to cause this phenomenon is discussed.

Cast Shop Technology: Modeling Solidification and Flow

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

Monday PM — Room: Mississippi
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Yogesh Sahai, Ohio State University, Dept. of Matls. Sci. and Eng., Columbus, OH 43210 USA

2:00 PM Opening Remarks

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3-D Modeling of Fluid Flow and Heat Transfer during the DC Casting Process-Influence of Flow Modeling Approach: *Gerd Ulrich Gruen*[1]; Dag Mortensen[2]; Andreas Buchholz[3]; [1]VAW Aluminium AG, Rsch. and Dev., Georg-von-Boeselager-Strasse 25, Bonn D-53117 Germany; [2]Institute for Energy Technology, Process Simulation Dept., P.O. Box 40, Kjeller N-2007 Norway; [3]Hoogovens Research & Development, Computational Fluid Dynamics, P.O. Box 10000, IJmuiden 1970 CA The Netherlands

The improvement of the DC casting of aluminum rolling and extrusion ingots is an ongoing process. Besides implementation of optimized mold systems and automated control of the main process parameters casting temperature, casting speed, metal level, and water cooling, the optimization of liquid metal distribution systems is an actual matter of research. Various numerical models of the coupled fluid flow and heat transfer phenomena dominating the DC casting process have been developed and used for understanding the complex interactions between liquid transport and temperature evolution in the solidifying ingot. Although they are based on the same set of equations some differences exist concerning the implementation of the flow phenomena. In order to evaluate the influence of the chosen fluid flow model (laminar, turbulent) on the resulting velocity and temperature distribution, three different 3-dimensional implementations used in the aluminum industry (based on the software ALSIM, FIDAP, PHOENICS/CFX) are compared by means of a set of well defined reference cases. The resulting temperatures and flow patterns of the three models are discussed in dependence of modified material properties, boundary and geometrical conditions and implemented flow model parameters. In addition, the results are qualitatively compared with temperature measurements in the liquid pool during an experimental trial program.

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Effect of Mushy Zone Mechanical Properties on the Calculated Stresses and Deformations during the Casting of an Aluminum Alloy Ingot: *Alvaro Giron*[1]; Men Glenn Chu[1]; Ho Yu[1]; [1]Alcoa Inc., Alcoa Tech. Ctr., 100 Tech. Dr., Alcoa Center, PA 15069 USA

An improved constitutive model which describes the mechanical behavior within the freezing range of an aluminum alloy has been used to estimate the stresses and deformations during the early stages of the DC casting process. Comparison is made with the stresses and deformations calculated with a similar constitutive model that extrapolates property data measured at lower temperatures.

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Determination of Boundary Conditions Using Inverse Stationary Methods: *Jean Marie Drezet*[1]; Gerd-Ulrich Gruen[2]; Marco Gremaud[3]; [1]Laboratoire de Metallurgie Physique Calcom SA, Ecole Polytechnique Federale de Lausanne, MXG Ecublens, Lausanne CH-1015 Switzerland; [2]VAW Research and Development, VAW Aluminum

AG, P.O. Box 2468, Bonn D-53014 Germany; [3]Calcom SA Parc Scientifique, Ecole Polytechnique Federale, De Lausanne PSE, Lausanne CH-1015 Switzerland

The direct chill (DC) casting process is widely used in the aluminium industry to produce rolling sheet ingots and extrusion billets. In both processes, the metal is cooled down firstly by contact with the mould (primary cooling) until an air gap forms and reduces the heat transfer to almost zero, and secondly by application of a water jet on the ingot surface (secondary cooling). In order to quantify the cooling conditions undergone by an ingot during DC casting, temperatures were measured by thermocouples immersed in the liquid pool and then entrapped by the solid. In run conditions of casting, the thermal field is stationary in a reference attached to the mould. Therefore, the measured temperature histories were converted into temperature profiles and used in the inverse method described by Rappaz et al. and adapted to stationary temperature conditions. This allowed to deduce in a first the temperature dependent thermal conductivity of the alloy, and to determine in a second step the highly non uniform distribution of the thermal heat flux extracted at the lateral surface of the ingot and corresponding to the primary and secondary cooling.

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Modeling of Solidification of Al Alloys in a Laboratory Scale DC-Simulator: *Xiaohong Yang*[1]; [1]University of Quebec at Chicoutimi, Dept. of Appl. Sci., 555 Blvd. De l'Universite, Chicoutimi, Quebec G7H2B1 Canada

A mathematical model has been developed in order to study the influence of natural convection on the solidification of aluminium alloys during direct-chill (DC) casting. The computer model simulates heat transfer, fluid flow, solid transport and microstructure evolution during solidification in a laboratory scale DC-simulator using the volume averaging method. It deals with both macroscopic and microscopic aspects of solidification. Both equiaxed structure and columnar structure, as well as the columnar-equiaxed transition, are incorporated in the model. The simulated results fitted well with the experimental data, and it was shown that natural convection has significant effects on the temperature field.

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Microporosity Prediction in Aluminum Alloy Castings: *Adrian S. Sabau*[1]; *Srinath Viswanathan*[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics, Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

As part of a program to develop models for the prediction of microporosity in cast lightweight automotive structural parts, a comprehensive model that takes into account solidification, shrinkage-driven interdendritic fluid flow, hydrogen precipitation, and porosity evolution has been developed for the prediction of microporosity fraction. Experimentally determined values of liquid metal permeability in the mushy zone were used to calculate pressure distributions in solidifying castings. The pressure distribution in a solidifying casting, coupled with a microporosity criterion involving the local hydrogen concentration and the effect of surface tension, is used to predict microporosity distributions in aluminum alloy castings. The results are compared with experimentally measured microporosity distributions in A356 aluminum alloy test castings.

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A 2D Cellular Automaton-Finite Difference (CAFD) Model of the Solidification of Aluminium-Rich Al-Cu-Si Alloys: *David John Jarvis*[1]; *John Anthony Spittle*[1]; Stephen Graham Brown[1]; [1]University of Wales, Matls. Eng. Dept., Singleton Park, Swansea, Wales SA28PP United Kingdom

A 2D cellular automaton-finite difference (CAFD) model has been developed for simulating the solidification transformations in ternary aluminium-rich Al-Cu-Si alloys. A novel feature of the model is that initial primary phase solidification occurs in a dendritic fashion, either columnar or equiaxed. A combined curvature/surface energy anisotropy routine ensures that dendrite arm growth occurs orthogonally. The model overcomes many of the limitations of traditional 1D plate numerical models by allowing solidification and solute redistribution to occur within a naturally evolving microstructure. In addition, the model allows for diffusional solute mixing in both the solid and liquid phases during the primary phase and monovariant Al-Si eutectic stage of freezing. This paper demonstrates the predicted influence of cooling rate on the proportions of primary phase, binary eutectic and ternary eutectic under non-equilibrium freezing conditions for a range of alloy compositions.

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Microstructure Prediction in A356 Alloy Castings: *Qingyou Han*[1]; *Srinath Viswanathan*[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics, Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

As part of a program to develop advanced tools for the design and optimization of structural A356 aluminum alloy castings, models have been developed and used for predicting phase fractions and microstructural length scales. The models predict phase evolution during solidification and the final length scales after solidification. These length scales are the primary dendrite size, secondary dendrite arm spacing, and cell spacing for the primary aluminum phase and the particle/rod size for the silicon phase. Mechanisms governing the growth of these phases are considered in the models. The predictions are compared with independent measurements by other researchers and with data from the literature. The results of the models are presented in the form of analytical equations for each of the length scales. The simple form of the equations allow them to be used in the post processing step of commercial solidification codes for the prediction of microstructure in shape castings.

5:05 PM

Enthalpy Variations and Latent Heat Evolution during Solidification of Lead-Tin Alloys: Sergio Fabian Gueijman[1]; Alicia Esther Ares[1]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Faculty of Sci., Azara 1552, Posadas, Misiones 3300 Argentina

In order to achieve improved predictions in a solidification process using mathematical modeling, it is necessary to couple the solidification modeling with an accurate thermodynamic model. In the present report, several thermodynamics aspects related to solidification of Lead-Tin alloys are considered. Such aspects are; the solidification path, the solid fraction vs. temperature, and the enthalpy and the latent heat evolution. A thermodynamic model including the above phenomena is developed and the values of the associated parameters are calculated, particularly the enthalpy of the solid and liquid phases of the alloys in the mushy zone, as a function of temperature and alloy concentration. From the calculated enthalpies, the latent heat released during solidification, which is a key input parameter in any solidification model, can be readily obtained. The total integrated heat released for complete solidification of Lead-Tin alloys calculated with the present model are in good agreement.

Cyclic Deformation and Fatigue of Materials; A Symposium in Honor of Professor Campbell Laird: Cyclic Deformation and Mechanism (I)

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

Monday PM — Room: Canal A
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: P. Lukas, Institute of Physics of Materials, Brno Czech Republic; Z. Wang, University of Toronto, Dept. of Met. & Matls. Sci., Toronto, Ontario, Canada

2:00 PM

Effect of Loading History on Cyclic Stress-Strain Response and Cyclic Creep: *L. Kunz*[1]; P. Lukas[1]; B. Weiss[2]; D. Melisova[2]; [1]Institute of Physics of Materials, ASCR, Brno Czech Republic; [2]University of Vienna, Instit. of Matl. Physics, Vienna, Austria

Cyclic plasticity of metals depends on loading history. The cyclic hardening/softening, steady-state behaviour, cyclic strain localisation, fatigue limit and fatigue lifetime are influenced by preceding deformation, both cyclic and monotonic. A cyclic mechanical equation of state, relating the saturation stress-amplitude with the saturation-strain amplitude, is generally not applicable. The changes of microstructure due both to the monotonic and cyclic strain are considered to be the main reason for these effects. The aim of this contribution is to summarise the effect of the loading history on the cyclic stress-strain response of Cu and Ni and to contribute to the completion of the partial understanding of the influence of the loading history represented by different ramp loading and pre-strain on the cyclic plasticity and cyclic creep. It will be shown that the cyclic stress-strain response (cyclic stress-strain curves), dislocation microstructure and the strain localisation both in single and polycrystals are strongly influenced by the start-up procedure (ramp loading) and pre-strain. Both the ramp loading (its length and the details of the load increase sequence) and the pre-strain in the first loading cycle (implicitly introduced in tests with the mean stress) affects the cyclic creep behaviour, plastic strain amplitude and the number of cycles to fracture elapsed at full stress amplitude. The saturated plastic strain amplitude has been found to be related to the cyclic creep strain in saturation. This indicates that the description of the cyclic plasticity entirely in terms of the plastic strain amplitude is insufficient and additional data on the cyclic creep (monotonic strain) are inevitable.

2:25 PM

Cyclic Plastic Deformation Behaviour of Ni Single Crystals Oriented for Single Slip as a Function of Hydrogen Content: *Thierry Magnin*[1]; C. Bosch[1]; K. Wolski[1]; [1]Ecole des Mines de Saint-Etienne, Ctr. SMS URA CNRS 1884, 158 Cours Fauriel, St Etienne, Cedex02 42023 France

Ni single crystals oriented for single slip exhibit as Cu crystals a well defined stress-strain curve at room temperature corresponding to PSB formation in a given plastic strain range. The aim of this paper is to study the influence of hydrogen on the cyclic plastic behaviour of such crystals, in comparison with pure crystals. This is of particular interest in two cases: when hydrogen enters the materials during the fatigue test (corrosion fatigue under cathodic charging) and when hydrogen-precharge has been made before cycling. It is known that hydrogen decrease the cross slip ability in fcc Ni crystals at room temperature. However, the influence of cross slip on PSB formation and evolution is known to be quite important. Thus the influence of hydrogen on the PSB formation and the corresponding cyclic plastic behaviour is analysed on pure Ni single crystals in two experimental conditions: during corrosion fatigue in a 0.5N H_2SO_4 solution at applied cathodic potential and during fatigue after hydrogen precharging. A particular attention is paid on the effect of hydrogen on cyclic softening. TEM analyses emphasize such influence. Mechanical consequences are then detailed.

2:50 PM

Mean Stress Effect on Cyclic Plastic Deformation of Industrial Pure Iron: *Hai Ni*[1]; Zhirui Wang[1]; [1]University of Toronto, Dept. of Metallu. and Matls. Sci., Toronto, Ontario M5S3E4 Canada

It has been reported recently that sagging behavior in spring steels is a direct result of asymmetrical cyclic loading. Although previous work showed that cyclic creep and cyclic softening are the two main causes for sagging in mechanical springs, the mechanisms of sagging are still not well understood. In order to characterize sag resistance more precisely and to understand sagging mechanisms, both symmetrical and asymmetrical cyclic loading tests, rather than stress relaxation and the Bauschinger effect test, were carried out systematically on model material: industrial pure iron. It was chosen based upon the need to simplify the complicated microstructural effect on dislocation motion in suspension spring steels. Mechanical results were explored in detail, and slip band evolution and dislocation structures were also studied using an in situ optical microscope and transmission electron microscopy (TEM), respectively, to gain additional information on the cyclic plastic deformation process. In addition, the effect of the pre-strains on cyclic softening and cyclic creep was evaluated from the energy point of view. Based upon the present study, the following conclusions have been drawn: first, in contrast with tensile slip bands, fatigue slip bands showed much less multi-slip even at high cyclic peak stress levels and a large amount of cumulative cyclic plastic strain; second, mean stress was found to have a significant effect on the magnitude of cumulate cyclic creep strain; third, proof was obtained demonstrating that dislocation cell size decreases with increasing cyclic stress amplitude; forth, compressive cyclic creep was found in all pre-strained samples due to Bauschinger effect and such an effect was found to disappear completely in only 25-30 cycles and, therefore, this is a strong proof that sag deformation is mainly due to cyclic creep and cyclic deformation; finally, both increasing pre-strain level and decreasing cyclic peak stress resulted in less overall recoverable energy, and this stored energy is believed to promote the material to enter its stable stage of dislocation substructure more earlier, even though it may be released very slowly late in the deformation if the load is not symmetrical.

3:15 PM

Analytical Solutions at 180° Twist Boundaries in Cubic Crystals: *Peter Neumann*[1]; [1]Max-Planck Institute for Iron Research, Max-Planck-Str. 1, Duesseldorf D-40237 Germany

Grain boundaries are efficient stress raisers in elastically anisotropic materials. Because of the complexity of the underlying equations, the elastic incompatibility stresses are usually determined numerically. In a recent paper the transformation behavior of compliances and elastic constants in anisotropic cubic crystals was treated analytically and the equations of compatibility and stress equilibrium were solved analytically for the most simple case of a coherent twin boundary. This treatment is extended in the present paper to the whole class of 180° twist boundaries. The point is stressed that no additional shear stresses on the boundary are produced by the piecewise constant incompatibility stresses. The observed slip activity and crack initiation at the surface and on planes parallel to the twin boundary is due to an additional logarithmic stress singularity at the intersecting line between the twin boundary and the specimen surface. The strength and direction of these singular stresses is determined by the tractions of the

incompatibility stresses on the surface. They are given analytically as well.

3:40 PM Break

4:05 PM

Characterization of Dislocation Glide Kinetics during Cyclic Deformation by Strain Rate Change Tests: *George C. Kaschner*[1]; Jeffery C. Gibeling[2]; [1]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA; [2]University of California, Dept. of Chem. Eng. and Matls., Davis, CA 95616 USA

A new experimental technique incorporating strain rate changes during cyclic deformation has been developed using plastic strain as the control variable. This technique was used to study the kinetics of dislocation interactions by evaluating the operational activation area and true stress as a function of cumulative plastic strain. In order to demonstrate the utility of this approach, it has been applied to three FCC metals: polycrystalline pure copper, 7075-T6 aluminum, and 304 stainless steel. These materials represent produce three distinct categories: pure FCC metals, precipitation-strengthened metals, and solution-strengthened metals. Plastic strain rate change tests were performed at plastic strain amplitudes between 0.2% and 0.6% from a base rate of $10^{-3}s^{-1}$ at room temperature. Initial values of the operational activation areas of copper evolved from approximately $5000b^2$ to $700b^2$ during cyclic loading to saturation. These values indicate a transition from forest dislocation cutting to cross-slip as the rate-controlling mechanism. Tests performed at saturation revealed a linear dependence of activation area on plastic strain amplitude. The back stress measured at saturation by extrapolating the activation area data compares favorably with the value determined from a Bauschinger analysis. The extrapolation method is an objective method of determining back stress and is especially suited for materials with asymmetrical hysteresis loops. The method is sensitive to the nuances of dislocation interactions of the various classes of materials tested; the characteristic responses of copper, 7075-T6 aluminum, and 304 stainless steel are distinct. Values of activation area correlate with the dominant rate controlling dislocation interaction mechanisms. In copper, these values correspond to a transition from cutting of forest dislocations to dislocation cross-slip. The results for 7075-T6 aluminum reveal that deformation is controlled by interactions with thermal obstacles in the form of GP zones and precipitates. Finally, this approach shows that rate-sensitive obstacles in the form of solutes control the cyclic deformation of type 304 stainless steel. This paper is based on work support by the National Science Foundation under Grant No. DMR-9208549.

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Cyclic Plasticity of Nickel at Low Plastic Strain Amplitude: *Y. Jia*[1]; D. J. Morrison[1]; J. C. Moosbrugger[1]; [1]Clarkson University, Dept. of Mech. and Aeronautical Eng., Potsdam, NY 13699-5725 USA

The cyclic plasticity of polycrystalline nickel was studied by accomplishing room temperature fully-reversed fatigue experiments at a constant plastic strain amplitude of 1.0x10-4 on nickel with a grain size of 290 μm. The cyclic plasticity behavior within a hysteresis loop was analyzed by measuring the curvature of the loop, $d^2\sigma_\Gamma/d\varepsilon_\Gamma^2$, where σ_Γ and ε_Γ are, respectively, the stress and total strain measured from the reversal point. In the classical Masing model of kinematic hardening, the curvature can be related to the frequency distribution of material volume element yield stresses. The results indicate that at low values of cumulative plastic strain, the cyclic plasticity within a loop approximates classical Masing kinematic hardening memory. However, at higher values of cumulative plastic strain, significant deviations from Masing memory are observed; and the behavior tends to reflect kinematic type III hardening as defined by Asaro[1]. The results will be discussed in terms of fundamental dislocation structures that are produced during low plastic strain amplitude cycling of pure fcc metals and the relationships between single crystal and polycrystal deformation behavior. This research was supported by the National Science Foundation under grant CMS 963407. [1]Asaro, R.J., 1975, Acta Metall., 23, 1255-1265.

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Micromechanisms of Fatigue Crack Growth in an a/b Titanium Alloy: *V. Sinha*[1]; S. Shademan[1]; A. B.O. Soboyejo[2]; W. O. Soboyejo[3]; [1]The Ohio State University, Dept. of Matls. Sci. and Eng., Columbus, OH 43210 USA; [2]The Ohio State University, Dept. of Food, Agri. Biolog. and Aero. Eng., Columbus, OH 43210 USA; [3]Princeton University, Dept. of Mech. and Aeros. Eng., Princeton Matls. Instit., Princeton, NJ 08544 USA

This paper presents the results of an experimental study of short and long fatigue crack growth in an a/b titanium alloy, Ti-6Al-4V. Differences between long fatigue crack growth rates at positive stress ratios are shown to be due largely to the effects of crack closure. Coarse Widmanstätten microstructures are also shown to exhibit higher intrinsic fatigue crack growth resistance than fine Widmanstätten or near-equiaxed a/b microstructures in the long crack regime. The improved fatigue crack growth resistance is associated with higher levels of roughness-induced crack closure and crack deflection. The fatigue fracture modes are summarized in fatigue mechanism maps before presenting multiparameter and mechanism-based fracture mechanics models for the prediction of fatigue crack growth.

Dislocations and Microscale Plasticity Modeling: Experimental Characterization of Dislocation Structures

Sponsored by: ASM International: Materials Science Critical Technology Sector, Materials Processing and Manufacturing Division, Structural Materials Division, Jt. Mechanical Behavior of Materials, Jt. Computational Materials Science & Engineering

Program Organizers: Elizabeth Holm, Sandia National Laboratories, Albuquerque, NM 87185-1411 USA; Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA; Jeffrey Rickman, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18105-3195 USA; David J. Srolovitz, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109 USA; Vaclav Vitek, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA

Monday PM — Room: Lincoln A
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Diana Farkas, Virginia Polytechnic Institute, Matls. Sci. & Eng., Blacksburg, VA 24061-0237 USA

2:00 PM Invited

Observation and Modeling of Grain Boundary Dislocation Structure and Behavior: *Douglas L. Medlin*[1]; [1]Sandia National Laboratories, Thin Film and Interface Sci. Dept., Mail Stop 9161, 7011 E. Ave., Livermore, CA 94551 USA

Incorporating the localized effects of interfaces on materials properties is a significant challenge for large-scale materials simulations. Critical to improving such models is developing an improved understanding of the behavior of dislocations incorporated at grain boundaries. In this presentation, we discuss atomistic and continuum models for interfacial dislocations in relation to experimental observations made by transmission electron microscopy, focussing initially on interfacial dislocations in the FCC Σ=3 system. Though a simple geometry, the interfacial dislocations present in this system exhibit a rich and complex set of behavior. From an analysis of these defects, we provide insight into the larger scale coupling of dislocation motion, by both glide and climb processes, with grain boundary behavior. Finally, we discuss means of extending these results to boundaries of increasing crystallographic complexity. This work is supported by the U.S. DOE under contract DE-AC04-94AL85000, and in part by the OBES-DMS.

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Micro- and Macro-Scale Subdivision of a Bicrystal Based on Experiment and Plasticity: *A. Godfrey*[1]; V. Prantil[1]; D. E. Boyce[2]; D. A. Hughes[1]; H. R. Wenk[3]; P. Dawson[2]; [1]Sandia National Laboratory, MS 9403, P.O. Box 969, Livermore, CA 94551-0969 USA; [2]Cornell University, Sibley School of Mech. and Aerospace Eng., Ithica, NY 14853 USA; [3]University of California, Dept. of Geology, Berkeley, CA 94720 USA

Bicrystals of high purity aluminum have been deformed at room temperature to strains of 15-60%. The shape change and local crystal orientations were investigated by optical and scanning electron microscopy to characterize the macroscopic deformation pattern, thereby providing data at a length scale suitable for comparison with crystal plasticity model predictions. Preliminary calculations corroborate the shape changes away from the boundary, confirming that the crystal plasticity models resolve the average slip system activity reasonably well. However heterogeneous dislocation microstructures are observed in the transmission electron microscope (TEM). These microstructures have been extensively investigated in the TEM in order to determine the pattern of microscale slip activity, using both Burger's vector analysis and Frank formula analyses of the observed rotation boundaries. The implications of the observed microscale slip pattern for crystal plasticity models are then discussed. Part of this work was supported by the Office of Basic Energy Sciences, U.S. DOE, under contract No: DE-AC04-94AL85000.

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Studies of Microscale Plasticity in Bulk Samples Using ECCI: *Benjamin Andrew Simkin*[1]; Boon Chai Ng[1]; Martin A. Crimp[1]; Thomas R. Bieler[1]; [1]Michigan State University, Dept. of Matls. Sci. and Mech., 3536 Eng., East Lansing, MI 48824 USA

Electron channeling contrast imaging (ECCI) is a scanning electron microscopy technique that allows the direct imaging of dislocations and other deformation structures in the near-surface region of bulk crystals. ECCI forms contrast from regions of crystal distortion and rotation, such as occur near dislocation cores, twins, and grain boundaries. Although the contrast obtained using ECCI is similar to that found using diffraction contrast transmission electron microscopy, as ECCI uses bulk samples, it has the advantages that dislocations can be imaged under well defined stress states and large areas can be surveyed. Direct images of the dislocation structures associated with crack propagation and arrest has been assessed by imaging at crack tips and along crack paths in single crystal NiAl. Crack deflections and arrest are found to be directly associated with variations in dislocation generation in the crack tip zone. Deformation transfer across grain boundaries has been studied in relation to sample loading and macroscopic stress states in polycrystalline γ+α_{2} Ti-Al alloys. Strain transfer across grain boundaries is often found to occur by primary twinning inducing dislocation emission in adjacent grains from the twin/grain boundary intersection. Portions of this work were supported in part by the National Science Foundation (DMR\#9302040), Office of Naval Research (N00014-94-1-204), and the MSU Research Excellence Fund.

3:20 PM Break

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Temperature-Dependent Onset of Yielding in Dislocation-Free Silicon: Evidence of a Brittle-to-Ductile Transition: Robert H. Folk[1]; *David P. Pope*[1]; M. Khantha[1]; Vaclav Vitek[1]; [1]University of Pennsylvania, Dept. of Matls. Sci., LRSM, 3231 Walnut St., Philadelphia, PA 19104 USA

An investigation of the brittle-to-ductile transition (BDT) in silicon has been conducted. Photolithography has been used to produce silicon test specimens from semiconductor grade silicon wafers that were essentially defect free. No pre-cracks or additional dislocation sources were introduced into the samples. High temperature three-point bending tests of the samples reveals a well defined transition from brittle fracture of the specimens to complete yielding near 730°C at a crosshead displacement rate of 0.01 cm/min. Limited plasticity is observed prior to the transition but is insufficient to prevent crack propagation. In addition, between 735°C and 745°C there exists a temperature interval in which only limited plasticity is observed. This suggests that yielding of the samples is not limited by the mobility of dislocations. Instead the transition may be controlled by the nucleation of a sufficient density of dislocations within the material. This idea is further supported by experiments that were conducted at temperatures below 730°C in which samples were preloaded within the linearly elastic regime and then immediately retested. This preloading effectively enhanced the yield behavior of the samples with the degree of plasticity displayed by the samples a function of the magnitude of the preload. At the highest preloads complete yielding occurred, indicating that the BDT temperature had been lowered.

4:00 PM

Analysis of Anomalous Slip in Ta Single Crystals Using Optical Atomic Force, Orientation Imaging and Transmission Electron Microscopies: *Geoffrey H. Campbell*[1]; James S. Stölken[1]; Mehdi Balooch[1]; Wayne E. King[1]; Adam J. Schwartz[1]; [1]Livermore National Laboratory, Chem. and Matls. Sci. Directorate, P.O. Box 808, L-356, 7000 E. Ave., Livermore, CA 94551 USA

High purity Ta single crystals oriented for single slip were deformed in compression at 300K and 77K. The sample deformed at 300K exhibited wavy glide whereas the sample deformed at 77K exhibited anomalous slip. Sharp load drops were recorded in the stress-strain curve of the sample tested at 77K. Previous work attributes such unloading events to either the formation of large deformation twins or to the anomalous slip process itself. Orientation imaging microscopy was applied to probe lattice rotations occurring as a result of deformation in an effort to detect the presence of large deformation twins, none were found. Optical and atomic force microscopies were applied to map the slip traces appearing on the sample surface. Atomic force microscopy revealed that the fine structure within the rather coarse anomalous slip bands is comprised of atomistic scale slip lines organized into packets. These slip packets appear to account for the "fine slip traces" often observed within anomalous slip bands. Transmission electron microscopy was used to characterize the difference in dislocation structures between the specimens deformed at 300K and 77K. The fine scale dislocation structure within the anomalous slip bands is compared to the corresponding slip trace structure examined with atomic force microscopy. This work is performed under the auspices of U.S. Department of Energy and Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

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Flow Stress Behavior of Polycrystalline OFHC Copper: *John E. Flinn*[1]; David P. Field[2]; Thomas M. Lilo[3]; Gary E. Korth[3]; Jenya Macheret[4]; [1]University of Idaho, 3450 S. 35 W., Idaho Falls, ID 83402 USA; [2]TSL Inc., 392 E. 12300 S., Ste. H, Draper, UT 84020 USA; [3]Idaho National Engineering and Environmental Laboratory, P.O. Box 1625, MSC2218, Idaho Falls, ID 83415-2218 USA; [4]U.S. Department of Energy, Idaho Operations Office, 850 Energy Dr., MS-1225, Idaho Falls, ID 83401 USA

The flow stress behavior of OFHC polycrystalline copper was evaluated from tensile specimens derived from cold roll and equal-channel angular extrusion processing. Prior to testing at room temperature, the specimens were annealed to provide grain sizes from 0.002 to 0.05 mm. The true stress-true strain behavior exhibit a parabolic pattern that correlates with four stages of hardening. In association with an increase in strain we observe: 1) dislocation source activation at the onset of plastic flow at annealing twin boundaries; 2) primary slip; 3) cross slip and forest hardening that is tied to stacking fault behavior; and 4) dynamic recovery. The tensile properties and analyses are accompanied by orientation imaging and transmission electron microscopy examinations and measurements.

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Effect of Temperature and Alloy Composition on Deformability of HfV_2+Ta C15 Laves Phase Alloy: *Won Yong Kim*[1]; David E. Luzzi[1]; David P. Pope[1]; [1]University of Pennsylvania, Matls. Sci. and Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

The occurrence of twinning in Laves phase alloys is sensitive to alloy composition and deformation conditions such as strain rate and temperature. However, it remains unclear based on the existing work in the HfV_2+Nb system whether the twinning is solely stress-driven or whether there exists a critical temperature for twinning. Results in the HfV_2 system are somewhat ambiguous due to a fairly complex phase

equilibria and the predicted presence of a low temperature phase instability. We have recently found that alloying HfV_2 with Ta yields more extensive room temperature ductility by twinning in ternary Laves phase alloys that are more easily studied. In the present paper, Ta is again chosen as the ternary alloying element in HfV_2. HfV_2+Ta Laves phase based alloys with various compositions are produced by arc-melting and float-zone-melting. X-ray diffraction is used to analyze the crystal structures and lattice parameters for each sample investigated. Compression tests are conducted at temperatures down to near liquid helium temperatures using a specially designed testing apparatus. The effect of temperature and composition on mechanical properties is investigated to gain a better understanding of the mechanisms of twinning. Alloy microstructures are characterized using conventional and high-resolution TEM. Based on the obtained results, the deformation mechanisms will be discussed with particular attention given to the nucleation of twinning in the C15 cubic Laves phase.

5:00 PM

Strengthening Effects from Nitrogen Content and Grain Size on the Flow Stress Behavior of Type 316 SS: *Nikki Y. Pearce*[1]; John E. Flinn[2]; [1]Bechtel-Bettis Inc., Naval Reactors Facility, P.O. Box 2068, Idaho Falls, ID 83403-2068 USA; [2]University of Idaho, 3450 S. 35 W., Idaho Falls, ID 83402 USA

The influence of grain size and nitrogen content on the tensile behavior of Type 316 SS were evaluated. The focus of the analysis was on the flow stress behavior in terms of parabolic hardening with true strain. The results show a bilinear pattern, defined as parabolic stages II)p and III)p with increasing square-root of strain. The effects from grain size and nitrogen content (in solid solution) are primarily associated with the onset of plastic flow with very little influence on strain hardening. Stage II)p is associated with primary and secondary slip and Stage III)p with cross slip related dislocation forest hardening.

General Abstracts: Intermetallics I

Sponsored by: TMS

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

Monday PM — Room: Bayou E
March 13, 2000 — Location: Opryland Convention Center

Session Chair: John J. Stephens, Sandia National Laboratories, Albuquerque, NM 87185-0367 USA

2:00 PM

On the Yield Strength Anomaly in CoTi and CoHf: *Ian Baker*[1]; M. Wittmann[1]; P. Bove[1]; [1]Dartmouth College, Thayer School of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

Mechanical tests have been performed on CoTi and CoHf alloys in order to determine whether the George-Baker vacancy-hardening model (Phil. Mag., 77 (1998) 737), which has been developed for FeAl, is applicable to the yield strength anomaly observed in these B2 compounds. Compression tests at elevated temperature reveal shifts in both the magnitude and temperature of the yield stress peak with changes in strain rate which are consistent with the model. However, quenching experiments, suggest that the vacancy concentrations at elevated temperature are much lower in CoTi and CoHf than in FeAl, and possibly too low for the vacancy hardening to be applicable. This work was funded by the National Science Foundation, Division of Materials Research through grant DMR-9812211 with Dartmouth College.

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Processing and Mechanical Properties of Mo-Si-B Alloys Containing High Volume Fractions of Mo_3Si and Mo_5SiB_2: *Joachim H. Schneibel*[1]; Hua-Tay Lin[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Div., P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

Mo-Si-B alloys have melting points on the order of 2000°C. Their optimum composition involves a trade-off between processing, fracture toughness, and oxidation resistance. Mo-12Si-8.5B (at. %) can be fabricated by casting and contains approximately 30 vol.% Mo_3Si, 30 vol.% Mo_5SiB_2, and 40 vol.% α-Mo. The α-Mo occurs in the form of toughening inclusions. At room temperature this alloy exhibits a 3-point flexure strength of 500 MPa and a fracture toughness on the order of 10 MPa $m^{1/2}$. Its tensile creep properties, determined at 1200°C in argon, will be discussed. Mo-Si-B alloys with a continuous α-Mo matrix were fabricated by powder-metallurgical techniques and their mechanical properties and oxidation resistance will be compared to the corresponding properties for cast alloys. This research was sponsored by the Fossil Energy Advanced Research and Technology Development (AR&TD) Materials Program, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

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Phase Formation and Interdiffusion in Silicides and Aluminides of Mo With and Without Re Additions: *Edward J. Ciecko*[1]; Mysore A. Dayananda[1]; [1]Purdue University, School of Matls. Eng., 1289 MSEE Bldg., West Lafayette, IN 47907 USA

Diffusion structures developed by interdiffusion between disks of Mo-aluminides in contact with disks of Mo, Si and Mo-silicides with and without additions of Re are investigated at selected temperatures between 700-1200°C. Phase formation and the effects of Al and Re on the interdiffusion of the components in the silicides of Mo will be discussed. Diffusion studies include an examination of the structures involving binary and ternary aluminides and silicides of the system Mo-Si-Re-Al. Diffusion structures will be described with the aid of diffusion paths. Interdiffusion coefficients for the silicides and aluminides observed in the diffusion structures will also be presented.

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Composition and Mechanical Properties of Mo_3Si: *Isai Rosales*[1]; Joachim H. Schneibel[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics, P.O. Box 2008, Oak Ridge, TN 37831-6115 USA

The A15 phase Mo_3Si is an important constituent of a new class of silicides based on Mo-Si-B (D. M. Berczik, U.S. Patent No. 5,595,616, 1997; C. A. Nunes, R. Sakidja, and J. H. Perepezko, in "Structural Intermetallics 1997," TMS). In this research we will show that, contrary to published results, single-phase Mo_3Si is slightly off-stoichiometric. Its room temperature fracture toughness is on the order of 3.5 MPa $m^{1/2}$. When the deformation rate is high (10^{-3} s^{-1}), compressive deformation at 1400°C in argon often results in fracture associated with a decrease in load. At low deformation rates (10^{-5} s^{-1}) and 1400°C, fairly constant loads are reached after a few percent deformation. Partial substitution of Mo by Cr increases the high temperature strength. The high temperature strength of Mo_3Si will be compared to that of other silicides and A15 phases. This research was sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract number DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation. I. Rosales acknowledges partial support from DGEP-FQ, Universidad Nacional Autonoma de Mexico.

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Influence of Aging Treatment on the Microstructure and Hardness of a Cr-Cr_2Ta Composite: *Peter K. Liaw*[1]; *Yuehui He*[1]; *C. R. Brooks*[1]; *C. T. Liu*[2]; [1]The University of Tennessee, Matls. Sci. and Eng. Dept., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals and Ceramics Div., Oak Ridge, TN 37831-6115 USA

A Cr-Cr_2Ta composite, as a new candidate of ultra-high temperature materials, is being studied. The excellent mechanical properties and good oxidation resistance of the composite at temperatures above

1000°C are provided by a solid-solution Cr(Ta) matrix reinforced with Cr_2Ta laves-phase plates. In this study, the effects of the as-cast process and aging treatment on the microstructure and hardness of a Cr-9.8 at.%Ta composite were examined. The hardness of the composite in the as-cast condition at room temperature decreased with increasing the aging temperature from 1000°C to 1300°C, which was due to the reduction of the solid-solution extent of the Cr(Ta) matrix. The morphology and formation mechanism of the microstructures of the composite in as-cast and aging-treatment states were explored. The orientation relationships between the Cr_2Ta phase and Cr(Ta) matrix were analyzed using transmission electron microscopy (TEM). This research is sponsored by the Fossil Energy Advanced Research and Technology Development (AR & TD) Materials Program under subcontract 11X-SP173V to the University of Tennessee with Dr. R. R. Judkins as the contract monitor.

4:00 PM

Effect of Temperature and Alloy Composition on Deformability of HfV_2+Ta C15 Laves Phase Alloy: *Won Yong Kim*[1]; David E. Luzzi[1]; David P. Pope[1]; [1]University of Pennsylvania, Matls. Sci. and Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

The occurrence of twinning in Laves phase alloys is sensitive to alloy composition and deformation conditions such as strain rate and temperature. However, it remains unclear based on the existing work in the HfV_2+Nb system whether the twinning is solely stress-driven or whether there exists a critical temperature for twinning. Results in the HfV_2 system are somewhat ambiguous due to a fairly complex phase equilibria and the predicted presence of a low temperature phase instability. We have recently found that alloying $HfV_2$with Ta yields more extensive room temperature ductility by twinning in ternary Laves phase alloys that are more easily studied. In the present paper, Ta is again chosen as the ternary alloying element in HfV_2.

4:20 PM

Room Temperature Deformation of Cr-Based Laves Phase Alloys: *Won Yong Kim*[1]; David E. Luzzi[1]; [1]University of Pennsylvania, Matls. Sci. and Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

Cr-based Laves phase alloys with AB_2 stoichiometry are promising material for high temperature structural applications due to their high melting temperatures, low density and potential resistance to corrosion and oxidation. Binary Laves phase alloys however, are extremely brittle at room temperature due to their complex crystal structure. Ternary alloying has been shown to yield room temperature deformability by twinning in HfV_2 with Nb or Ta. In the present study, refractory metals such as Hf, Ti, Ta and Nb are used as ternary alloying elements in various Cr-based Laves phases through consideration of chemistry, atomic size and electron-atom ratio. Laves phase alloys with a wide range of compositions are produced by arc-melting. Crystal structure, microstructure and lattice parameter analyses are carried out using optical microscopy and x-ray diffraction. We find that room temperature ductility is more common than currently understood with extensive room temperature ductility possible in $ZrCr_2$- and $NbCr_2$-based cubic Laves phase alloys after ternary alloying.

General Non-Ferrous Pyrometallurgy: Thermochemical Modeling and Physical Properties

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee

Program Organizers: Robert L. Stephens, Cominco Research, Trail, British Columbia V1R 4S4 Canada; Pekka Taskinen, Outokumpu Research Oy, Pori FIN-28101 Finland

Monday PM — Room: Bayou B
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Pekka A. Taskinen, Outokumpu Research Oy, Pori FIN-28101 Finland

2:00 PM

Heat and Material Balance Model of the Kokkola Zinc Roaster Based on the HSC Chemistry 4.0 Software: *Antti Roine*[1]; Jens Nyberg[2]; [1]Outokumpu Research Oy, P.O. Box 60, Kuparitie 5, Pori 28101 Finland; [2]Outokumpu Zinc Oy, P.O. Box 26, Kokkola 67101 Finland

Sulfidic zinc concentrates are usually roasted in a fluidized bed furnace into sulfur-free oxide form. Metallic zinc is produced from this oxidic calcine using hydrometallurgical leaching, solution purification, and electrowinning stages. The roasting step is needed because zinc oxides are easier to dissolve than zinc sulfides using traditional leaching methods. The Kokkola Zinc Smelter uses various types of zinc concentrates and secondary materials as the feed mixture. Because the composition and particle size distribution of the feed mixture varies even on a daily basis, it is difficult to find the optimum process parameters for the process. Certain conditions may even cause difficult accretion problems or increase the amount of calcine in the process gas stream to an unacceptable level. A thermochemical heat and material balance model was developed in order to estimate the effects of different process parameters (temperature, process air coefficient, concentrate feed, oxygen enrichment, water feed, etc) on the heat balance, roasting temperature, and calcine composition. This model was constructed using the heat balance model of the HSC Chemistry 4.0 software. The prevailing chemical compounds in the concentrate and calcine were verified with the mineralogical analyses and equilibrium calculations. The roasting process seems to operate near the chemical equilibrium conditions, if the behaviour of the main components are considered. This paper describes briefly the basic principles of the calculation model and outlines the results with several diagrams and discussion. The calculated results were in reasonable agreement with the recent process air oxygen enrichment and water feed test campaigns carried out at Kokkola in 1999.

2:25 PM

Evaluation of New Process Parameters and Operating Conditions in Non-Ferrous Pyrometallurgy Through Thermochemical Modeling: *Florian Kongoli*[1]; Ian McBow[1]; [1]Flogen Technologies, P.O. Box 49529, CP Du-Musee, Montreal, Quebec H3T2A5 Canada

New operating conditions and process parameters are often necessary in today's industrial practice of non-ferrous smelting and converting in order to accommodate various new chemical compositions of mineral charges and environmental requirements or to conceive new and more efficient industrial technologies. An effective way to evaluate and predict these important parameters and conditions is thermochemical modeling, which decreases considerably the cost of pure experimental evaluation. This work presents thermochemical modeling of various phases in non-ferrous smelting and converting and their effective use in the evaluation of several factors such as the liquidus temperatures of slag, matte/slag/metal distributions, activities of crucial components etc. Several practical easy-to-use diagrams are

presented along with examples of cost decrease. Future developments are also discussed.

2:50 PM
The Correlation of Thermodynamic Properties of Multicomponent Liquid Alloy Systems: *Dajian Wang*[1]; *Tae-Kyu Kim*[1]; [1]University of British Columbia, Dept. of Metals and Matls. Eng., AMPEL 2355 E. Mall, Vancouver, BC V6T1Z4 Canada

To obtain the thermodynamic properties of a multicomponent liquid alloy system over the whole range of concentration at an arbitrary temperature, the free volume cell theory has been applied to derive the formula of activity coefficients of liquid solutions based on the infinite dilute solutions at some temperature. The linear relationship of logarithm of activity coefficients of binary dilute solutions versus reciprocal absolute temperature was verified in good agreement with the experimental values from the literature. The Monte-Carlo algorithm was used to calculate the nonlinear end value equations of binary systems, and then the free volume parameters obtained were directly correlated with the activity coefficients of liquid binary, ternary, quaternary, and quinary systems at an arbitrary temperature. The three dimensional diagrams of a-x-T of some binary systems representing the temperature and concentration dependence of activities were plotted. The calculated results regarding non-ferrous alloy systems have been statistically examined with the experimental values from the literature. Hence, this simple approach could be useful for analyzing thermodynamic properties of liquid alloy systems.

3:15 PM Break

3:30 PM
Viscosities of Some Binary and Ternary Slags in the System CaO-FeO-SiO2 with CaF2 Additions: *Fatemeh Shahbazian*[1]; Sichen Du[1]; Seshadri Seetharaman[1]; [1]Royal Institute of Technology, Metallu., Brinellv. 23, Stockholm 100 44 Sweden

Among the physical properties of slags, viscosity is very important in understanding the mass transfer phenomena in metallurgical processes. The viscosities of fayalitic slags are of great interest in the non-ferrous metal industry, with special reference to copper production. In an attempt to systematize the knowledge on slag viscosities, the present work was carried out. The viscosities of CaO-SiO_2, "FeO"-SiO_2 and CaO-"FeO"-SiO_2 slags with CaF_2 additions were carried out in the temperature range 1438-1762 K. The rotating cylinder method was used in the viscosity measurements. Crucibles and spindles made out of pure iron were employed. The slags were prefused before the measurements. In order to take into account the vaporization reactions involving CaF_2, pre- and post measurement samples of the slags were chemically analysed and the compositions were ascertained. The results show that in the case of the CaO-SiO_2 system, CaF_2 additions tend to decrease the viscosity, in accordance with the expectations. In the case of the "FeO"-SiO_2 system, at higher "FeO"/SiO_2 ratios in the slags, a reverse trend was observed. The measurements in the CaO-"FeO"-SiO_2-CaF_2 system indicate that the trends in this case are somewhat similar to the CaO-SiO_2 system. The results are discussed on the basis of a viscosity model for silicate melts developed in the present laboratory as well as the structural considerations of silicates.

3:55 PM
The Viscosity of Liquid Lead-Tin Alloys and its Temperature Dependence: *José Deodoro Trani Capocchi*[1]; [1]University of São Paulo, Dept. of Metallu. and Matls. Eng., Polytechnic School, Av. Prof. Mello Moraes, 2463, São Paulo, SP 05508-900 Brazil

Viscosity of liquid Pb-Sn alloys has been measured by the rotational viscometer method in the temperature range from 50 K above their respective liquidus temperature to 823 K. The results are accurate to within ±1.0%. The variation of viscosity with temperature followed the Arrhenius-type equation $m=m_0.exp^{(Q/RT)}$. The composition dependence of the isothermal viscosity and the activation energy for viscous flow has not shown anomalous changes at the eutectic composition of the Pb-Sn system.

4:20 PM
Removal of Copper from Slags with the Aid of Reducing and Sulfiding Gas Mixtures: *Andrey V. Tarasov*[1]; S. D. Klushin[1]; [1]Gintsvetmet Institute, State Rsch. Instit. of Non-Ferrous Metals, 13 Acad. Korolyov St., Moscow 129515 Russia

The most common method for decreasing the copper content of slags from autogenous smelting of copper sulfide concentrates is treatment of molten slag in an electric furnace, among other things, by adding solid reducing and sulfiding agent, i.e., pyrite. One of the drawbacks of this technique is production of final slag with a copper content of about 0,5%. An innovative method was tested based on treatment of molten slag from autogenous smelting process by a gaseous mixture of sulfur dioxide and methane at a ratio of CH_4:SO_2=1:(3,0-4,7) in the presence of oxygen. Cleaning of slag to remove copper was carried out at a pilot plant in the second zone of the furnace where slag was maintained within a range of 1250-1350°C. Reactions in the SO_2-O_2-CH_4 system proceed at a high rate at temperatures within 1100-1300°C to form reducing-sulfiding agents: H_2S, S, COS, CO, CO_2, H_2, H_2O. The required amount of the gas mixture is in the order of 100-150 Nm^3 per 1 tonne of slag. A decrease in the ferric iron concentration (Fe^{3+}) from 10-15% to 2-3% results in lower copper solubility in slag, while agitation of the melt with gas leads to an accelerated separation of the sulfide phase formed and the slag. The copper content of the final slag is about 0,12-0,14%. The yield of bottom matte is 7-10% of the weight of the slag processed and its copper content is about 12-15%. The matte with a copper content of 50-55% sent to converting is a mixture of bottom phases from two zones of the smelting furnace, i.e., from the settling zone and from the slag decopperizing zone. Off-gases from both zones are mixed inside the furnace and sent to a sulfuric acid plant. This process has been proposed for commercial introduction at two smelters in Russia and the CIS Republics.

High-Temperature Superconductors: Synthesis & Processing

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

Program Organizers: U. Balu Balachandran, Argonne National Laboratory, Argonne, IL 60439 USA; Pradeep Haldar, Intermagnetics General Corporation, Latham, NY 12110-0461 USA; Chandra Pande, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5000 USA

Monday PM — Room: Canal D
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Donglu Shi, University of Cincinnati, 498 Rhodes Hall, Cincinnati, OH 45221 USA

2:00 PM Invited
Dopants in Processing of High Temperature Superconductors: *Sharmila Mitra Mukhopadhyay*[1]; [1]Wright State University, Mech. & Matls. Eng., Colonel Glenn Hwy., Dayton, OH 45432 USA

The possibilities of "Dopant enhanced texturing (DET)" of perovskite superconductors will be discussed. It is seen that an anionic dopant such as Br on the surface of superconducting Y-Ba-Cu-O (YBCO) results in enhancement of sintering and grain growth rates without any adverse compositional or crystallographic changes. This effect will be compared with that of a cationic dopant (Yb) that lowers the eutectic temperature of the 123 structure. Our studies indicate that these dopants may cause activated sintering and/or enhanced liquid phase sintering. Possible atomistic mechanisms underlying all these effects will be discussed. Since large domain sizes and preferred orientations are key to electrical and levitation properties of these materials, the possibility of adding a dopant that increases grain growth rate without increasing processing temperature may be very useful in controlling texture. Future studies that can make this a technological reality will be discussed.

2:30 PM Invited

Dislocations and Plastic Flow in Flux-Line Lattices of High-Temperature Superconductors: *David O. Welch*[1]; [1]Brookhaven National Laboratory, Dept. of Appl. Sci., Bldg. 480, P.O. Box 5000, Upton, NY 11973-5000 USA

Since the classic work of E. J. Kramer[1] and many others during the 1970's, it is known that for conventional superconductors, under conditions which depend on the magnetic flux density and the density, topology, and spatial distribution of pinning centers, the onset of flux flow, and thus the critical current density (J_c), results from the plastic deformation of the flux line lattice. Recently by means of advanced methods of electron microscopy, Tonomura et al.[2] have imaged a variety of types of plastic flow in FLLs within $Bi_2Sr_2CaCu_2O_{8+\delta}$ crystals. Previously, within the context of a simple analytical theory based on the theory of dislocations and plastic flow in metals and alloys, I have shown how stress states arise in FLLs and discussed the role of temperature, current density, and pinning center strength, topology, and distribution in determining whether plastic shear of the FLL or depinning limits the critical current density.[3] In this paper, I will discuss how the character of dislocations in the FLL and its consequent shear strength depend on the anisotropic, layered nature of cuprate superconductors and will describe a theory of flux creep, flux flow, and the EJ characteristics of HTS materials based on dislocations and the plasticity of FLLs in cuprates. [1.]E. J. Kramer, J. Appl. Phys. 49, 1360 (1973). [2.]A. Tonomura et al., Nature 397, 308 (1999). [3.]D. O. Welch, IEEE Trans. Appl. Supercond. 3, 1476 (1993). This research was supported by the US. Department of energy, Division of Materials Sciences, Office of Basic Energy Sciences under Contract No. DE-AC02-98CH10886.

3:00 PM Invited

Congruent Growth Mechanism of Peritectic Phase in Undercooled Nd-Based Superconducting Oxides: *Kosuke Nagashio*[1]; Yuzuru Takamura[2]; Kazuhiko Kuribayashi[2]; Yuh Shiohara[3]; [1]The University of Tokyo, Matls. Sci. Dept., 7-3-1 Hongo, Bunkyo-Ku, Tokyo 113-8656 Japan; [2]The Institute of Space and Astronautical Science, 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan; [3]Superconductivity Research Laboratory, ISTEC, 1-10-13 Shinonome, Koto-ku, Tokyo 135 Japan

Congruent growth mechanism of a peritectic phase, $NdBa2_{Cu2}O7_{-x}$ (Nd123), from undercooled melt below a peritectic temperature (T_p) is discussed from the viewpoint of a phase selection theory based on a competitive growth in the peritectic system for free growth condition. The seeding experiment to clarify the temperature dependence of phase appearance shows that the volume fraction of Nd123 drastically increased when the seeding was carried out at the temperature below T_p. The growth velocity of the Nd123 phase measured concurrently well agreed with the theoretical calculation based on the phase selection theory where it was assumed that melting point of the Nd123 phase is T_p and the Nd123 phase grow congruently from undercooled melt below T_p. These results suggest that the driving force of the Nd123 phase is activated below T_p, not the hypothetical congruent melting point.

3:30 PM Invited

Reactive Field Assisted Sintering of BSCCO-Ag_2O Ceramic Produced from Freeze Dried Precursor Powders: *Petre Badica*[1]; George Aldica[1]; Joanna R. Groza[2]; M. C. Bunescu[3]; S. Mandache[1]; [1]National Institute for Materials Physics, POB MG-7, Bucharest, Magurele R-76900 Romania; [2]University of California-Davis, Chem. Eng. and Mat. Sci. Depts., One Shields Ave., Davis, CA 95616 USA; [3]Metav-S.A., Pob 18/3, Bucharest, Romania

Nitrate freeze dried powder (Bi:Pb:Sr:Ca:Cu=1.7:0.3:2.0:2.5:3.5) was decomposed at 750°C for 60 min. in air and mixed with commercial Ag_2O (1.2% wt.). Two types of samples with and without silver oxide addition were processed by Reactive-Field-Assisted-Sintering Technique (RFAST) at 750°C for 4 minutes under a pressure of 17.5 MPa in vacuum. Final heat treatments were applied at 800-870°C for 70-200 h. RFAST-pellets produced from precursor powder with and without Ag_2O have shown different behavior. Reaction of 2212-phase formation during RFAST was sluggish in Ag_2O-BSCCO pellets. No 2223-phase could be observed after RFAST processing, but this phase formed during final heat treatment. The role of Ag_2O during BSCCO-RFAST is discussed.

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

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

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

Monday PM — Room: Johnson A/B
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Robert W. Cahn, FRS, Cambridge University, Matls. Sci. and Metallu., Cambridge CB2 3QZ England; Peter W. Voorhees, Northwestern University, Matls. Sci. and Eng. Dept., Evanston, IL USA

2:00 PM Opening Remarks

2:05 PM Keynote

Theoretical Characterization of Alloy Structures at Microscopic and Mesoscopic Scales: *Armen G. Khachaturyan*[1]; [1]Rutgers University, Cer. and Matls. Eng. Dept., Piscataway, NJ 08855-0909 USA

Development of the theory of alloy structure from atomic to nanoscale level is discussed. It is shown that the structure on the atomic scale is described by the occupation probability function, which can be formulated in terms of Static Concentration Waves. Amplitudes of the waves are the long-range order parameters, the wave vectors are the superlattice reciprocal lattice vectors. This approach is especially effective in the mean-field approximation. However, it was also successfully used if the correlation effects are taken into account. The Concentration Wave method describes both the atomic scale and the nanoscale (if the variation of lro parameters becomes considerable). With this feature, this method provides a bridge between the scales. It is shown how this method can be used to make an accurate transition to the Phase Field theory of evolution of the microstructure on the mesoscopic scale. The Phase Field theory of the mesoscopic microstructure evolution in coherent structurally inhomogeneous alloys with multivariant domains of ordered intermetallics is discussed. The evolution is driven by the minimization of transformation strain. This theory is based on the Phase Field micromechanics incorporated in the alloy thermodynamics. This approach allows one to realistically simulate the mesoscopic microstructure evolution for a wide spectrum of materials (metal and ceramics) with different types of transformations (diffusional and displacive). Input data required to carry out the computer simulation are the crystal lattice parameters, compositions and elastic moduli of phases, and the interfacial energy.

3:00 PM Invited

Application of Khachaturyan's Elasticity Theory to Modeling Coherent Phase Transformations and Structural Defects: *Long-Qing Chen*[1]; [1]Penn State University, Matl. Sci. and Eng. Dept., 118 Steidle Bldg., University Park, PA 16802 USA

One of many Khachaturyan's major contributions to materials science is his elasticity theory for any arbitrary coherent microstructures. It has become a routine tool for many experimentalists to predict the equilibrium shapes and habit planes of precipitates using independent parameters such as lattice parameters and surface energy, and to interpret their experimental observations. In the last few years, it

has also become an integral part of phase-field modeling of coherent phase transformations in solids. In this talk, a brief review will be given for the application of Khachaturyan's elasticity theory to phase-field simulation of microstructure evolution during coherent phase transformations. In particular, the effect of an applied external load (strain/stress) or internal stress fields created by local defects such as dislocations and point defects will be discussed. A simple method for introducing the local fields created by structural defects into the phase-field model of coherent phase transformations will be presented. This method will be applied to the diffusional nucleation and growth of coherent precipitates at dislocations and the effect of local tetragonal distortions on a cubic to tetragonal transformation. It will be shown that structural defects can have a significant influence on coherent phase transformations. For example, nucleation at a dislocation may become barrierless as a result of coupling between the coherency strain and the local fields by the dislocations.

3:30 PM Break

3:45 PM Invited

Gamma' Precipitate Shape Evolution and Splitting in Ni-Based Alloys: *Alan J. Ardell*[1]; Dongman Kim[1]; [1]University of California-Los Angeles, Matls. Sci. and Eng., 6531-G BH, Los Angeles, CA 90024-1595 USA

It is well known that the shapes of gamma'-type (Ni3X, X = Al, Si, Ti and combinations thereof) precipitates in aged Ni-X alloys change from spheres to cuboids as their size increases. When the volume fraction is small, generally less than 0.03 to 0.04, the shapes continue to evolve as the particles grow by diffusion-controlled coarsening. They become nearly perfect cubes, which evolve into a concave cuboidal shape at yet larger sizes. In alloys cooled directly from the solution-treatment temperature to the aging temperature the concave cuboidal particles undergo splitting transitions into either pairs of parallel plates or groups of 8 cuboids. These types of transitions are consistent with predictions of computer simulation experiments of Khachaturyan and his co-workers. In alloys that are quenched and aged, which is the typical heat-treatment procedure in our laboratory, the evolution of shapes into concave cuboids is observed, but splitting of these particles (Ni3Al and Ni3Ti, for example) has not been seen. Experiments are in progress to determine whether there is something inherent in the heat-treatment procedure that stimulates the splitting transition. We will also present the results of aging experiments on Ni-Ga and Ni-Ge alloys containing small volume fractions of Ni3Ga and Ni3Ge, in which the shape transitions have not been previously characterized. This research is supported by the National Science Foundation.

4:15 PM Invited

Coherency Strain in Elastically Inhomogeneous Systems: *Jong K. Lee*[1]; [1]Michigan Technological University, Dept. of Metallu. and Matls. Eng., 1400 Townsend Dr., Houghton, MI 49931 USA

Professor Khachaturyan advanced a multitude of seminal works on strain effects in crystalline solids, especially in elastically homogeneous systems. His theories shed light, among others, on the role of both elastic anisotropy and tetragonal misfit strain, which led us to clear understanding of particle splitting in nickel-based superalloys and strain-induced ordering behavior in interstitial alloys. This presentation is to complement his works with recent findings on coherency strain effects in elastically inhomogeneous systems, i.e., in solids where the competing phases have different elastic constants. The origin for particle splitting has been usually understood in terms of anisotropic strain energy. This view is incomplete in light of recent theoretical works demonstrating that a particle in an isotropic system splits into multiple particlets. Splitting phenomena can be classified into a commensurate and an incommensurate elastic instability. In the former, a non-equilibrium elastic state may cause particle splitting provided that relaxation of the excess strain energy can overwhelm the accompanying interfacial energy increase. The second case of incommensurate instability arises when the anisotropy ratios of the precipitate and matrix phase have opposite signs, for example, when the elastically soft direction of the matrix is parallel to the hard direction of the particle phase. One of the driving forces for ordering is long known to be the relaxation of strain energy due to difference in atomic size. Coherency-induced ordering in a substitutional alloy is examined in a simple model with a two-dimensional square lattice. When both elastic and chemical interactions join together for ordering, the order-disorder transition temperature is raised to a value greater than the sum of the two individual cases. Other aspects of elastic inhomogeneity effects, including a case with tetragonal misfit strains, will be discussed.

4:45 PM Invited

Generalized Phase Field Modeling of Microstructural Evolution in Solids: Incorporation of Rigid-Body Motion of Grains and Mobility/Energy Anisotropy of Grain Boundaries: *Yunzhi Wang*[1]; Andrei Kazaryan[2]; Chen Shen[1]; Bruce R. Patton[2]; [1]The Ohio State University, Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]The Ohio State University, Phys. Dept., 174 W. 18th Ave., Columbus, OH 43210 USA

The phase field approach to microstructural evolution during solid state phase transformations, originally popularized by Armen Khachaturyan and his coworkers, has received an increasing amount of attention in the last few years. Several reasons may have contributed to its popularity. For example, it is able to simulate realistic microstructures and their evolution during both diffusional and diffusionless transformations under conditions of high volume fraction, elastic interactions arising from both internal lattice misfit and external fields, and multiple ordered domains and orientation variants. However, modeling microstructural evolution in solids containing grain boundaries and free surfaces demands that the model accounts for the movement of individual particle as a rigid body. Further more, the mobility and energy of grain boundaries are usually anisotropic rather than isotropic in may solids. In the current formulation of the phase field model for solid state microstructural evolutions, both rigid-body motion and crystal anisotropy are not considered. In this presentation, recent efforts in extending the phase field method for simulating phenomena such as sintering and anisotropic grain growth will be discussed in the context of incorporating rigid-body motion of grains and mobility/energy anisotropy of grain boundaries. These advances allow different phenomena occurring during sintering and similar processes to be simulated in a single, consistent methodology. A number of problems which require consideration of rigid-body motion and anisotropic grain boundary properties will be addressed.

International Symposium on Global Innovations in Materials Processing and Manufacturing: Tutorials and Overviews of Solid Free Form Fabrication Techniques

Sponsored by: Materials Processing and Manufacturing Division

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

Monday PM — Room: Canal E
March 13, 2000 — Location: Opryland Convention Center

Session Chair: John E. Smugeresky, Sandia National Laboratory, Dept. 8724, Livermore, CA 94551 USA

2:00 PM
Overview of Direct Fabrication Processes and Materials-An Industrial User Perspective: *W. R. Schmidt*[1]; D. L. Anton[1]; A. F. Giamei[1]; [1]United Technologies Research Center, East Hartford, CT 06108 USA

Computer-based design and visualization technologies are critical for achieving significant reductions in time-to-market for new products and production parts. In parallel with this revolution has been the development of additive manufacturing technologies, which can rapidly and directly render these complex computer-based ideas into physical articles via a Solid Freeform Fabrication (SFF) process. SFF opportunities exist when conventional manufacturing techniques cannot easily be used, due either to high processing costs, one-of-a-kind or low volume production levels, geometry limitations, long lead times, and/or specialty materials. This presentation will provide an overview of several commercial and developmental techniques for directly fabricating parts and tools for aerospace or commercial use, including a description of planned usage in the manufacturing environment. Examples of current capabilities, limitations, and challenges, along with a wish list of process and product attributes will be discussed.

2:30 PM
Tutorial on Stereolithography: *Paul F. Jacobs*[1]; [1]Express Tool, Warwick, RI USA

This presentation will explain the fundamental principles underlying the stereolithography process. Information will be provided regarding the nature of the UV laser radiation/photopolymer reaction, the log linear "working curve" relationship between cure depth and laser exposure, the basic parabolic cylinder cured volume element, and the functional dependence of the cured line-width. The evolution of build styles, from "Tri-Hatch" to "WEAVE" to "STAR-WEAVE" to ACES and QuickCast will also be covered. Specific applications involving the generation of QuickCast Patterns for the investment casting of functional metal components, the use of ACES models for photo-stress analysis, and the Direct AIM process for prototype plastic injection molding will also be described.

3:00 PM
Laminated Object Manufacturing (LOM): *Mukesh K. Agarwala*[1]; [1]University of Dayton, Rsch. Instit., 300 College Park, Dayton, OH 45469-0172 USA

Laminated Object Manufacturing (LOM) is a Rapid Prototyping process that uses feed materials in the form of sheets. The LOM process fabricates a part by laminating sheets of material and cutting out cross-sections on each sheet layer. Commercially the LOM process offers adhesive backed paper as the material of choice to its users. The fabricated LOM parts from paper appear like wood models and are used in a variety of applications. This tutorial will discuss the basics of the LOM process and review the state-of-the-art in LOM technology. Due to sheet based processing, the LOM process has also been developed to use green ceramic and metal sheet materials to fabricate ceramic and metal parts. Developments in the LOM processing of ceramic, metal, and fiber reinforced composites will also be reviewed.

3:30 PM Break

3:40 PM
Fused Deposition Modeling (FDM): *Mukesh K. Agarwala*[1]; [1]University of Dayton, Rsch. Instit., 300 College Park, Dayton, OH 45469-0172 USA

Fused Deposition Modeling (FDM) is a Rapid Prototyping process that extrudes and deposits a thermoplastic material in selective areas as defined by the CAD data. The extruded material solidifies as it is deposited onto a substrate or onto previously built layers. Commercially the process offers a variety of thermoplastic materials for fabrication of prototypes. The FDM thermoplastic parts can be used in a variety of applications, including certain functional applications. This tutorial will describe the FDM process and review the state-of-the-art in the FDM technology. Several developments are currently underway to apply the FDM technology to the processing of ceramics and metals. Ceramic and metal processing by FDM process employs a powder injection molding type of green feedstock to fabricate green ceramic and metal components. This presentation will review the developments taking place in the FDM processing of metals and ceramics.

4:10 PM
Tutorial on Selective Laser Sintering: *Christian Nelson*[1]; David Bourell[2]; [1]DTM Corporation, 1611 Headway Circle, Bldg. 2, Austin, TX 78754 USA; [2]University of Texas, Mech. Eng., MC C2200, Austin, TX 78712-1063 USA

The SLS® Selective Laser Sintering process is a rapid prototyping (RP) process which uses a laser to selectively fuse powdered materials together, creating objects layer by layer. The SLS process has the unique advantage of processing a broad range of materials in a single RP platform. Plastic, ceramic, and metal material systems are commercially available, and the development of new materials is ongoing at a number of universities in around the world. This discussion will touch on the technical issues of processing the different types of material systems, but will focus on the processing of two types of metal material systems.

4:40 PM
Tutorial on Selective Area Laser Deposition (SALD) of Ceramics: *Harris Marcus*[1]; [1]University of Connecticut, Instit. of Matls. Sci., Storrs, CT 06269-3136 USA

The processing necessary to do solid freeform fabricating (SFF) from vapor phase precursors will be described. Included will be a description of the processing instrumentation used. The nature of the gas/laser beam interactions and specific systems studied will be described. This will include both selective area laser deposition (SALD) and SALD vapor infiltration (SALDVI) SFF approaches. SALD is direct writing from the localized decomposition from the gas phase and SALDVI involves infiltration into powder layers to create the SFF shapes. The range of processing parameters and their influence on the character of the deposits will be described.

5:10 PM
Overview of Rapid Solidification Phenomena in Direct Metal Deposited Materials: *Dan J. Thoma*[1]; *John E. Smugeresky*[2]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [2]Sandia National Laboratory, Livermore, CA 94551 USA

Direct metal deposition processes display microstructures that are consistent with rapid solidification phenomena. For example, a continuous liquid/solid interface is maintained while achieving constant cooling rates that can be varied between 10 to 10^5 K s^{-1} and solidification growth rates (that scale with the beam velocity) ranging up to 10^{-2} m s^{-1}. Moreover, microsegregation profiles do not necessarily adhere to interfacial equilibrium conditions at the solid/liquid interface. The rapid solidification results from approximately 100 micron layers being epitaxially deposited onto a cooled, prior substrate. Traditional rapid solidification techniques rely on at least one thin dimension from which heat can be rapidly removed. Direct metal deposition techniques also rely on a thin deposition layer, but permits bulk rapidly solidified samples to be produced. The characterization of the rapid solidification behavior and the implications related to the properties of fabricated materials will be discussed.

International Symposium on Iridium: Structures and Properties

Sponsored by: Structural Materials Division, Refractory Metals Committee

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

Monday PM — Room: Jackson A/B
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Hiroshi Harada, National Research Institute for Metals, Ibaraki 305-0047 Japan; Joseph G. Biondo, Engelhard-CLAL LP, Cateret, NJ 07008 USA

2:00 PM Invited

On Specific Features in Mechanical Behaviour of Iridium: *P. Panfilov*[1]; [1]Urals State University, Lab. of Strength, Ekaterinburg 62001 Russia

There is only one face centred cubic metal whose mechanical behaviour may be called “puzzling.” Many tests have shown that high purity iridium could be both a highly plastic and a brittle substance simultaneously. This anomaly is the subject of this paper. Octahedral slip is the main deformation mechanism in iridium, while the contributions of mechanical twinning and other mechanisms are insufficient for deformation over wide temperature ranges. Brittle transcrystalline fracture or cleavage is the inherent tensile fracture mode observed in the necked region of polycrystalline samples or after considerable elongation of single crystals. Analysis of the evolution of transcrystalline cracks has shown that the inclination to cleavage is a property of bulk crystals. Recrystallization leads to catastrophic decrease in the plasticity of iridium: grain boundaries are dangerous places in polycrystalline metal. However, the intercrystalline brittleness of iridium does not depend on contaminants only.

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Types and Fundamental Properties of States Occupied by 57 Co Atomic Probes in the Grain-Boundary Core and Adjacent Regions of the Lattice in Polycrystalline Iridium: V. N. Kaigorodov[1]; *S. M. Klotsman*[1]; A. V. Ermakov[2]; V. K. Rudenko[2]; A. N. Timofeev[1]; N. I. Timofeev[2]; [1]Institute of Metal Physics, Urals Div. of Russian Acad. of Sci., S. Kovalevskaya 18, Ekaterinburg 620219 Russia; [2]Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 610014 Russia

Iridium polycrystals with the grain size of the order of $2\text{-}10^{-4}$ m were produced by deformation of a pure (4N) single crystal of iridium at 800°C and its recrystallization in a ultra-high vacuum furnace at 1273 K for 2 hours. Radioactive ^{57}Co tracers diffused into an iridium polycrystal at 500K under conditions of preferable intercrystallite diffusion (at temperatures below 800K). The examination by the method of nuclear gamma resonance spectroscopy showed that only two types of states in the intercrystallite diffusion (ICD) zone, one in the core of the intercrystallite conjugation regions and the other in adjacent regions of the lattice, were populated even at extremely low temperatures of the diffusion introduction of the ^{57}Co atomic probe in poly-Ir. These states differed by isomer shifts, local Debye temperatures, and Curie temperatures of magnetic ordering of residual magnetic active impurities.

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An Atomistic Study of the Dislocation Core Structure and Interatomic Interaction Potentials in Iridium and Rhodium: *Ludmila Yakovenkova*[1]; Bella Greenberg[1]; Yurii Shamanaev[1]; Lidia Karkina[1]; [1]Institute of Metal Physics, Ural Div. of Russian Acad. of Sci., Ekaterinburg 620219 Russia

The interatomic potential for Ir, which describes with high accuracy phonon spectra, lattice properties, including energy characteristics of point defects was obtained. The analogous potential is plotted for Rh. The structure and energy stacking faults as well as dislocation core was calculated by computer simulation method. Simulation of screw and edge dislocations showed that these split effectively on the {111} planes. Comparative analysis of the structure and energy of {110} planar defects in Ir, Rh and Cu was carried out. It is shown that, contrary to the case of copper, the stacking-fault-energy surfaces of iridium and rhodium exhibit minima for the shear by the vector a/4<110>. Analysis of the gain in energy upon dissociation of the a/2<110> dislocations shows that in iridium and rhodium this dislocation dissociates into two a/4<110> partials on the {110} planes.

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Investigations of Microstructure-Property-Relationships in Iridium: *J. Merker*[1]; D. F. Lupton[1]; H. -J. Ullrich[2]; M. Schlaubitz[3]; B. Fischer[4]; [1]W. C. Heraeus GmbH & Company KG, Matls. Tech. Div., Dev. Dept., Heraeusstrasse 12-14, Hanau D-63450 Germany; [2]Technical University Dresden; [3]Infineon Technologies Dresden GmbH & Company OHG; [4]University of Applied Science, Jena

Because of its chemical stability and the high melting point of 2454°C iridium is especially suitable for applications at the highest temperatures. In spite of its face-centered cubic lattice structure iridium, even of very high purity, tends to brittle behaviour both in hot and cold forming. It shows unusually strong work hardening in forming. Therefore, the formability of iridium presents a technical problem in the manufacturing of semi- and finished products. The formability and the strength properties are essentially influenced by the generation of lattice distortions during forming. In the same way, the influence of trace impurities on mechanical properties is known. Investigations by means of the back reflection X-ray divergent beam technique and the Kossel technique stimulated by an electron beam were accomplished at the TU Dresden as a contribution to explaining the causes of brittleness. The Kossel technique stimulated by an electron beam shows a high sensitivity with regard to mechanically influenced surfaces. It was possible to eliminate the influence of the surface work-hardened layer on the generation of Kossel interferences by using high energy and high intensity synchrotron radiation. The influence of selected manufacturing conditions (rolling and annealing conditions) on the real structure of compact samples was shown by using the back reflection X-ray divergent beam technique. High dislocation densities, local mechanical distortions in the lattice and small angle grain boundaries were indicated. These are the main reasons for the difficult processing of iridium. By means of microhardness measurement under testing force, both the elastic and plastic components of the impression and deformation processes were determined. Significant differences in the hardness were indicated dependent on the different manufacturing conditions. These results correspond with the investigated dislocation densities.

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The Distribution of Ir in Ni-Based Single-Crystal Superalloys: *H. Murakami*[1]; T. Yokokawa[1]; Y. Koizumi[1]; *H. Harada*[1]; [1]National

Research Institute for Metals, High Temp. Matls. 21 Project, 1-2-1 Sengen, Tsukuba Science, Ibaraki 305-0047 Japan

The addition of platinum group metals (PGMs) to Ni-based superalloys are being considered for the next generation superalloys with higher temperature capabilities. Among PGMs, Ir is of particular interest because of its high melting point and high corrosion resistance. In addition, since Ir and Ni both have fcc structure and they form a complete solid solution system, a high amount of Ir is expected to be alloyed to Ni-based superalloys without destroying phase stability. This study is aimed at investigating the microscopic distribution of Ir in Ni-based single-crystal superalloys. Atom-probe field ion microscopy (APFIM) revealed that Ir atoms have a small preference to be located in the gamma phase and to substitute for the Al site in the gamma prime precipitates, which is in agreement with numerical estimations by cluster variation method (CVM) and Monte Carlo simulations (MCS). In this presentation, the distribution of other PGMs in Ni-based alloys is briefly discussed in comparison to Ir-containing alloys.

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The Effect of Deformation and Annealing Conditions on Recrystallization of Deformed Single Crystals and Polycrystals of Iridium: N. I. Timofeev[1]; A. V. Ermakov[1]; *S. M. Klotsman*[2]; V. G. Pushin[2]; V. N. Kaigorodov[1]; L. I. Yurchenko[2]; V. K. Rudenko[1]; A. N. Timofeev[2]; P. E. Panfilov[3]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia; [2]Institute of Metal Physics, Urals Div. of Russian Acad. of Sci., S. Kovalevskaya 18, Ekaterinburg 620219 Russia; [3]Urals State University, Lab. of Strength, Ekaterinburg 62001 Russia

The recrystallization threshold of pure (4N) single crystals of iridium was analyzed as a function of the deformation (atmosphere and temperature) and annealing (air or ultra-high vacuum) conditions. Similar to other FCC metals, primary recrystallization of iridium was observed at 0.3-0.4 T_{melt} (T_{melt}-Ir melting point) after it was deformed in a "jacket" at room temperature and annealed under a ultra-high vacuum.

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Microscopic Theory of Defect Structure and Peculiar Mechanical Properties of Iridium: *Yu. N. Gornostyrev*[1]; O. N. Mriasov[2]; A. J. Freeman[2]; N. I. Medvedeva[3]; M. I. Katsnelson[1]; A. V. Trefilov[4]; [1]Russian Academy of Sciences, Instit. of Met. Physics, S. Kovalevskaya 18, Ekaterinburg 620219 Russia; [2]Northwestern University, Dept. of Phys. and Astronomy, Evanston, IL 60208-3112 USA; [3]Institute of Solid State Chemistry, Pervomaiskaya str. 91, Ekaterinburg 620219 Russia; [4]Kurchatov Institute, Russian Sci. Ctr., Kurchatov Sq., Moscow 123182 Russia

The brittle failure after a long stage of plastic deformation is the most surprising feature in mechanical properties of Ir and its analog, Rh, separating them from other FCC metals. On the base of ab initio total energy calculations, the peculiarities of the structure and energetic characteristics of defects in Ir (vacancies, dislocation, stacking faults) and cleavage decohesion process are investigated. We have carried out corresponding calculation also for Au which is an example of an FCC metal with a typically ductile behavior. Comparing the dislocation and vacancy formation characteristics (scalable by such factors as melting temperature, or FCC metals). A distinguishing feature of Ir is a relatively small decohesion energy (in comparison with elastic moduli) which leads to its brittle fracture, according to standard Rice-Thomson criteria of brittle-ductile behavior. The reason of this is a peculiar character of chemical bonding which is very strong for small displacements due to ion overlap butdiminishes rapidly with increased distance.

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About Martensitic Transformation in ZrIr Compound: *Yu V. Kudryavtsev*[1]; E. L. Semenova[2]; [1]Institute of Metal Physics, Acad. of Sci. of Ukraine, Vernadsky str., Kiev 252142 Ukraine; [2]I.N. Frantsevich Institute for Problems of Material Science, Acad. of Sci. of Ukraine, Krzhyzhanovsky str., Kiev 252180 Ukraine

There is some controversy as to crystal structure and nature of martensitic transformation (MT) in the equiatomic compound ZrIr. The latter is known to relate to the class of compounds formed as a result of interaction between transition metals of IV and VIII groups and to reveal transformation in solid similar to that in TiNi. Crystal structure of high-temperature modification of ZrIr was previously reported to be cubic B2-type (at 1050°C) and of low-temperature modification-a monoclinic B19-type (at room temperature). The transformation in ZrIr was recorded by means of X-ray, microstructure, electric resistivity, and differential thermal analysis methods. Shape memory effect (SME) for ZrIr was predicted by analogy with ZrRh and TiNi. An attempt to reveal SME in ZrIr was previously undertaken but failed. The martensitic phase of ZrIr was identified as a stacking variant of CrB-type structure. The current report presents new finding of our study carried out with aim to make clear when the SME would be displayed in ZrIr. The heating and cooling of ZrIr specimen were conducted in two regimes. By the first, speed of cooling and heating was quite low, about 10°/min. The five cycles (heating up to temperature above A_f, ~940°C, loading, cooling under the load down to temperature below M_f, 710°C, unloading, heating again up above A_f) were fulfilled and in each of them the specimen was bent on cooling substantially but did not reveal the shape restoration on heating. The lack of SME in ZrIr as compared to ZrRh (in spite of isostructurality in both origin and martensitic phases, similarity of shape of both electric resistivity and thermal curves) was suggested to be due to increased diffusion processes at higher temperatures. To inhibit diffusion processes in ZrIr, a specimen heating rate of about 100°/scc was used. In this case, the shape restoration in ZrIr of about 71-75% was observed. The possibility of the restoration degree increase in ZrIr as well as the divergence of our data on crystal structure of the ZrIr martensitic phase is discussed.

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Characteristic Features of High Temperature Properties of Iridium Among FCC Metals: M. I. Katnelson[1]; A. V. Trefilov[2]; K. Yu. Khoromov[2]; A. Yu. Rumyantsev[2]; *Yu. N. Gornostyrev*[1]; [1]Institute of Metal Physics, Urals Brnch. Russian Acad. of Sci., Ekaterinburg 620219 Russia; [2]Kurchatov Institute Russian Science Center, Moscow 123182 Russia

The dispersion in the entire Brillouin zone and the temperature dependence (right up to the melting temperature) of the anharmonic frequency shift and phonon damping in a number of FCC metals is investigated on the basis of microscopic calculations. It is found that the anharmonic effects depend sharply on the wave vector in the directions of the G-X, X-W, and G -L and, in contrast to BCC metals, the magnitude of the effects is not due to the softness of the initial phonon spectrum. It is shown that the relative frequency shifts and the phonon damping near melting do not exceed 10-20%. The relative role of various anharmonic processes is examined, and the relation between the results obtained and existing experimental data is discussed.

Kleppa Symposium on High Temperature Thermochemistry of Materials: Session II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Extraction & Processing Division, Thermodynamics & Phase Equilibria Committee, Process Fundamentals Committee

Program Organizers: Ray Y. Lin, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA; Y. Austin Chang, University of Wisconsin, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Dr. Susan Meschel, The University of Chicago, Chicago, IL 60637 USA; Ramana Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487 USA

Monday PM — Room: Lincoln E
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Y. Austin Chang, University of Wisconsin, Dept. of Matls. Sci. & Eng., Madison, WI 53706-1595 USA; Robert J. Gottschall, US Department of Energy, Germantown, MD 20874-1207 USA

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Enthalpies of Formation of NiAl and Compounds in the Al-Ni-Y System: *Philip Nash*[1]; Ole Kleppa[2]; [1]IIT, Mmae Dept., 10 W. 32nd St., Chicago, IL 60616 USA; [2]University of Chicago, James Frank Instit., S. Ellis Ave., Chicago, IL USA

The enthalpy of formation of NiAl as a function of composition has been determined by high temperature reaction calorimetry. The value for the Ni0.5A10.5 composition is -61.0±1.1 kJ/mole. The enthalpies of formation of the ternary compounds All6Ni3Y, Al4NiY, and A12NiY and of the binary compound A12Y containing nickel have also been measured. The enthalpy values measured are compared to previously published results where available.

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High Temperature Calorimetry in Solid-Gas Reactions: Application to the Intermetallic Hydride Compounds: *P. Dantzer*[1]; [1]Universite de Paris-Sud, Cnrs Umr 8647, Bat 415, Orsay, Cedex 91405 France

In order to study the intermetallic compounds-H_2 systems, one has to consider the problems created by the presence of a powdered activated sample and the manifestation of hysteresis during the formation-decomposition of the hydrides. Due to the low thermal conductivity of the powder, strong non isothermal behaviors can lead to strong temperature gradients within the sample, which in turn may induce metallic diffusion and disproportionation of the compound. Hysteresis implies a non reversible character of the solid phase transformation and the possibility of describing different thermodynamic paths during the scan of the hysteresis loops. Solutions to these problems have been brought by a sharp control of the thermodynamic variables during the phase transformation. Thus to maintain quasi-isothermal conditions, a reliable control of the temperature inside the sample is insured by optimizing the hydrogen gas flow rate. The measurements have been carried out with an automatic apparatus, consisting of a heat flow calorimeter coupled with high precision volumetric devices. It provides accurate characterizations of the thermodynamic properties as well as informations of the dynamic aspects of the hydride phase growth, over a wide range of pressures 0-4 MPa and temperatures 250-800 K. The ensemble constitutes a closed system in which high purity hydrogen gas is permanently transferred between hydrides reservoirs and reactors with high thermal transfer capacity. The excellent stability of the signal of the calorimeter, ±4nV over a long period of time (>10 days), allows to measure directly the heat evolved during the scan of an hysteresis loop, with an average accuracy of 1%. Kinetic of the phase transformation is based on the analysis of the measured heat flux, where the true rate law at the sample level is obtained by deconvoluting the measured signal. It is shown that only overall informations can be expected; the results of the numerical treatment raise the problem of the location of the heater used for calibration of the calorimeter. Investigations have been carried out with the ZrNi-H_2 system.

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Calorimetric Study on Hydration of CaO-Based Oxides: *Yasutaka Iguchi*[1]; Takayuki Narushima[2]; Chihiro Izumi[3]; [1]Tohoku University, New Industry Creation Hatchery Ctr., Arakaki Aza Aoba, Aoba-ku, Sendai 980-8579 Japan; [2]Tohoku University, Dept. of Metallu., Aramaki Aza Aoba, Aoba-ku, Sendai 980-8579 Japan; [3]Tohoku University, Grad. Schl., Aramaki Aza Aoba, Aoba-ku, Sendai 980-8579 Japan

Weathering disintegration of steelmaking slag is caused by volume expansion due to the hydration of free-CaO and CaO-based materials in the slag. In the present work, heat of hydration of CaO solid solutions (CaO-MnO and CaO-FeO systems) and CaO compound oxides were measured by a solution calorimetric technique and the relationship between the heat of hydration and expansion due to hydration was clarified for these materials. The heat of hydration was evaluated with enthalpy change of a reaction with distilled water at 327K, and the expansion was determined in an argon-water vapor atmosphere at 353K. Effects of MnO and FeO contents in the solid solution on heat of hydration or expansion were shown. The difference of expansion rates for CaO compound oxides was discussed from the point of view of hydration products.

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The Standard Enthalpies of Formation of the Compounds of Early Transition Metals with Late Transition Metals and with Noble Metals as Determined by Kleppa and Co-Workers at the University of Chicago-A Review: *Qiti Guo*[1]; [1]University of Chicago, James Franck Instit., 5640 S. Ellis Ave., Chicago, IL USA

Since the early 1980's, experimental studies of the standard enthalpies of formation of the binary intermetallic compounds of early transition metals with late transition metals and with noble metals have been a major long-term research project in this laboratory. Tabulated in this review are 265 enthalpy of formation values for 252 such compounds, all determined in this laboratory during the last two decades. The calorimetric methods used in these investigations have included solution calorimetry, solute-solvent drop calorimetry, and direct synthesis calorimetry. Among these methods the direct synthesis approach has been the most frequently used technique. In this review our results will be compared with values published by other laboratories and with values predicted by the Miedema semi-empirical model. However, the emphasis will be placed on the systematic variation of the standard enthalpy of formation for some characteristic alloy families from group to group in the periodic table. A few examples will be presented to show the correlation between the enthalpy of formation and the pertinent atomic number in the binary alloy families.

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Analysis of the Enthalpy of Mixing Data of Binary and Ternary (Rare Earth (Nb, La, Y, Yb)-Alkali Metal) Halides Systems: *Marcelle Gaune Escard*[1]; Michael Hoch[2]; [1]Universite de Provence, IustiI-Cnrs Umr 139, Technopole de Chateau Gombert, 5 rue Enrico Fermi, Marseille, Cedex 13 13453 France; [2]University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA

We analyzed the enthalpy of mixing data of several liquid Rare Earth–Alkali metal halides using the Hoch-Arpshofen solution model. We investigated the $NdCl_3$-MCl (M is Na, K, Rb, Cs,), $NdBr_3$-MB_r (M is Li, Na, K, Rb, Cs), $LaBr_3$-MBr (M is Li, Na, K, Rb, Cs), (LaF_3, YF_3, YbF_3)-MF (M is Li, Na, K, Rb, Cs)" binary systems, and the ternary $NdCl_3$-LiCl-KCl, LaF_3-NaF-LiF systems in the binary systems the larger the M+ radius, the larger the maximum (in absolute terms) of the

enthalpy of mixing. Larger the anion radius L- the smaller the maximum of the enthalpy of mixing. In the binary systems NdL3-ML the maximum of the enthalpy of mixing is given by the equation: Hm max (in kK) =-(2.392± 0.165)*r_M+ +(0.742±0.112) *r_{L-}. In the binary systems LaL_3-ML the maximum of the enthalpy of minimum is given by the equation: Hm max (in kK) +- (2.029±0.179)*r_M+ +(0.642±0.133)*r_{L-}. In the ternary system $NdCl_3$-LiCl-KCl along the line $NdCl_3$-(0.58 LiCl + 0.42 KCl) the enthalpy of mixing equals that of NdCl3-NaCl, because the weighted average radius of (0.58 Li+ + 0.42 K^+) equals that of Na^+. In the ternary system LaF_3-NaF-LiF the enthalpy of mixing could be computed from the binary systems. The composition of the maximum of the enthalpy of mixing depends on the radius of the rare earth and anion. If the rare earth radius is large compared to the anion radius (La vs F) the maximum is at x_{MF}+ 0.5. if the rare earth radius is smaller, (Dy vs Cl) the maximum shifts to ^xM_V + 0.69.

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Enthalpies of Mixing in Fe-C-Si Melts: *Mark E. Schlesinger*[1]; Qinfang Xiang[1]; [1]University of Missouri-Rolla, Dept. of Metallu. Eng., 1870 Miner Circle, Rolla, MO 65409-0340 USA

The accuracy of mass and energy balance calculations in iron- and steelmaking is highly dependent on the enthalpy of mixing of molten iron-based solutions, in particular Fe-C-Si melts. These enthalpies of mixing are difficult to determine experimentally, and as a result are calculated using a variety of models derived from Gibbs energy of mixing data. A comparison of calculated enthalpies of mixing in the Fe-C-Si ternary system is made, using several different modeling approaches. Featured modeling methods include the regular-solution model of Schmid and Wagner's model for multicomponent dilute solutions. The modeling results are compared with experimental data reported by Vitusevich et al.

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Thermochemical Study on Stability of Zro² Fine Particles: *T. Mitsuhahsi*[1]; [1]National Institute for Research in Inorganic Materials, 1-1 Namiki, Tsukuba, Ibaraki 305-0044 Japan

It is well known that high-temperature tetragonal Zro² with fine particle sizes occurs even at room temperature. Though this problem has been discussed from both views of kinetics and thermodynamics, a clear explanation has not been made. A high-temperature solution calorimetry may give the clear answer under consideration of heat capacity. In the present work, some of tetragonal and monoclinic fine particles were prepared. A Calvet twin micro-calorimeter was used to measure heats of solution of samples into a solvent. The sample powders were dropped from 298K into $3Na_20_4MoO_3$ solvent at 970K. Heat capacities were measured from 298K to 900K by DSC method. Measurements of heats of solution gave the values of 9.5 to 18 kJ/mol for t-Zro² and 21 to 29 kJ/mol for mZro², respectively. By considering surface areas and strain in particles, enthalpy of transition and surface energy difference between t- and m-Zro² were estimated to -12.5kJ/mol and 0.1J/m² at 298K, respectively. It is clearly concluded under consideration of heat capacity data that t-Zro² fine particles are thermodynamically unstable below 700K, compared with m-Zro² fine particles.

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Spinal Oxide Calorimetry: *M. Wakihara*

Abstract text is unavailable

Magnesium Technology 2000: Thermal Reduction and Environmental Issues

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

Monday PM — Room: Bayou C
March 13, 2000 — Location: Opryland Convention Center

Session Chair: John N. Hryn, Argonne National Laboratory, Argonne, IL 60439 USA

2:00 PM

Fundamentals of Serpentine Leaching in Hydrochloric Acid Media: *J. E. Dutrizac*[1]; T. T. Chen[1]; C. W. White[2]; [1]CANMET, 55 Booth St., Ottawa, Ontario K1A0G1 Canada; [2]Metallurgie Magnola Inc., 620 Rene Levesque W., Montreal, Quebec H3B1N7 Canada

In the Magnola process, magnesium metal is produced by the electrolysis of anhydrous MgCl2 derived from the leaching of serpentine, which is the major constituent (>90%) of asbestos tailings. Serpentine reacts rapidly in concentrated HCl media, and the leaching rate is essentially independent of the rotation speed of disks of the massive mineral. The implication is that the rate is chemically controlled, and this conclusion is supported by the moderately high temperature dependence and the approx. 40 kJ/mol apparent activation energy. At 95°C, the leaching rate of the asbestos tailings increases as the 0.5 power of the HCl concentration, but is independent of the concentrations of FeCl3 or FeCl2, for concentrations as high as 1.0 M of either salt. The accumulation of the MgCl2 reaction product in the leaching solution suppresses the leaching rate, and high total chloride concentrations result in the "boiling" of HCl from the solution. The leaching reaction occurs over a diffuse reaction zone up to 400 µm thick. As a consequence, the leaching rate is nearly independent of the particle size for serpentine particles less than 600-800 µm in diameter. The implication is that fine grinding of the asbestos tailings is not required. The leaching reaction generates soluble magnesium and an amorphous silica reaction product. In acid media, only trace silica dissolution occurs; virtually all of the silica forms an insoluble psuedomorph after the original serpentine.

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Reduction of Molten MgO-Bearing Slags with Ferroaluminium: *José Deodoro Trani Capocchi*[1]; V. Rajakumar[2]; [1]University of São Paulo, Dept. of Metallu. and Matls. Eng., Polytech. Schl., Av. Prof. Mello Moraes, 2463, São Paulo, SP 05508-900 Brazil; [2]CSIRO Minerals, Light Met. Product., Clayton, Vic 3172 Australia

Molten slags of the composition 5.6% MgO, 48.1% Al2O3 and 46.3% CaO were reacted with ferroaluminium (80% Al and 35% Al), in graphite crucibles, at 1435-1450°C and reduced pressure 3,066.36-8,399.16 Pa(23-63mmHg). A technique was developed for measuring the rate of evolution of magnesium vapour transported from the reaction zone to a condenser, which was continuously weighted by means of a load cell. It was found that, in the range of operating pressures and composition studied the final yield of magnesium increased linearly as the pressure decreased. The rate per unit driving force with the 80% Al-alloy was 4.7 times that with the 35% Al-alloy mainly because of the larger slag/metal interfacial area when the 80% Al-alloy was used. The overall process appears to be controlled by transport in the slag phase and/or a first order reversible reaction taking place at the slag/metal interface.

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Magnesium Metal by the Heggie-Iolaire Process: *Michael W. Wadsley*[1]; [1]Austherm Pty Limited, P.O. Box 2049, North Brighton, Victoria 3186 Australia

The Heggie-Iolaire Process involves the ambient pressure aluminothermic reduction of magnesia derived from magnesite and dolomite in a thermal plasma arc furnace using selected sources of scrap aluminium. Test work has been conducted at the scale of 2.5 kg/hr and 10 kg/hr magnesium metal production rates. This paper presents some of the results of this test work and of process modeling. A comparison is made between this process and published information concerning other methods for the metallothermic production of magnesium metal. The factors affecting the recovery of liquid metal from its vapour are discussed. A comparison is made between the condensation of magnesium metal and published information for the commercial recovery of liquid zinc metal from its vapour.

3:15 PM

Protective Atmospheres for the Heat Treatment of Magnesium Alloys: *P. F. Stratton*[1]; E. K. Chang[1]; [1]BOC Gases, European Dev. Ctr., Rother Valley Way, Holbrook, Sheffield S203RP UK

Most magnesium alloys are used for die castings which, due to the presence of porous cores, cannot be heat treated at present without blistering. However, as technology improves, pore-free castings will become available whose properties can be optimised by heat treatment and environmentally friendly protective atmospheres will be required for mass production processing. The remaining sand cast magnesium-zirconium alloys, mainly used for aerospace components, are currently heat treated. The most common alloy, ZE41, is usually only treated to the T5 temper whereas the increasingly popular WE43 is treated to T6 temper. During that heat treatment there is a potential fire hazard which can be solved by the correct application of a protective atmosphere. There is always a risk of fire in any magnesium heat treatment due to equipment failure or local furnace hot spots. The risk of ignition is particularly acute for alloys which must be solution treated at over 400°C at which temperature some form of protective atmosphere has historically always been advised. The atmospheres recommended leave a great deal to be desired in terms of effective control, toxicity and environmental impact. A study of non-toxic environmentally friendly alternatives is presented for ZE41 and WE43. One route to pore-free castings is hot isostatic pressing (HIP). If HIP of magnesium castings is to become a standard process within the automotive industry, the cost must not be prohibitive. One way of reducing costs would be to replace the argon pressurisation medium with nitrogen and the effects of doing so are examined.

3:40 PM Break

3:50 PM

The Use of Sulphur Dioxide as a Cover Gas for Molten Magnesium: *S. P. Cashion*[1]; N. J. Ricketts[1]; [1]Australian Magnesium Corporation, Level 6, 9 Sherwood Rd., Toowong, Queensland 4066 Australia

The use of sulphur dioxide (SO_2) as a cover gas component for the oxidation protection of molten magnesium is a viable alternative to sulphur hexafluoride (SF6). The two major problems with SF6 are its high cost and extremely high Global Warming Potential of approximately 24,000. In comparison, SO_2 is relatively inexpensive and has a Global Warming Potential of zero. However, there has been some concern regarding the safety of SO_2, in particular its use as a cover gas in high pressure die casting furnaces. During the 1940's and 1950's, numerous incidents were reported in magnesium die casting operations, occurring in the sulphur domes used to protect the magnesium die casting alloys. This "Sulphur Dome Effect" was attributed to the formation of magnesium sulphate, resulting from the use of SO_2. An investigation was conducted into the Sulphur Dome Effect. Variables examined covered the range of processing operations generally experienced in primary magnesium production and magnesium die casting. The Sulphur Dome Effect was simulated in the laboratory under a range of operating conditions. The reaction associated with the Sulphur Dome Effect appears to occur from a reaction between an accretion on the wall of the crucible and the molten magnesium. Chemical analyses of the accretion and surface film revealed the presence of MgO and $MgSO_4$ in the surface film and the crucible scale. Highly unstable $Al_2(SO_4)3$ was also detected in the crucible scale. These findings support the current proposed mechanism. Further investigations were conducted into the use of sulphur dioxide gas mixtures in ingot and sow casting of pure magnesium and magnesium die casting alloys. The results from this study indicate that the use of SO_2 gas mixtures for the protection of molten magnesium may present a safety problem under certain operating conditions. However, for some applications, such as ingot casting of magnesium die casting alloys, dilute sulphur dioxide gas mixtures are a viable alternative to SF6.

4:15 PM

EPA's Voluntary Partnership with the Magnesium Industry for Climate Protection: *Scott Charles Bartos*[1]; [1]U.S. Environmental Protection Agency, Climate Protect. Div., 401 M St. S.W. (6202J), Washington, DC 20460 USA

The U.S. Environmental Protection Agency (EPA) develops and fosters cooperative partnerships with a wide range of industries to reduce U.S. emissions of greenhouse gases. One such voluntary partnership is the SF6 Emission Reduction Partnership for the Magnesium Industry. Started in 1998, this partnership seeks to reduce the U.S. magnesium industry's emissions of the extremely potent greenhouse gas, sulfur hexafluoride (SF6). The magnesium industry employs SF6 to prevent the rapid oxidation and burning that occurs when the molten metal directly contacts air. A continuous flow (and subsequent release) is required to maintain a protective layer of gas at the melt surface. SF6, an odorless and non-toxic gas, has been the industry standard for melt protection for more than 20 years. However, the industry has recognized that continued emission of this long-lived, extremely potent greenhouse gas is a costly and unsustainable business practice and has begun to work with EPA to reduce emissions and evaluate emission control technologies. In response to this environmental concern, EPA has launched a new initiative to assist the industry in its effort to reduce SF6 emissions. As a voluntary partner, an individual magnesium producer or casting company signs a memorandum of understanding (MOU) with EPA committing to annually report their emissions of SF6 and take cost-effective and technically feasible actions aimed at reducing those emissions. EPA works together with its industry partners to review and evaluate emission reduction strategies and technologies, promote technical information sharing by preparing annual reports and hosting technical conferences, record and verify the partner's progress, and provide positive public recognition for the partners' achievements. The MOU encourages partners to follow a pollution prevention approach to reduce SF6 emissions. This approach, as outlined in the United States Pollution Prevention Act of 1990, presents a hierarchy of emission reduction options that includes source reduction of SF6 by reducing leaks and assuring appropriate cover gas concentrations and flow rates, substitution of SF6 with a more environmentally benign chemical, capture and reuse of SF6, and lastly, destruction of the chemical before release to the environment. Many companies have already implemented various cost-effective source-reduction efforts including regular inspection and maintenance of the gas distribution system, installation of central cover gas blending equipment, and analysis of cover gas concentration and distribution at the molten metal's surface. In addition, a small group of magnesium companies are currently evaluating the technical feasibility and occupational safety concerns associated with installing either SF6 capture/recycle systems or SO_2-based alternative cover gas systems. While the casting technologies of magnesium producers are distinctly different from those of casting companies, both rely heavily on SF6 to provide crucial melt protection. Seven of the approximately forty companies that produce and cast magnesium in the U.S. have joined EPA as partners. As of June 21, 1999, the following companies have signed MOUs with EPA: Acme Die Casting, Chicago White Metal Casting, Del Mar Die Casting, Diemakers, Hyatt Die Cast & Engineering Corporation, Magnesium Products of America, Spartan Light Metal Products. Several more companies are expected to join the partnership this year.

Packaging & Soldering Technologies for Electronic Interconnects: Applications of Multicomponent Phase Equilibria in Electronic Packaging

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

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

Monday PM Room: Lincoln D
March 13, 2000 Location: Opryland Convention Center

Session Chairs: K.-N. Tu, University of California, Dept. of Matls. Sci. Eng., Los Angeles, CA 90095 USA; H. Mavoori, Bell Laboratories, Murray Hill, NJ 07974 USA

2:00 PM Invited
Phase Equilibria of Sn-In Base Micro-Soldering Alloys: *Kiyohito Ishida*[1]; [1]Tohoku University, Dept. of Matls. Sci., Aoba-yama 02, Aoba-ku, Sendai, Miyagi Prefecture 980-8579 Japan

We recently developed a thermodynamic database for the calculation of phase diagrams in micro-soldering alloy systems, which consists of the elements Pb, Bi, Sn, Sb, Cu, Ag and Zn. The important element In was not available at the time. In order to include In in this database, therefore, the phase equilibria of some In base alloys have been experimentally determined by DSC, EDX, X-ray diffraction, etc., and thermodynamic assessments have been made by CALPHAD method. In the present paper, the phase equilibria and thermodynamic properties of some Sn-In-X (X: Ag, Zn, Sb, Bi) ternary systems will be shown, which have practiced applications in the development of Pb-free solders with low melting temperatures.

2:25 PM
Growth of a Au-Ni-Sn Intermetallic Compound on the Solder-Substrate Interface after Aging: *Andrew Murphy Minor*[1]; John W. Morris[1]; [1]University of California, Dept. of Matls. Sci. and Min. Eng., Lawrence Berkeley Labs. Bldg. 66-200, One Cyclotron Rd., Berkeley, CA 94720 USA

When Ni/Au metallization is used to form joints with eutectic solder, the as-solidified joints have $AuSn_4$ precipitates distributed throughout the bulk, with Ni_3Sn_4 at the interface. Recent work has shown that the Au-Sn redeposits onto the interface during aging, compromising the strength of the joint. The present work shows that the redeposit is a ternary intermetallic $Au_{0.5}Ni_{0.5}Sn_4$. While this intermetallic has, to our knowledge, not been seen previously, it appears to be a ternary variant of $AuSn_4$. It does not form during the initial soldering since the solubility of Au in molten Pb-Sn solder separates the Au and Ni constituents.

2:45 PM
Formation and Migration of $AuSn_4$ in BGA Solder Joints Having the Au/Ni Surface Finish: *C. E. Ho*[1]; Y. M. Chen[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Chem. Eng., Chungli, Taiwan

The Au/Ni two-layer structure is a very common surface finish for the solder-ball pads in the Ball-Grid-Array (BGA) packages. During reflow, a layer of $AuSn_4$ will form from the reaction of the Au layer with Sn in the solder. The $AuSn_4$ layer will subsequently break off and fall into the solder joints as individual $AuSn_4$ particles. In the past few years, it was found that post-reflow aging will bring these $AuSn_4$ particles back to the solder-joint/pad interface, forming a dense, continuous $AuSn_4$ layer. This $AuSn_4$ layer will break off and come back repeatedly upon further reflow-aging cycles. The mechanism for this scientifically very interesting phenomenon remains unknown. Being very brittle, the $AuSn_4$ layer will severely deteriorate the strength of a solder joint. Therefore, this phenomenon is also technologically very important. The purpose of our study is to investigate the mechanism for this phenomenon. We will present the most recent results of our study. It is believed that this phenomenon strongly correlates with the low melting point of $AuSn_4$, high diffusivity of Au in solder, and the phase relationships in the Au-Ni-Sn ternary system.

3:05 PM
Use of Multicomponent Phase Diagrams for Predicting Phase Evolution in Solder/Conductor Systems: *Kejun Zeng*[1]; Weiqun Peng[1]; Jorma Kivilahti[1]; [1]Helsinki University of Technology, Dept. Elect. and Comm. Eng., Lab. of Elect. Prod. Tech., Otakaari 5A, PL 3000, Espoo FIN-02015 Finland

Although the complete phase equilibrium is never reached in interconnection applications, the assumption of local equilibrium at the interfaces is generally valid in most systems composed of dissimilar materials. Therefore, the tie lines in ternary (or multicomponent) phase diagrams-together with the relevant stability diagrams and the mass balance requirements-can be used for predicting the phase sequences (i.e. diffusion paths) formed, for example, in solder/conductor joints. Generally, binary phase diagrams cannot provide sufficient information on the phase formation in a solder/conductor systems because they do not bear any information on the relative stabilities between different binary phases in multicomponent systems. As examples, the formation of intermetallic compounds in several solder/conductor systems with Au- or Cu-metallization was studied with the help of ternary phase diagrams as well as experimentally, demonstrating the inadequacy of the binary information. The phase diagrams were calculated by using the thermodynamic methods. The experimental results confirmed that the dependence of formation of intermetallic compounds on temperature and solder composition is clearly represented by the ternary phase diagrams supplemented with the stability diagrams.

3:25 PM Break

3:40 PM Invited
Thermodynamic Assessment of the Sn-Ag-Cu System: *U. R. Kattner*[1]; K. -W. Moon[1]; W. J. Boettinger[1]; C. A. Handwerker[1]; [1]NIST, Metallu. Div., Mail Stop 8555, Gaithersburg, MD 20899 USA

Experimental data on the Sn-Ag-Cu system will be summarized. Alloys in this system are being studied for their potential as Pb-free solders. Thus the location of the ternary eutectic involving L, Sn, Ag_3Sn and Cu_6Sn_5 phases in the Sn-rich corner is of critical interest. Thermodynamic extrapolation of the diagram from the three constituent binary systems using the CALPHAD method will be presented. A comparison to the experimental data will indicate the need for refined binary assessments and/or ternary interaction parameters. For various solder compositions, solidification paths and freezing ranges will be predicted with a Scheil analysis. These predictions will be compared to experimental solidification results obtained in this work and in the literature.

4:05 PM
Phase Equilibria and Related Properties of Sn-Ag-Cu Based Pb-free Solder Alloys: *I. Ohnuma*[1]; X. J. Liu[1]; H. Ohtani[1]; K. Ishida[1]; [1]Tohoku University, Dept. of Matls. Sci., Aoba-yama 02, Aoba-ku, Sendai, Miyagi Prefecture 980-8579 Japan

We recently developed a thermodynamic database for micro-soldering alloys which consists of the elements Pb, Bi, Sn, Sb, Cu, Ag, Zn, and In. In this paper, the phase equilibria and the related thermodynamic properties of the Sn-Ag-Cu base alloys are presented using this database. These alloy systems are promising candidates of Pb-free solders. The isothermal section diagrams of the Sn-Ag-Cu ternary system were experimentally determined by EDX, X-ray diffraction and metallographic techniques. Based on the present results as well as previous data on phase boundaries, thermodynamic assessments of this system were carried out. The isothermal and vertical section diagrams, liquidus surface, mole fractions of the phase constitution etc. have been calcu-

lated. Moreover, non-equilibrium solidification process using the Scheil model has been simulated and compared with the equilibrium solidification behavior in some Sn-Ag-Cu base alloys. In addition, the predictions of surface energy and viscosity are presented.

4:25 PM

Ag and Cu Migration Phenomena on Wire-Bonding: *Ker-Chang Hsieh*[1]; Theo Martens[2]; [1]National Sun Yat-sen University, Instit. of Matls. Sci. and Eng., Kaohsiung, Taiwan; [2]Philips Electronic Building Elements Industries Limited, Tech. Dev. Div., 10, Chin 5th Rd. N.E.P.Z., P.O. Box 35-48, Kaohsiung, Taiwan

The plastic packaged sample stored at 250°C for 588hrs and found the Au plus 1%Pd wire composition changed. The Ag and Cu atoms can migrate from the wedge bond through the wire surface and arrive the ball bond. At the same time, the Ag and Cu atoms diffuse into the gold wire itself and form a layer type structure. These migration phenomena can be explained after detailed microstructure analysis. The microstructure analysis was done by using the apparatus: Joel Superprobe JXA-8900R. The quantitative line scan analysis result included the identified phases and phase thickness. A similar line scan analysis was done on the ball bond section. The results has the similar layer structure as the wire section. There is two type of driving force for these atom migration phenomena. One is the Ag and Cu concentration gradient to drive the diffusion process. The other is the new alloy phases formation, which can reduce the free energy of this alloy system, based on the thermodynamic rules. The phases formation sequence formed on the wire or ball bond section is consistent with the equilibrium Ag-Au-Cu phase diagram. Conclusions: 1. Ag and Cu atoms can migrate on the wire surface and form new alloy phases. 2. The properties of gold wire will change under high temperature and long time conditions. 3. The diffusion rate of Cu is higher than Ag according to this study. 4. Impurity atoms in the molding compound may transport through the wire surface and reach the ball bond area causing corrosion or other degrading problems.

4:45 PM

The Tin-Rich Corner of the Copper-Magnesium-Tin Phase Diagram: *Eduardo E. Vicente*[1]; Alicia N. Alcaraz[1]; [1]Comisión Nacional de Energía Atómica, Matls. Dept., Av. del Libertador 8250, Buenos Aires 1429 Argentina

The copper-magnesium-tin ternary phase diagram was studied in the region Sn-Cu_6Sn_5-CuMgSn-Mg_2Sn. The employed experimental techniques were: optical microscopy, scanning electron microscopy, X-ray diffraction, electron-probe microanalysis and differential thermal analysis. A liquidus projection is proposed, which includes three invariant reactions: two ternary eutectics and a pseudobinary eutectic.

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The Tin-Rich Corner of the Copper-Tin-Zinc Phase Diagram: Alicia N. Alcaraz[1]; *Eduardo E. Vicente*[1]; Luis M. Gribaudo[1]; [1]Comisión Nacional de Energía Atómica, Matls. Dept., Av. del Libertador 8250, Buenos Aires 1429 Argentina

The copper-tin-zinc ternary phase diagram was studied in the region delimited by the tin corner and the 50 at%Sn isopleth. The employed experimental techniques were: optical microscopy, scanning electron microscopy, X-ray diffraction, electron-probe microanalysis and differential thermal analysis. A liquidus projection and a subsolidus isothermal section are presented.

Pressure Technology Applications in the Hydrometallurgy of Copper, Nickel, Cobalt and Precious Metals: Pressure Technology Applications in the Hydrometallurgy of Gold

Sponsored by: Extraction & Processing Division, Copper, Nickel, Cobalt Committee

Program Organizers: James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

Monday PM — Room: Lincoln C
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Christopher A. Fleming, Lakefield Research Limited, Lakefield, Ontario K0L2H0 Canada; James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA

2:00 PM

A Novel Process for the Simultaneous Dissolution of Gold, Platinum Group Metals and Base Metals: *Christopher A. Fleming*[1]; C. Joe Ferron[1]; Dave B. Dreisinger[2]; P. Terry O'Kane[3]; [1]Lakefield Research Limited, 185 Concession St., P.O. Box 4300, Lakefield, Ontario K0L2H0 Canada; [2]University of British Columbia, Dept. of Met. & Matls. Eng., 309-6350 Stores Rd., Vancouver, British Columbia V6T1W5 Canada; [3]O'Kane Consultants Inc., 502-455 Granville St., Vancouver, British Columbia V6C1V2 Canada

There are many examples of ores, concentrates and other materials containing base metals (i.e. Cu,Ni,Co) as well as gold and platinum group metals PGMs in which either the distribution of base metals and PGMs is unfavorable, or their concentrations are too low, for these materials to be economically treated by conventional technology (matte smelting, acid pressure leaching, chlorination, etc.). A new hydrometallurgical process has been developed for treating these feed materials, and will be described in this paper. The process has the potential to significantly improve the economics of treating feeds containing base metals and PGMs. The main feature of the process is high temperature pressure leaching, under conditions that allow simultaneous and efficient (88 to 99%) dissolution of all the base metals and PGMs. Technology has also been developed to treat the product of the leaching process and recover the base metals and PGMs as separate saleable products.

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The Dissolution of Gold during the Pressure Leaching of Refractory Gold Ores in the Presence of Chloride: *Michael J. Nicol*[1]; Jim Qing Liu[1]; [1]Murdoch University, A J Parker CRC, South St., Murdoch, Western Australia 6150 Australia

Pressure oxidation is one of the preferred methods for the recovery of gold from refractory ores and concentrates. There have been several reports of the dissolution of gold during the acid pressure oxidation process and this has been attributed to the presence of chloride in the plant water or ore. This paper will describe the results of an electrochemical study of the behaviour of gold in acidic sulphate solutions containing varying amounts of chloride ions at high temperatures. It will be shown from both thermodynamic and kinetic studies that the oxidation of gold occurs as a result of the coupled anodic dissolution of the metal as the chlorocomplex ion with the cathodic reduction of ferric ions. Oxygen is not directly involved in the dissolution process. Conditions which will either minimise or maximise the dissolution of gold can be deduced from the thermodynamic and kinetic models.

3:00 PM
A Mineralogical Study of the Cyanide Leach Residues from Pressure-Oxidized Twin Creek Gold Ore: *T. T. Chen*[1]; J. E. Dutrizac[1]; G. L. Simmons[2]; [1]CANMET, 555 Booth St., Ottawa K1A0G1 Canada; [2]Newmont Mining Corporation, Englewood, CO 80112 USA

Gold in the Twin Creek ore is primarily associated with arsenian pyrite. During pressure oxidation, the sulphides are solubilized, but part of the dissolved iron reprecipitates as jarosite and as an iron sulphate-arsenate-phosphate phase. The pressure oxidation products are subjected to cyanide leaching. The cyanide leach residues consist of quartz, gypsum, bassanite, anhydrite, small amounts of jarosite, an iron-sulphate-arsenate-phosphate phase, as well as trace amounts of orthoclase, rutile, pyrite, hematite and monazite; all these particles are dispersed in a fine grained matrix of illite-muscovite. The autoclave leach residues contain a small amount of carbonaceous matter. Many gold grains are detected on the surface of the carbon particles, and these gold particles are not leached during cyanidation. The gold particles are typically 0.1-0.2 µm in size and commonly are embedded in the carbon surface or are coated with a K-Al-Si-S-O phase on the surface of the carbon. The gold grains are consistently associated with elevated CI contents (50-300 ppm Cl), and the implication is that the gold was transported to the surface of the carbon as a gold chlorocomplex. The poor gold cyanide leach recoveries, which initially occurred in the Twin Creek developmental circuit under certain operating conditions, are attributed to the presence of chloride in the feed. This leads to the formation of soluble gold chlorocomplexes in the autoclave and the subsequent sequestering of the gold on the surfaces of the carbon particles.

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Alkaline and Acid Autoclaves at Barrick Gold: *Kenneth Glyndwr Thomas*[1]; *Richard Williams*[1]; [1]Barrick Gold Corporation, Operations Dept., 200 Bay St. P.O. Box 119, S. Tower Ste. 2700, Toronto, Ontario M5J 2J3 Canada

This paper reviews the alkaline and acid gold autoclaving experiences gained at Barrick Gold Corporation. Alkaline autoclaving was operational at Barrick Mercur, Utah, USA, on carbonaceous/sulphide refractory ore for eight years commencing in 1988. Acid autoclaves have been operational at Barrick Goldstrike, Nevada, USA., on sulphide refractory ore since 1990 and now process 17,500 stpd of ore through six large autoclaves. Information is presented on operating costs, materials of construction, reagent requirements and flowsheet developments that reduced capital and operating costs.

Professional Registration

Sponsored by: TMS, Young Leaders Committee, Professional Registration Committee
Program Organizers: Ned Bahtishi, Westinghouse Electric Company, Western Zirconium Plant, Ogden, UT 84404-9799 USA; David Alven, GENCORP/AEROJET, Jonesborough, TN 37659 USA

Monday PM — Room: Bayou A
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Ned Bahtishi, Westinghouse Electric Company, Commercial Nuclear Fuel, Ogden, UT 84404 USA; David A. Alven, GenCorp, Aerojet Ordnance, Jonesborough, TN 37659 USA

2:00 PM Opening Comments
Dr. Patrick Taylor, Professional Registration Committee, Chairman

2:10 PM
Metallurgical P.E.; What Do I Do and Why?: *Charles V. White*[1]; [1]Kettering University, I&MSE, 1700 W. Third Ave., Flint, MI 48504 USA

A discussion of the mechanics of getting licensed, what's on the exam, and why I need to do it? A review of the requirements to be licensed and the paper work needed and the timing will be covered. Secondly, what's on the exam (not a crib) but how the exam is prepared, what are the guidelines for the items and the test make up. Lastly, why do I want to put myself through all this pain? What are the issues with industrial employment vs. independent contractors and how these issue effect you as a professional.

2:35 PM
Professional Registration: A Higher Standard: *Christy Allen*[1]; [1]Tennessee Department of Commerce and Insurance, Legal Sect., 312 8th Ave. N., 25th Floor Tennessee Tower Bldg., Nashville, TN 37243 USA

State registration boards exist to protect the health, safety and welfare of the public. Boards accomplish this mission by ensuring that only properly qualified people become registered as professional engineers and that, once registered, they provide professional services in a manner consistent with protection of the public. Professional engineers are held to high standards of practice and rules of professional conduct; they face disciplinary action if they fail to uphold those standards. This program is a discussion of the ways in which the regulation of professional engineers, through the enforcement of rules of conduct, benefits professional engineers as well as the public.

3:00 PM Break

3:10 PM
The Registered Engineer as an Expert Witness: *J. Mike York*[1]; [1]York Engineering Services, 2107 N.W. Filmore Ave., Corvallis, OR 97330 USA

The current environment for consulting engineers working as expert witness is demanding. A 1993 Supreme Court decision has placed judges in the role of gate keepers to prevent "junk science" from being presented to the juries. These requirements, in turn, place an increasing burden on the expert witness to provide evidence as to their qualifications to testify. Professional registration provides an excellent reference to establish the individual as having met the requirements of knowledge in their designated field. Registration confirms the individual has met peer reviewed examination in one or more jurisdiction, i.e. state(s), that extend well beyond academic institution or single organization. For engineers trying to break into the expert witness field, professional registration credentials must be substituted for courtroom experience until that experience can be obtained. Professional registration is a key element in credential building for the aspiring expert witness.

3:35 PM
The Practice of Being a Professional Engineer: It's More Than Just a Stamp: *Larry M. Southwick*[1]; [1]L.M. Southwick & Associates, 992 Marion Ave., Ste. 306, Cincinnati, OH 45229 USA

A professional engineer's license is often required to certify the competence of a particular design or other document. These situations usually arise to fulfill certain legal requirements. However, many, probably even most, design and other engineering work do not require formal certification, but they always require, and deserve, a competent effort. Here is where professional registration perhaps has its greatest value: as a sign of trust in the work being performed. Many engineering and operating companies require that the lead engineers be registered, again not necessarily to autograph documents, but more as an indication of overall confidence in the staff and its work. While obtaining an engineering registration requires only several years of practice and satisfactorily passing a qualifying exam, certainly necessary and no mean achievements to be sure, practicing to that standard afterwards demands a continuing effort. This entails not only maintaining quality work, but doing so with integrity and forthrightness. The presentation will provide several examples of how being a professional engineer means more than just having a P.E. license stamp in one's kit.

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The P.E. License: Its Value in Industry: *Nick Gianaris*[1]; [1]Visteon, an Enterprise of Ford Motor Company, Chassis Sys., 6100 Mercury Dr., Dearborn, MI 48126 USA

Engineering is a field whose practitioners have various educational and experiential backgrounds in all industries and academe. Unlike other professional fields such as medicine and law, an engineer is not always required to be licensed to practice engineering in many industrial sectors. In this paper, the following will be presented: who is required to become licensed as an engineer today, why licensure in industry is significant for both the engineer and the company, personal experience in obtaining and using the P.E. license, and why the P.E. license will become more important in the future.

Surface Engineering in Materials Science I: Coatings/Films Synthesis and Processes (SP)-II

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

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

Monday PM — Room: Canal B
March 13, 2000 — Location: Opryland Convention Center

Session Chairs: Narendra B. Dahotre, University of Tennessee Space Institute, Dept. of Matls. Sci. & Eng. Ctr. for Laser App., Tullahoma, TN 37388 USA; Enrique Lavernia, University of California Irvine, Dept. of Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697-2575 USA

2:00 PM

Thin Films of Magnetic Semiconductors-Transport Phenomena and Magnetic Properties: *L. J. Maksymowicz*[1]; M. Lubecka[1]; B. Cieciwa[1]; [1]University of Mining and Metallurgy, Instit. of Elect., Al. Mickiewicza 30, Kraków 30-059 Poland

Thin films of CdCr2Se4:In and CdCr2-2xIn2xSe4 were obtained by rf sputtering technique. We used the deposition device equipped with three cathode system and rotatable substrate holder. As-deposited samples are in amorphous state and have a form of multilayered structure of Cr/Cd-Cr-In-Se/Cr. Heat treatment provides uniform polycrystalline single films with the required composition. The samples belong to the class of soft magnetic materials; CdCr2Se4:In has the reentrant transition (REE), the energetic structure is modified diluting by In and CdCr2-2xIn2xSe4 is in the spin glass state. Both types of samples have potential for use as photodetectors. The maximum of photoconductivity is within the wavelength from 680nm to 840nm. It was found that the maximum of voltage sensitivity is shifted towards the infrared region when dilution levels are reduced. As far as the magnetic properties are concerned, we are classifying the type of magnetic order by determination the temperature dependence of magnetization (M) and unidirectional magnetic anisotropy field (Han) [1]. We have also determined the surface magnetic anisotropy energy constant versus temperature. It was found that two components contribute to this constant [2]. One originates from the exchange interaction term due to the lack of translation symmetry for surface spins as well as from the stray field of surface roughness. The second one comes from the demagnetizing field of close-to-surface layer with grad M. Both terms linearly decrease when temperature increased from 5 to 123K, but dominant contribution is from the first component. The work was partly supported by KBN Grant Nr 10.120.68. Literature [1] E.M. Jackson, S.B. Lio, S.M. Bhagat and M.A. Manheimer, J. Magn. Magn. Mater. 80(1989) 229. [2] L.J. Maksymowicz, M. Lubecka and R. Jablonski J. Magn.Magn.Mater. 192 (1999).

2:20 PM

Laser Surface Modification of TiAl Intermetallics: *S. A. McElroy*[1]; D. Yang[1]; R. G. Reddy[1]; [1]University of Alabama, Metallu. and Matls. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

Surface modification of TiAl intermetallics was investigated using Nd-YAG pulse laser. Laser processed samples were characterized for their surface morphological and compositional changes using SEM, XPS and X-ray diffraction. Large cracks along the grain boundaries and surface oxide layers in the laser processed samples were observed. Oxide layers consisted of Al2O3, TiO2, TiO and absorbed O2. Aluminum enrichment in the oxidation layers of TiAl was observed.

2:40 PM

The Synthesis, Processing and Properties of Graded Thin Film/Coating: *John J. Moore*[1]; [1]Colorado School of Mines, Adv. Coat. and Surf. Eng. Lab., Golden, CO 80401-1887 USA

The paper will discuss the design, processing and the resultant properties of graded coating systems that are needed to meet specific performance criteria. Three examples of graded coating systems will be used to demonstrate the philosophy used for coating systems for (a) forming dies, (b) oxidation resistant coatings for Mo, and (c) glass molding dies, as used in the current research programs in the Advanced Coatings and Surface Engineering Laboratory at the Colorado School of Mines.

3:00 PM Break

3:15 PM

Indium Oxide Thin Film with a Large Surface Area: *T. Pisarkiewicz*[1]; T. Stapinski[1]; A. Sutor[1]; K. Zakrzewska[1]; [1]University of Mining and Metallurgy, Dept. of Elect., Al. Mickiewicza 30, Krakow 30-059 Poland

Indium oxide (In2O3) is known as one of the gas sensing materials and then the films with a high surface-to-volume ratio are promising candidates for gas sensors. The authors deposited the films by rheotaxial growth and thermal oxidation (RGTO) technique. This technology is a two-step process in which the first step is the deposition of metallic indium onto substrates heated above the melting temperature of In and in the second step the metallic droplets are thermally oxidized in air forming the polyctystallline film. Optical reflection with the help of hemispherical attachment were performed enabling determination of light scattering. Electrical transport (conductivity, Hall effect) measured in varying gas atmosphere and temperature indicate the essential role of thin film surface in the interaction of semiconductor with the ambient gas.

3:35 PM

Si-N Coating by Plasma CVD Method: *Y. Sato*[1]; S. Ohtani[1]; N. Iwamoto[1]; [1]Ion Engineering Research Institute Corporation, 2-8-1, Tsuda-yamate, Hirakata, Osaka 573-0128 Japan

Si-N coating on steel by plasma CVD method was investigated to improve mechanical properties as hard coating. Application of pulsed DC was used for plasma formation. SiCl4 was used for silicon source. Nitrogen gas and ammonia gas were used for nitrogen source and effects of gas species on properties of films was investigated. In the case of nitrogen gas coating, nitrogen contents in Si-N films were increased with the increment of pulsed DC power up to 30at% at 2.0kW. Nitrogen contents in films coated with ammonia gas were also increased with the increment of DC power up to 50at%, which approached stoichiometry of stable silicon nitride. All of obtained Si-N coating films were formed in amorphous structure. Si2p peaks at 99.5eV (Si-Si bond) and 101.6eV (Si-N bond) were appeared with 30at% nitrogen containing films by XPS measurement. Coating with 50at% nitrogen content showed only one peak at 101.6eV. These results show that

more Si-Si bond was included in low nitrogen containing film and high nitrogen content film consist of Si-N bond. Hardness of Si-N film was changed with variation of applied DC power. The maximum hardness of HV1800 was obtained at 0.5kW DC power.

3:55 PM

Selective Area Laser Surface Alloying of Mild Steel with Carbon: *V. Sinha*[1]; G. L. Goswami[2]; G. B. Kale[2]; I. Manna[3]; [1]Ohio State University, Dept. of Matls. Sci. and Eng., Columbus, OH 43210 USA; [2]BARC, Atomic Fuels Div., Trombay, Mumbai 400085 India; [3]I. I. T., Kharagpur, Dept. of Metallu. and Matls. Eng., Kharagpur, WB 721302 India

Carburizing is an age old surface engineering technique to enhance hardness and wear resistance of steel. However, carburizing exposes the entire component to a complex heat treatment schedule and possesses several limitations in precision, energy/time economy and degree of improvement. The present study concerns laser surface alloying (LSA) with a pre-deposited carbon coating for selective area hardening of steel components without affecting the entire bulk. Rectangular specimens of 0.2% C steel were coated with 40 and 90 mm thick (d) carbon deposited by physical vapor deposition and irradiated with a 300 W Nd-YAG pulsed laser with output energy (E) = 6-12 J and pulsed duration (t) = 8-14 ms, respectively. LSA with an optimum conditions develop a predominantly martensitic microstructure with a very high (750-1000 VHN) microhardness. However, an increase in d, t and/or E leads to a higher w with a greater volume fraction of retained austenite in the microstructure and a lower hardness in the near surface region. Finally, an attempt has been made to correlate the microstructure, composition and hardness of the AZ with the LSA parameters.

4:15 PM

Synthesis of Nanocrystalline Diamond Thin Films by Chemical Vapor Depositions: *D. Zhou*[1]; A. Hussian[1]; L. Chow[2]; [1]University of Central Florida, Adv. Matls. Process. and Analy. Ctr., Dept. of Mech., Matls., and Aero. Eng., Eng. 381, Orlando, FL 32816 USA; [2]University of Central Florida, Dept. of Physics, Orlando, FL 32816 USA

Polycrystalline diamond films whose microstructures typically consist of crystallites with sizes on the order of nanometers have been successfully synthesized by a hot filament chemical vapor deposition (CVD) technique. Mixtures of methane, hydrogen, and argon were used as the reactant gases for the CVD processing, and the substrate (Si) temperatures ranged from 450 to 800°C. X-ray diffraction, transmission electron microscopy, and electron energy loss spectroscopy characterizations show the films consist of a pure crystalline diamond phase with grain sizes ranging from 10 to 30 nm. Scanning electron and atomic force microscopies analyses demonstrate that the surface of the nanocrystalline diamond films remain smooth and independent of the film thicknesses. The growth morphology of the diamond coatings, particularly the transition from microcrystalline to nanocrystalline diamonds, has been discussed in the light of the functions of the atomic hydrogen and the secondary nucleation, which are strongly affected by the hot filament CVD processing parameters. Furthermore, the potential applications of the nanocrystalline diamond thin films, such as electron field emitters, diffusion barriers, and protective coatings have been addressed.

4:35 PM

Preparation and Properties of Mo/CIS/CdS Thin Film Interfaces for Photovoltaic Applications: *S. R. Kumar*[1]; [1]National Institute of Foundry and Forge Technology, Dept. of Matls. and Metallu. Eng., Ranchi, Bihar 834 003 India

Copper indium selenide (CIS) thin film solar cells can be prepared by electrodeposition, dip coating and flash annealing at 400°C. The CIS thin films were prepared by single step electrodeposition process on molybdenum substrate potentiostatically or galvanostatically. The as deposited films are crystalline and are preferably oriented along (112) direction have low resistivity which in turn is further reduced by annealing in air. The cadmium sulphide films were prepared by dip coating technique on glass, as well as on molybdenum and CIS thin film substrates. The as deposited films are highly resistive and oriented along (002) direction. The resistivity is considerably reduced and crystallinity increases by flash annealing at 500°C. The X-ray diffractogram of the CIS thin film solar cell shows the crystalline and single phase orientation. The SEM analysis indicates well connected spherical grains which are densely packed. The I-V characteristics of the films are forward and reverse biasing is similar to the ideal diode. The open circuit voltage Voc and short circuit current density Jsc are respectively 100 mV and 5 mA/cm2. The results are preliminary in nature and by optimizing the growth conditions further improvement in the results are expected.

Teaching Electronic, Magnetic and Optical Materials: A Symposium in Memory of Professor Gregory E. Stillman: Session II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Education Committee

Program Organizer: Mark A. Palmer, Virginia Commonwealth University, Richmond, VA 23284-3015 USA

Monday PM — Room: Bayou D
March 13, 2000 — Location: Opryland Convention Center

Session Chair: Mark A. Palmer, Virginia Commonwealth University, Richmond, VA 23284-3015 USA

2:00 PM Invited

Linking Mathematics to Materials Science Through Interactive Visualization: *Shannon Pixley*[1]; Krishna Rajan[1]; [1]Rensselaer Polytechnic Institute, Depts. of Comp. Sci., Matls. Sci., and Eng., Troy, NY 12180-3590 USA

A number of mathematical manipulation skills are needed in many materials science courses. In this project, we are developing interactive computer applets teaching linear algebra principles in the context of crystallographic applications. The modules are designed to provide a direct relationship between specific skill building exercises, such as matrix manipulation, to a materials science topic. A series of applets have been built permitting the student to visualize graphically the crystallographic perspectives of specific matrix operations. This project is part of a larger effort known as Project Links, providing teaching tools in mathematics for application in a variety of engineering subjects.

2:30 PM Invited

Laboratory Experiences in Electronic Materials at the University of Michigan: *Rachel S. Goldman*[1]; [1]University of Michigan, Dept. of Matls. Sci. and Eng., Ann Arbor, MI 48109-2136 USA

We are developing a series of laboratory modules in electronic materials and have started to incorporate these into our undergraduate curriculum at the University of Michigan. Example modules include: (a) resistivity vs. temperature of metals, semiconductors, and insulators, (b) Hall effect measurements, (c) current-voltage measurements of solar cells, (d) scanning tunneling microscopy, and (e) atomic force microscopy. In this talk, I will discuss the new laboratory modules we have begun to incorporate into our junior level laboratory courses. I will also discuss some of our future plans which include the addition of magnetization and measurements of ferromagnetic materials.

3:00 PM Invited

Multidisciplinary Program in Sensor Materials and Devices: *Sheikh Akbar*[1]; Prabir Dutta[1]; Marc Madou[1]; Bruce Patton[1]; Yunzhi Wang[1]; [1]The Ohio State University, Ctr. for Indust. Sens. and Measure., 291 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Under the umbrella of the NSF Center for Industrial Sensors and Measurement (CISM), a research and development program for harsh environment sensors is being actively pursued at Ohio State University. Research teams include students and faculty from Departments of Chemistry, Physics, Materials Science and Engineering, and Electrical,

Chemical and Mechanical Engineering. The CISM model has enriched engineering and physical science interdisciplinary education and has allowed us to develop a multidisciplinary industry-oriented curriculum that is currently being funded by the NSF-CRCD program and OSU Honors House. Under this program, a new three-course sequence (9 credit hours) in sensor materials including instructional laboratories with industrial experience is currently being developed. The courses are being designed around the multidisciplinary approach of CISM, and are being team-taught by faculty members from a wide range of disciplines. These courses are targeted for senior undergraduate and beginning graduate students. The first course covering basic scientific principles of sensor materials has already been offered once during the Spring quarter of 1999. The second course will cover different sensor applications and related technological issues. Both courses have a laboratory component. The third course will be group projects with participating industries. Group projects will target specific industries, identify a sensor need, develop a prototype and perform field-tests at the industrial site. Each project will be a team effort involving multiple students working in close collaboration with a faculty advisor and an industry co-adviser. Students entering the courses will have the appropriate background in science and engineering. Students taking this sequence along with 11 credit hours of relevant courses in participating departments including Business and Law will have the option to receive a minor or certificate degree in "Sensors and Measurements."

Ultrafine Grained Materials: Fundamentals and Process Mechanisms: II

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

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

Monday PM — Room: Polk A/B
March 13, 2000 — Location: Opryland Convention Center

Session Chair: C. Suryanarayana, Dept. of Metallu. and Matls. Eng., Golden, CO 80401 USA

2:00 PM Invited

Role of Water in Energetics of Zeolites and Layered Materials: *Alexandra Navrotsky*[1]; [1]University of California, Dept. of Chem. Eng. and Matls. Sci., Thermochemistry Facility, Davis, CA 95616 USA

The UC Davis Thermochemistry Facility has been studying the enthalpies of formation of zeolites, octahedral molecular sieves, and layered materials by a combination of high temperature reaction calorimetric techniques. We find several common trends. Dehydrated frameworks are metastable with respect to dense phases by 5-15 kJ/mol but hydrated frameworks are energetically stable by a similar amount (referred to liquid water and a 2-oxygen framework formula unit). This energetic stabilization is counterbalanced by a negative entropy of hydration. The ΔH and ΔS terms scale with each other. Thus the localization of water within the cage or layer is a major driving force in the formation of both framework and layered materials. New thermochemical data for a variety of zeolites and manganese oxide based nanomaterials will be reviewed in the context of hydration energetics.

2:25 PM Invited

Thermal Spraying of Nano-Composite Coatings: *E. J. Lavernia*[1]; R. Rodriguez[1]; M. Ice[1]; M. L. Lau[1]; [1]University of California, Dept. of Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697-2575 USA

Recent advancements developed in the thermal spraying of nanocrystalline materials have attracted scientific and industrial interest. Due to the relatively short dwell time of the powder particles during the thermal spray process and their inherent thermal stability, the feedstock powders are able to preserve the unique property of nanocrystalline materials in the as-sprayed coatings. The use of nanocermet powders such as WC-Co has produced coatings with increased microhardness while maintaining toughness values as compared to conventional coatings. In the present study, nano-composite Al coatings were produced by plasma spraying. The feedstock nanocrystalline powders were synthesized by mechanical milling of gas atomized 3003-Al powders with the addition of 10 vol.% SiC in liquid nitrogen for 8 hrs to produce agglomerates with increased surface area. The cryomilled powders were thermally sprayed by vacuum plasma spraying to produce Al-composite coatings. The residual stress of the nano-composite coating will be determined by X-ray diffraction analysis and compare to that of the conventional coating. The results will be rationalized based on the microstructural features observed in the transmission electron microscopy and scanning electron microscopy analyses. In addition, coating properties were characterized by microhardness and wear measurements performed on the coating cross sections and compared to those of the conventional Al-SiC coating.

2:50 PM Invited

A Cost-Effective Way to Make Nanostructured Carbides and Nitrides: *Leon L. Shaw*[1]; [1]University of Connecticut, Dept. of Metallu. and Matls. Eng., Storrs, CT 06269 USA

The time and energy needed to make nanostructured or conventional carbide and nitride powders for sintered components, such as valves, seals and bearings, could be cut to a fraction of what is now needed through a novel process developed recently at the University of Connecticut. The new process combines thermal and mechanical activation to enhance the reaction of compound formation. The basic form of the new process is to mechanically activate the reactants at room temperature through high energy milling, followed by completing the synthesis reaction at high temperatures. High energy milling at room temperature has substantially increased the reactivity of the reactants and dramatically reduced the final reaction temperature and time. The enhanced reaction has been attributed to the structural and energy state changes of the reactants caused by the mechanical treatment prior to the reaction. These structural and energy state changes contribute to the enhanced reduction through the increased reaction kinetics as well as the increased reaction driving force. Examples to illustrate these underlying mechanisms will be presented.

3:15 PM

Synthesis of Fe-TiN Composites by Thermal Plasma Processing: *Sutham Niyomwas*[1]; Banqiu Wu[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metallu. and Matls. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

The Fe-TiN composite was synthesized in a non-transferred arc d.c. plasma reactor from ilmenite ore concentrate using methane and ammonia as the reactive gases. The standard Gibbs energy minimization method was used to calculate the equilibrium composition of reaction species. The products were characterized by X-ray diffraction and SEM. Effect of plasma power, gas composition, ilmenite particle size, feeding rate and particle injection position on purity of composite were investigated. Experimental and theoretical results were discussed.

3:35 PM

Activated Sintering of Al_2TiO_5 Nanoceramics: *Joanna Groza*[1]; Vladimir Kodash[1]; Lia Stanciu[2]; Maria Zaharescu[2]; [1]University of California, Chem. Eng. Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Institute of Physical Chemistry-Romanian Academy, Bucharest, Romania

The most critical stage in obtaining nanoceramics is sintering. Key to success is preserving the nanocrystalline size of starting powders in final consolidated ceramics. One way to achieve this objective is by sintering activation using an externally applied electrical field. The

essence of field activation is to enhance densification of ceramic powders by decreasing the temperature and time of sintering, thereby considerably suppressing the grain growth. Field activated sintering technique (FAST) was applied to Al_2TiO_5 nanoceramics formed from BINARY Al_2O_3-TiO_2 powders obtained by sol-gel method. It was found that electric field activates formation of Al_2TiO_5 and its densification in the temperature range 1050-1250°C. The influence of electric field on Al_2TiO_5 formation, crystallization densification and final properties will be presented.

3:55 PM Break

4:05 PM Invited

Synthesis of Nanoparticles by a Novel Laser-Liquid-Solid Interaction Technique: *J. Singh*[1]; [1]Pennsylvania State University, Appl. Rsch. Lab., University Park, PA 16804 USA

A novel laser-liquid-solid interaction (LLSI) technique has been developed for synthesizing nanoparticles of materials including Ag, Ni, Ag-Ni alloy, SnO_2, Cu from liquid precursors. Rotating niobium substrates immersed in the liquid precursor were irradiated by a continuous wave CO_2 and Nd-YAG laser (λ = 1064 nm). Size and morphology of nanoparticles was dependent on various process parameters including laser energy, wavelength, precursor chemistry and interaction time. Two-phase alloys containing silver and nickel were fabricated by LLSI technique. The powders were characterized by x-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and high-resolution transmission electron microscopy (HRTEM). The synthesis mechanism of non-equilibrium Ag-Ni alloy nanoparticles has been proposed to occur primarily at the laser-liquid-solid interface by a nucleation and growth mechanism.

4:30 PM

Consolidation of Mechanically Alloyed Cu-In-Ga-Se Powders by a Field Activated Sintering Technique (Fast): C. Suryanarayana[1]; J. Curtis[2]; *J. R. Groza*[2]; [1]Colorado School of Mines, Dept. of Metallu. and Matls. Eng., Golden, CO 80401 USA; [2]University of California, Chem. Eng. and Matls. Sci., One Shields Ave., Davis, CA 95616 USA

Recently, copper indium diselenide ($CuInSe_2$)-based photovoltaic solar cells have received considerable attention due to a high conversion efficiency, up to 17.7% for a Cu-In-Ga-Se device. The present work presents an alternate processing route to the usually long deposition methods. The new approach involves mechanical milling of elemental powders followed by consolidation into bulk parts. Mechanical milling achieved powder blending and synthesis of the stoichiometric $CuIn_{0.7}Ga_{0.3}Se_2$ compound. The mechanically milled powders were consolidated to a 5.51 g/cm^3 density by a field assisted sintering technique. The final grain size was in the nanometer range (~ 63 nm, as determined by XRD Warren-Averbach method).

4:50 PM

A Unified Viscoplastic Model for Densification of Powder Compacts: *Hyoung Seop Kim*[1]; Yuri Estrin[2]; Elazar Gutmanas[3]; [1]Chungnam National University, Dept. of Metallu. Eng., Taejon 305-764 Korea; [2]The University of Western Australia, Dept. of Mech. and Matls. Eng., Nedlands, WA 6907 Australia; [3]Technion, Dept. of Matls. Eng., Haifa 32000 Israel

A model for densification of metallic powders is proposed. It involves viscoplastic constitutive equations based on dislocation density evolution and also accounts for effects of porosity. The model was applied to the case of cold powder compaction under uniaxial compression conditions. Densification behaviour during powder compaction was simulated using a combination of the implicit and explicit integration methods as applied to the dislocation density evolution and the variation of the relative density of the compact, respectively. The model was gauged by comparing the experimental data generated by the cylindrical die compaction tests on Cu powder with the simulation results.

5:10 PM

Synthesis of TiC/Fe-Al Nano-Nano Composites: *E. G. Baburaj*[1]; R. Fielding[1]; E. Nyberg[2]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]Pacific Northwest National Laboratory, Battelle Blvd., P.O. Box 999, Richland, WA 99352 USA

This paper presents work on the formation of metal matrix composites formed from nanocrystalline TiC and Fe-Al. The nano-nano composite powder was produced by milling the commercially available pre-alloyed Fe-Al with ultrafine TiC powder. The high hardness and faceted nature of the TiC particles easily shears the Fe-Al powder particles to an ultrafine size, resulting in the formation of a nano-nano powder mixture. The reduction in particle size of Fe-Al as a function of milling time and TiC content has been investigated, using XRD and TEM. Consolidation of the powder mixtures to retain a nanoscale structure in the composites is in progress.

5:30 PM

Effect of Processing Parameters on Structure and Properties of Nanocrystalline FeCrP Electrodeposits: C. T. Kunioshi[1]; *L. V. Ramanathan*[1]; [1]Cidade Universitaria, Instit. de Pesquisas Engerget. e Nucl., C.P. 11049, São Paulo 05422-970 Brazil

Some nanocrystalline alloys have shown superior corrosion resistance and can be used to protect other materials less resistant in aggressive environments. Several techniques have been used to obtain nanocrystalline alloys and one of these is conventional electrodeposition. Fe-Cr-P alloy deposits were obtained from acid citrate baths using sodium hypophosphite as the source of P. The influence of processing parameters such as nature of complexing agent, bath current density, bath temperature and use of ion selective membranes on deposit characteristics such as crystallite size, composition, and morphology were investigated. The corrosion behavior of steel substrates coated with nanocrystalline Fe-Cr-P deposits under specific plating conditions was evaluated from potentiodynamic measurements in 0.05M sulphuric acid and 0.1M sodium chloride. Nanocrystalline deposits with ~6-7% Cr and average crystallite size of 15 A were obtained under a variety of plating conditions. Deposits obtained at ~100mAcm-2, in the presence of formic acid as complexant and after aging of the bath, were thick, homogeneous and adherent. The electrochemical tests revealed increased corrosion resistance of the nanocrystalline deposit covered surfaces, as compared to the uncoated surfaces.

5:50 PM

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

Electron beam processing offers great technical and economic capabilities of improvement of nanostructured materials. Heating combined with radiation treatment offering by the electron beam processing allows unique changes and modification in nanostructured materials. This paper will consider fundamentals and applications of the electron radiation in processing sintered powder components. The specific effects leading to increase in the component lifetime, wear, and heat resistance will be identified. Possible mechanisms for strengthening and improving the structure and creep performance will be discussed.

TUESDAY AM

Daily Personal Schedule - Tuesday - March 14

Time	Session	Exhibits	Meeting	Other
7:00 am				
7:30 am				
8:00 am				
8:30 am				
9:00 am				
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10:00 am				
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Tutorial Luncheon Lecture
"Ancient Arts of Sword Making"
Daniel Eylon
12:00noon-1:30pm
Convention Center, Canal B

❖❖❖

EPD Luncheon
"Bridging the Gap Between Technology Development & Commercial Applications"
Edward Dowling
12:00noon
Convention Center, Presidential Ballroom - Adams A

❖❖❖

EPD Distinguished Lecturer
"Aspects of Technology Transfer"
Derek Fray
1:45pm
Convention Center, Presidential Ballroom - Jefferson A

12th International Symposium on Experimental Methods for Microgravity Materials Science: Session 1

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

Tuesday AM — Room: Memphis A
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Hideki Minagawa, Hokkaido National Industrial Research Institute, Materials Division, Toyohira-ku, Sapporo 062-8517 Japan

8:30 AM Welcome and Introductions
R. A. Schiffman, R.S. Research Inc., Crystal Lake, Barton, VT 05822 USA

8:50 AM
Halting Convection during Solidification Experiments by Application of a Susceptibility Dependent Magnetic Body Force: *Chris Seybert*[1]; J. W. Evans[1]; W. K. Jones[2]; [1]University of California, Dept. of Matls. Sci. and Mineral Eng., Berkeley, CA 94720 USA; [2]Motorola Inc., 8000 W. Sunrise, Plantation, FL 33324 USA

The role of convection in transporting solute during solidification is well known. Ground-based experiments where such convection is eliminated, e.g. to study solidification in the diffusive limit, have not been successful and several solidification experiments in Earth orbit have been carried out with NASA support. The paper describes an experimental investigation with associated mathematical modeling, that is intended to eliminate convection in ground-based experiments, or to further still convection in microgravity. This is achieved by exploiting a magnetic body force that is dependent on the magnetic susceptibility of the material under investigation and the fact that susceptibilities are temperature dependent (or concentration dependent in the case of solutions). In a sufficient magnetic field (gradient), the reduction in gravitational body force resulting from expansion (or a concentration change) can be balanced by an opposite change in the magnetic body force. Sufficient fields require superconducting magnets on Earth but simple solenoids may suffice in space. Experiments have been conducted in a superconducting magnet at Marshall Space Flight Center to measure velocities in convecting solutions as a function of field strength using particle image velocimetry. A cell with a hot wall at one end and a cold at the other (the other walls being glass) was position a little below the magnetic center of the magnet. A particular current has been identified at which convection is halted, to within the precision of the measurements. Initial results have been on experiments without solidification but similar results with solidification should be available at the time of the conference. Results are interpreted in terms of mathematical models for the magnetic field and the flow. Research supported by NASA.

9:10 AM
The Influence of Gravity on Particle Pushing and Agglomeration during the Freezing of Reinforced Aluminum Alloys: *Reginald W. Smith*[1]; [1]Queen's University, Dept. of Matls. and Metallu. Eng., Kingston K7L 3N6 Canada

Particle reinforced metal matrix composites (PRMMCs) appear to have significant industrial potential, however there are still processing problems associated with them. The main obstacle to good MMC processing is the limited ability to control particle pushing, particle settling, and particle clustering. Experimental results given in the literature have been inconclusive as to estimating the critical velocity, a term which describes the freezing rate above which a particle is engulfed rather than pushed, and for which calculated rates appear to vary over a range of 10^4 when applied to the Al + SiC particle system. A series of experiments were performed on the Russian Space Station MIR in an attempt to further understand these problems in PRMMC processing. A semi-automated furnace apparatus was used on MIR for the particle pushing experiments. The samples consisted of three different PRMMC systems processed at growth rates varying from 1μm/sec to 100μm/sec. Some particle pushing was observed under all of the growth conditions examined. However, during cellular growth, most of the pushing was done laterally so that the reinforcement eventually became trapped between cellular projections. The effects of G-Jitter were also examined and found to increase the critical velocity for engulfment. These results will be discussed in the light of current theory for the interaction of inclusions suspended in the liquid with themselves and advancing solid/liquid interface.

9:30 AM
Containerless Solidification of Zr63Al10Ni11Cu16 Alloy Using a Short Drop Tube: *Takahiro Suwa*[1]; Toshio Suzuki[1]; Kazuto Tokumitsu[1]; Katuhisa Nagayama[2]; [1]The University of Tokyo, Dept. of Metallu., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan; [2]Shibaura Institute of Technology, Dept. of Matls. Sci., 3-9-14, Minato-ku, Tokyo 108-8548 Japan

By ejecting the molten alloy from the nozzle of a silica crucible into a short drop tube filled with helium gas, the small droplets of Zr63Al10Ni11Cu16 alloy were containerlessly solidified. By measuring the crystallization heat of droplets the amorphous fractions were determined for droplets within certain diameter ranges. The obtained relationship between the amorphous fraction and the droplet diameter was compared with those obtained from the classical nucleation theory. The result showed the homogeneous nucleation was attained in the experiments. The fine particles of TiB2, TiC and SiC were added in the alloy and the same kinds of experiments were carried out. The heterogeneous nucleation by the addition of the particles was confirmed.

9:50 AM
Thermodynamic Properties of Bulk Metallic Glass Forming Alloys in the Stable and Undercooled Melt: *R. K. Wunderlich*[1]; H. -J. Fecht[2]; D. S. Lee[3]; S. Glade[3]; W. L. Johnson[3]; [1]Technical University of Berlin, Instit. of Metals Rsch., Hardenbergstr. 36, PN 2-3, Berlin

10623 Germany; [2]University of Ulm, Dept. of Eng., Albert-Einstein-Allee 47, Ulm 89081 Germany; [3]California Institute of Technology, Keck Lab. for Eng. Matls., 138-78, Pasadena, CA 91125 USA

The thermophysical properties of the metallic glass forming alloys $Zr_{65}Al_{7.5}Cu_{7.5}Ni_{10}$, $Zr_{60}Al_{10}Cu_{18}Ni_9Co_3$, $Ti_{34}Zr_{11}Cu_{47}Ni_8$ and, $Zr_{57}Nb_5Cu_{15.4}Al_{10}$ have been investigated in the stable and undercooled melt in a reduced gravity environment. The specimen were processed in the electromagnetic containerless processing device TEMPUS on board spacelab. Specific heat measurements were performed by the method of non contact accalorimetry. Combined with c_p data obtained by heating a glass prepared in earth laboratory by rapid quenching, the thermodynamic functions of these alloys have been evaluated over a large range in temperature allowing a systematic comparison of the thermodynamic factors determining glass forming ability of these alloys. These data are combined with results from ground based viscosity measurements to model temperature-time-transformation curves with surface tension as a free parameter. Comparison with temperature-time-transformation curves measured in a electrostatic levitation device allows estimates of the interfacial tension and the influence of heterogeneous nucleants on the glass forming ability in these alloys.

10:10 AM Break

10:30 AM

Gravitational Effects on Combustion Synthesis of Advanced Porous Materials: *X. Zhang*[1]; J. J. Moore[1]; F. D. Schowengerdt[1]; D. P. Johnson[1]; [1]Colorado School of Mines, Ctr. for Commercial App. of Combustion in Space, Golden, CO 80401-1887 USA

Combustion of self-propagating high-temperature synthesis (SHS) of the B_4C-Al_2O_3, Ti-TiB_x and NiTi-TiB_x systems has been studied with respect to their sensitivity to the SHS reaction parameters of stoichiometry, green density, gasifying agents, ambient pressure, diluents and microgravity in an effort to engineer the required porosity and mechanical properties in these composite materials to meet the requirements of a consumer, such as for the application of bone replacement materials. Gravity serves to restrict the gas expansion and the liquid movement during SHS reaction, as a result, it affects the microstructure and properties of the SHS products. Microgravity testing in KC135 has extended the ability to form porous products. This paper will emphasize the gravitational effects on the SHS process, the microstructure and properties of those porous composites, by comparing the combustion synthesis of porous materials under microgravity (~Og), normal gravity (1g), and higher gravity (~2g).

10:50 AM

The Measurement of Diffusion Coefficient of Molten Eutectic Alloys by Using the Shear Cell under Microgravity: *Tadahiko Masaki*[1]; Satoshi Matsumoto[1]; Shuji Munejiri[1]; Misako Uchida[1]; Hirokazu Kato[1]; Makoto Natsuisaka[1]; Shinichi Yoda[1]; Tomihisa Nakamura[1]; Yasuhiro Nakamura[1]; Naokiyo Koshikawa[1]; Minoru Kaneko[1]; Tomoharu Fukazawa[1]; Yoshito Arai[1]; Kyoichi Kinoshita[1]; Toshio Itami[1]; [1]National Space Development Agency of Japan, Tsukuba Space Ctr., 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan

The measurement of diffusion coefficient of high temperature melts is one of the most important subjects for the utilization of microgravity environment. The shear cell is an advanced and capable method for the diffusion experiment. The shear cell for the high temperature melts was developed and tested due to the sounding rocket TR-IA 7th. The shear cell was applied to the diffusion experiment of molten Ag-Cu alloys and InGaAs semiconductors. The six furnaces were boarded on the sounding rocket. Ten or five different diffusion couples were contained within each furnace. The microgravity experiment was performed successfully in November 1998. The isotope diffusion and interdiffusion coefficient of molten Ag-Cu alloys were determined in the 6 different compositions at three different temperatures. Interdiffusion coefficients of molten InGaAs semiconductor at three different temperatures were also determined. The correlation between the experimental results under microgravity and on ground is discussed.

11:10 AM

Interfacial Temperature Measurements using Seebeck Effect during Directional Solidification of Bi-Sn Alloys: Y. Lian[1]; F. Chen[1]; *R. Abbaschian*[1]; H. C. de Groh[2]; [1]University of Florida, Matls. Sci. and Eng., P.O. Box 116400, Gainesville, FL 32611 USA; [2]NASA, Matls. Div., Processing Sci. and Tech. Brnch., M/S 105-1, Cleveland, OH 44135 USA

The Seebeck technique was used to measure the solid/liquid interfacial undercooling during directional solidification of Bi-lat%Sn. The measurements were conducted under microgravity conditions during the STS-87 flight of the space shuttle Columbia using the MEPHISTO directional solidification facility. The results show that the Seebeck signals depend not only on the interface velocity and growth distance, but also on the structure of the solid behind the interface. The latter, termed the structural Seebeck, is due to the translation of the solidified structure through the temperature gradient zone behind the interface. The structural Seebeck contributions to the solidification measurements were determined by the post-flight analysis of the space-grown samples.

11:30 AM

A Unique Sample Quench Technology for Microgravity Furnaces: *Michael R. Fiske*[1]; April M. Heaton[1]; Kerry D. Moody[1]; Daniel Popok[2]; [1]Morgan Research Corporation, Matls. Sci., 2707 Artie St., Ste. 17, Huntsville, AL 35805 USA; [2]Popok Analytical Consulting & Engineering Inc., 6302 Homestead Rd., N.E., Huntsville, AL 35811 USA

NASA's Microgravity Materials Science research programs require rapid cooling of samples to enhance microstructural analysis, evaluate concentration/impurity profiles, and preserve the geometry of the solid-liquid interface. A sample quench rate of up to 100°C/sec is desired which has been shown to be unattainable using traditional quench systems and Sample Ampoule Cartridge Assembly (SACA) designs. This paper will present the results of a Phase I SBIR project aimed specifically at solving this problem. A unique "Quench-in-Cartridge" (QIC) system has been designed for SACAs in which the quench media (water and liquid gallium have been evaluated) is delivered internal to a metallic containment cartridge. This concept reduces furnace design complexity and allows it to be kept independent of SACA design while achieving significantly higher quench rates. None of the existing or planned quench system designs/methodologies incorporated in either terrestrial or microgravity furnaces offer the simplicity and overwhelming advantages provided by the QIC concept.

Advanced Technologies for Superalloy Affordability: Affordability Technology for Casting

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: K. M. Chang, West Virginia University, Mechanical & Aerospace Engineering, Morgantown, WV 26506 USA; K. R. Bain, GE Aircraft Engines, Cincinnati, OH 45215 USA; D. Furrer, Ladish Company, Cudahy, WI 53110 USA; S. K. Srivastava, Haynes International, Kokomo, IN 46904 USA

Tuesday AM — Room: Canal C
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: David Furrer, Ladish Company, Cudahy, WI 53110 USA; Gerhard Fuchs, University of Florida, Gainesville, FL 32611 USA

8:30 AM Invited

Effect of Trace Elements on Directional Solidification of Superalloys: Yaoxiao Zhu[1]; Langhong Lou[1]; Baiyun Tong[1]; *Changxu Shi*[1]; [1]National Natural Science Foundation of China, 35 Huayuan Beilu, E. Gate, Beijing 100083 China

Technology of low segregation superalloy controlled by trace elements was developed two decades ago at Institute of Metal Research in

China. The principle and experimental effects will be described. The Low segregation technology used for superalloy development is very effective in the following way. (1) New alloys could be used 20-25°C higher then conventional ones at similar composition. (2) Nickel base superalloy that can substitute cobalt base superalloy X-40 with superion properties has been developed by low segregation technology. (3) Low segregation technology can make more superalloys suitable for directional solidification with pronounced improvement of properties at elevated temperatures. (4) The properties of DS superalloy Rene'125 can be improved with low segregation technology without addition of Hf. Experimental facts with explanation will be presented.

8:55 AM Invited

Large Diameter Superalloy Ingots: *Richard L. Kennedy*[1]; Laurence A. Jackman[1]; A. Stewart Ballantyne[1]; Betsy J. Bond[1]; [1]Allvac, An Allegheny Teledyne Co., 2020 Ashcraft Ave., Monroe, NC 28110 USA

In recent years, there has been a very substantial increase in the size and operating temperature of turbine engines for both aircraft and land base power generation. This has led to the demand for larger superalloy components. Processing and current size capabilities are reviewed for several superalloys with reference to metallurgical defects, which can result from the melting process. These can include freckles, dirty white spots and oxide-nitride inclusions. Many of these defects may be detected as ultrasonic indications in finished forgings. Smaller components, made from small diameter forged billet, allow for an in-process inspection and selective removal of such defects at the billet stage. This is not possible for very large parts where the entire ingot is one part. With continuing improvements in the technology of melting superalloys, it has been possible to meet today's requirements for larger parts with the conventional and low cost cast-wrought approach.

9:20 AM Invited

Niobium Segregation in 718 VAR Ingots: *J. Brooks*[1]; J. Krafcik[1]; J. VanDenAvyle[1]; [1]Sandia National Laboratories, P.O. Box 969, MS 9402, Livermore, CA 94550 USA

Segregation of Nb can be a major issue in high quality rotating grade Alloy 718. The tight commercial specifications on Nb content, and on Nb containing macrosegregation defects can only be met if large ingots are melted under closely controlled conditions. To better understand the segregation of Nb and the formation of defects, ingots have been melted over a wide range of processing parameters. Ingots have been longitudinally sectioned and compositionally analyzed using x-ray fluorescence techniques to generate composition maps over the total ingot cross section, as well as in isolated regions at higher spatial resolution. The nature of Nb segregation, its relationship to melting conditions, and the mechanisms by which the segregation occurs will be discussed. This work was supported by the U.S. Department of Energy under contract DE-AC04-94AL85000, the Specialty Metals Processing Consortium.

9:45 AM

Modeling of Grain Movement and its Influence on Microstructure Evolution during Solidification of Superalloy IN718: *B. J. Yang*[1]; D. M. Stefanescu[1]; [1]The University of Alabama, Solid. Lab., P.O. Box 870202, Tuscaloosa, AL 35487 USA

An existing model was further developed to account for the effect of grain movement on the microstructure evolution and the final microstructure of alloys. The continuum model is used in the macro-transport calculation. It considers thermosolutal flow, solidification contraction. The micro-transport model is coupled to a solidification kinetics model that assumes continuous nucleation and calculates grain growth. A distribution function is introduced to handle the non-uniformity of grain size for each control volume. At this time only equiaxed dendrites are considered. The model was used to calculate the solidification of INCONEL 718. The results of calculation can be presented in terms of amount of Laves phase, grain distribution and final macro-segregation. It was demonstrated that grain movement during solidification has a significant effect on the final solidification microstructure.

10:10 AM Break

10:25 AM

Carbides and Grain Defect Formation in Directionally Solidified Nickel-Base Superalloys: *Sammy Tin*[1]; Tresa Pollock[1]; Wendy Murphy[2]; [1]Carnegie Mellon University, Matls. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]General Electric Aircraft Engines, One Neumann Way, Cincinnati, OH 45215 USA

Demands for increased efficiency in turbine engines have led to the development of multi-component single crystal nickel-base superalloys. As levels of refractory alloying additions have gradually increased to improve high-temperature creep properties, grain defect formation during directional solidification has become an increasingly important problem. Recent solidification experiments have been conducted to investigate the relationship between alloy chemistry and the mechanism(s) which lead to grain defect formation. Carbon additions (up to 0.1wt. %) have a strong beneficial effect in reducing the number of grain defects. While it is known that carbon additions result in the precipitation of MC carbides, the interactions of these carbides with the defect generating mechanisms are not well understood. The role of carbides has been studied via segregation measurements coupled with DTA for a large set of alloys. The results of these experiments will be discussed with respect to the possible mechanisms by which carbon influences solidification.

10:50 AM

The Hot Deformation Modeling of As-Cast High-Strengthened Superalloys: *Zhengdong Long*[1]; Jingyun Zhuang[1]; Bo Deng[1]; Ping Lin[1]; *Zengyong Zhong*[1]; [1]Central Iron & Steel Research Institute, Dept. of Superalloys, 76 Xueyuan Nan Rd., Haidian District, Beijing 100081 PRC

The high-strengthened superalloys have harsh hot workability because of their high flow stress, low deformation ductility and narrow available deformation temperature range. So, the controlling of hot deformation parameters is very difficult and important. In the conventional manufacturing routes, a great amounts of trial-and-error steps must be taken to get suitable processing parameters, so, it is very worthy to develop a effective hot deformation modeling to predict the deformation behavior. In this paper, the hot deformation behaviors were examined, and the hot deformation modeling was built up based on the test results. First, the mathematical relationship between flow stress and deformation parameters, especially the peak flow stress vs hot deformation parameters were built up. Second, the mathematical relationship between deformation ductility and deformation parameters was also built up. Generally, these equations not only fit to the experimental results accurately but also reflect the physical phenomena occurring during the hot deformation.

11:10 AM

A New Technology of Superalloy Surface Metallurgy–Double Glow Plasma Surface Alloying: *Xishan Xie*[1]; Xu Zhang[1]; Jianxin Dong[1]; Zhong Xu[2]; [1]University of Science and Technology, Dept. of Matls. Sci. and Eng., 30 Xueyuan Lu, Beijing 100083 China; [2]Taiyuan University of Technology, Taiyuan 030024 China

The Double Glow Plasma Surface Alloying Technology (DGPSA) is an advanced new surface metallurgy technology. It was invented in China by Zhong Xu and patented in China, Canada, Japan, United Kingdom, United States and so on. The premium quality plasma alloying layers with special properties can be formed on the common material surfaces. In this paper the surface alloying layer similar to nickel-base superalloy Inconel625 is obtained on the surface of three kind steels (low carbon steel, pure iron and stainless steel 304) by using DGPSA. The chemical composition and microstructure of surface layers were analyzed by SEM, EDAX and XRD and show that the alloying layers consist of matrix and several precipitates which depends on the local chemical composition. The corrosion tests in 20%H2SO4 and 20%HCl solution show that the corrosion rates of surface alloying layer on pure iron are equal to that of nickel-base superalloy Inconel625 and one order of magnitude lower than that of AISI 304 stainless steel.

11:30 AM
Multizone Sonic Inspection of Ingot Metallurgy Superalloy Billet Material: *T. Reay*[1]; H. Waldal[1]; B. Zirbel[1]; D. Furrer[1]; [1]Ladish Company Inc., P.O. Box 8902, 5481 S. Packard Ave., Cudahy, WI 53110-8902 USA

Quality level and flaw content are key factors for design requirements and component lifting issues for aircraft turbine engine manufacturers. Cast and wrought nickel-base superalloys are used for many demanding applications in turbine engines, such as the low and high pressure turbine disks. Inspection of these critical components is of significant importance. An effort has been undertaken to develop a new method of ultrasonic inspecting cast and wrought superalloy material to allow for a more complete assessment of input material in the billet stage prior to extensive value-added operations being performed. The utilization of multizone ultrasonic inspection has proven to be effective and beneficial for improving ultrasonic inspection of cast and wrought superalloy billet material.

Aluminum Reduction Technology: Process Control/Anodic Phenomena

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

Tuesday AM — Room: Sewanee
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Bernd Rolofs, Hoogovens Aluminium Huttenwerk GmbH, Voerde D-46549 Germany

8:30 AM Invited
Overview of Process Control in Reduction Cells and Potlines: *Pierre Homsi*[1]; Jean-Michel Peyneau[1]; Michel Reverdy[1]; [1]Aluminium Pechiney, LRF-BP 114, Saint-Jean-de Maurienne 73303 France

Hardware and software developments have enabled the control of reduction cells to evolve dramatically over the last two decades. Process computers allow the control of cells as well as the technical management of modern potlines. The objectives of cell automatic process control are reviewed. They normally encompass alumina feeding, cell resistance regulation and bath chemistry and cell heat balance control. Routine operations on the cells are also accounted for. Tools are made available to assist potline supervisors in the optimization of operations, quick diagnosis of process changes, correction of abnormal and exceptional situations on individual cells or potlines as well as scheduling of work on the cells. New developments in cell and potline control provided by the ALPSYS system are presented.

9:00 AM
The Effects of Process Operations on Smelter Cell Top Heat Losses: *M. Derek Gadd*[1]; Barry J. Welch[1]; Tony D. Ackland[2]; [1]The University of Auckland, Chem. and Matls. Eng., Private Bag 92019, Auckland New Zealand; [2]New Zealand Aluminium Smelters Limited, Tech. Dev., Private Bag 90110, Invercargill, New Zealand

With modern smelting cells operating to a fine heat balance the impact of various operating and cell condition changes on the heat balance needs to be examined more closely. A study of the changes in top heat losses has been made by monitoring duct gas flow rate, duct gas and hood temperatures while regularly measuring ambient air and electrolyte temperature. With a continuous recording of cell process event messages, correlations between heat loss and the various routine operations and process changes have been possible. Particular attention has been placed on anode changing, metal tapping, alumina feeding and cell anode effects. The differences between individual cells have also been studied and it has been found these differences have a direct effect on the individual cell conditions.

9:25 AM
Anodic Phenomena–Observations of Anode Overvoltage and Gas Bubbling during Aluminium Electrolysis: *Xiangwen Wang*[1]; Alton T. Tabereaux[1]; [1]Smelter Technology Laboratory, Corp. Rsrch. and Dev., Reynolds Metals Co., 4276 Second St., Muscle Shoals, AL 35661-1625 USA

Anode overvoltage and the gas formation-release process (gas bubbling) are two important anodic phenomena during the electrolytic production of aluminum in Hall cells. Comprehensive electrolysis studies and physical modeling of these anodic phenomena including bubble formation and release observations, overvoltage, and their relations to anode size and configuration have been reported in the literature. But, these anode bubbling studies have been conducted mainly with two extreme anode scales; 1) extremely small (low amperage) anodes in laboratory cells 2) large industrial (high amperage) anodes in production cells. This paper reports on electrolysis studies conducted with an intermediate scale (15.2 cm diameter) anode in a laboratory cell to provide experimental data and explanations to bridge the results obtained from previous studies using the two extreme anode scale cases with bubble formation. Information on bubble size and the formation-release processes were obtained by measuring the bubble formation-release process frequency as well as the voltage magnitude from bubble growth, coalescence and release from the anode during a continuous 72 electrolysis test. The impact of gas bubbles on increasing the cell resistance, the anode overvoltage and gas bubble phenomena as a function of anode consumption (shape change) were observed and recorded. Variation in anode current density was investigated. The results provide an insight in understanding the relationship between bubbling phenomena and anode overvoltage variations during aluminium production.

9:50 AM
On the Anode Effect in Aluminium Electrolysis: *Jomar Thonstad*[1]; Torstein A. Utigard[2]; Helmut Vogt[3]; [1]Norwegian University of Science & Technology, Dept. of Matls. Tech. & Electrochem., Trondheim 7491 Norway; [2]University of Toronto, Dept. of Matls. Sci., 184 College St., Toronto, Ontario M5S3E4 Canada; [3]University of Applied Sciences, Berlin D-133353 Germany

Anode effects are detrimental in that they result in reduced energy efficiency and cause emissions of CF4 and C2F6. With prospects of future CO2, taxes, the emissions of these greenhouse gases may become costly. With a CO2 tax of 15 US$ per tonne, each anode effect minute per day per cell will increase the production cost by about 1.2%. Research work related to anode effects has been reviewed and analyzed. Although it is well known that anode effects occur when the alumina content becomes too low to maintain normal electrolysis, the mechanism of the initiation of the anode effect as well as the current distribution and gas behavior in industrial cells are still not fully understood. The paper concludes by analyzing various methods which may be used to decrease the frequency and duration of anode effects in prebake as well as Soderberg cells.

10:15 AM Break

10:25 AM
An Electroanalytical Study of Electrode Reactions on Carbon Anodes during Electrolytic Production of Aluminium: Hongmin Zhu[1]; *Donald R. Sadoway*[1]; [1]Massachusetts Institute of Technology, Dept. Matls. Sci. and Eng., 77 Massachusetts Ave., Rm. 8-109, Cambridge, MA 02139-4307 USA

The electrode reactions occurring on carbon anodes during the electrolytic production of aluminum are being studied in a laboratory-scale cell. Electroanalytical techniques such as voltammetry and electrochemical impedance spectroscopy are being used to determine the mechanisms associated with the electrochemical reactions at the anode. The work is motivated by the belief that an understanding of the elementary reactions occurring under a variety of process conditions will enable the development of strategies for reducing the intensity of perfluorocarbon (PFC) emission. The research is sponsored jointly by the Aluminum Association and the U.S. Environmental Protection Agency.

10:50 AM
Analysis of Excess AlF_3-Harmonization in Hydro Aluminium: *Lorentz Petter Lossius*[1]; Helge Hoie[1]; Hanne Hoel Pedersen[1]; Trygve Foosnaes[1]; [1]Hydro Aluminium Technology Centre Ardal, P.O. Box 303, Ovre-Ardal N-6881 Norway

When comparing process data from different aluminium reduction plants it is important to ensure that the results really are comparable. This presentation describes the harmonisation of the analysis of bath acidity in Hydro Aluminium Metal Products (HAMP). Each step from sampling to instrument calibration and analysis was scrutinised and the critical steps identified. The most complex part was establishing new bath standards, and some problems connected with the accuracy of the standards are addressed. The presentation also outlines how the individual steps in the analysis contribute to the standard deviation in the reported results and the effect of using different equipment in the participating laboratories. It is shown that, although the instrument precision can be less than O.O5wt%, other steps in the analysis contribute to a real standard deviation in the reported results of 0.20 to 0.45wt% depending on equipment and methods. At present round robins are conducted annually to ensure that the harmonisation is preserved. After each round robin the results are analysed and used as feedback to improve the next round.

11:15 AM
Distribution Aspects in Reduction Line Control Systems: *Carsten M. Ritter*[1]; Luiz F. R. Neves[1]; Leonel V. M. Ivo[1]; José H. S. Trigueiro[1]; [1]ATAN Automation Systems, Aluminium Div., Rua Pernambuco 353, Belo Horizonte, Minas Gerais 30130-150 Brazil

This paper analyzes the different degrees of distribution adopted by aluminium reduction line control systems. The concepts, approach, and results to implement centralized, semi-distributed and totally distributed automation topologies are described. The pros and cons for some architectures are discussed. A framework for topologies comparison is then presented, providing a valuable decision tool when implementing, replacing, expanding or revamping the reduction line control system.

Automotive Alloys 2000: Applied

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Subodh K. Das, University of Kentucky College of Engineering, Center for Aluminum Technology, Lexington, KY 40506-0043 USA

Tuesday AM — Room: Knoxville A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Subodh K. Das, University of Kentucky, College of Eng., Lexington, KY 40232 USA; Andy M. Sherman, Ford Motor Company, Dearborn, MI 48121-2053 USA

8:30 AM
Aluminum Tailor-Welded Blanks for Automotive Applications: *P. A. Friedman*[1]; G. T. Kridli[2]; [1]Ford Research Laboratory, Manufact. Sys. Dept., P.O. Box 2053, MD 3135/SRL, Dearborn, MI 48121-2053 USA; [2]University of Michigan-Dearborn, Dept. of Indust. and Manufact. Sys. Eng., 2300 Eng. Complex, Dearborn, MI 48128 USA

The push to manufacture lighter-weight vehicles has forced the auto industry to look to alternative materials than steel for vehicle body structures. Aluminum is one such material that can greatly decrease the weight of vehicle body structures and is also consistent with existing manufacturing processes. As in steel structures, cost and weight can be saved in aluminum structures with the use of tailored blanks. These blanks consist of two or more sheets of dissimilar thicknesses and/or properties joined together through some type of welding process. This enables the design engineer to "tailor" the blank to meet the exact needs of a specific part. Cost savings can be gained by the elimination of reinforcement parts and the stamping dies used to manufacture them. Weight savings can be attained based on the fact that one thicker piece is more efficient than a welded structure and therefore can allow for down-gauging of parts. While TWBs offer both potential weight and cost benefits, the continuous weld-line and thickness differential in TWBs can often result in difficulty in stamping. This problem is more severe in aluminum because of its limited formability as compared with typical drawing-quality steels. Additionally, welding of steel TWBs tends to increase the strength of the weld material which helps prevent failure in the weld during forming. Aluminum TWBs do not experience this increase in strength and therefore have a greater tendency to fail in the weld. In this study, several aspects of TWBs manufactured from 6111-T4, 5754-O and 5182-O aluminum alloys were analyzed and compared with those of a more conventional steel TWB. The effect of gauge mismatch on the formability of these blanks is discussed as well as the overall potential of these blanks for automotive applications.

8:50 AM
Surface Roughness Development in 6022 and 6016 Aluminum Alloy Sheet and its Relation to Microstructure Analyzed with the Disorientation Correlation Function: *Anthony D. Rollett*[1]; Paul S. Lee[1]; Brent L. Adams[2]; Henry R. Piehler[1]; Hasso Weiland[3]; [1]Carnegie Mellon University, Dept. Matls. Sci. & Eng., 3327 Wean Hall, 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; [2]Brigham Young University, Dept. Mech. Eng., Provo, UT USA; [3]Alcoa Technical Center, PA 15069 USA

An important aspect of sheet forming is the development of surface roughness as plastic strain accumulates. Such roughness may decrease the usefulness of the formed part or even limit the formability. Taking the crystallographic nature of slip as a basis for investigation, orientation imaging microscopy has been used to characterize sections through samples of 6022 and 6016, both in the T4 condition, deformed in plane strain tension. In order to quantify strain heterogeneities and correlations in texture that are not readily apparent in conventional images, the disorientation correlation function (DCF) has been used. The DCF calculates the average misorientation as a function of distance and direction. The results indicate the presence of long-range orientation correlations extending over more than five grain diameters. Substantial changes in the DCF are observed as a function of strain level. At large strains, for example, grains lying along the extension direction are more highly correlated than in any other direction. When bands observed in the DCF maps are compared with surface ridging, similar spacings are found. Also, a comparison of sheets with different surface roughening susceptibilities suggested that stronger texture correlations are observed in materials with greater roughening tendencies. These observations of long range orientation correlation may have implications for strain localization in the deformation of sheets. In samples of 6016 treated to develop the ridging phenomenon, the DCF reveals a complex banded structure. An interesting result of the sheet tensile tests is that the fracture angle follows the Hill theory as the angle from the rolling direction increases except that, when the tensile axis is within a few degrees of the transverse direction, the fracture path becomes parallel to the ridges, i.e. the rolling direction. This work was primarily supported by the Office of Energy Research, US Department of Energy under grant number DE-FG02-96ER45601.

9:10 AM
Precipitation Behavior in Grain Boundary Regions of Modified 5083 Aluminum Alloys: *M. C. Carroll*[1]; P. I. Gouma[1]; M. J. Mills[1]; G. S. Daehn[1]; B. R. Dunbar[2]; [1]The Ohio State University, Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43201 USA; [2]Century Aluminum Corporation, Ravenswood Operations, P.O. Box 98, Ravenswood, WV 26164 USA

The susceptibility of 5000 series alloys to stress corrosion cracking (SCC) has been largely attributed to the precipitation of Mg (beta phase) along grain boundaries. The alloys of this series that contain levels of Mg higher than about 3.5 wt% have been specifically singled out as the most susceptible stress corrosion cracking as a result of Mg enrichment and subsequent Mg-based phase formation in grain boundary regions. The precipitation behavior of various alloys with near-5083 composition will be evaluated. Emphasis will be placed on additions of elements that produce a precipitate microstructure that may be less susceptible to SCC as a result of limiting beta phase (Al8Mg5)

formation. Particular attention will be paid to analyzing local precipitation changes in the regions adjacent to grain and subgrain boundaries. Information will be presented based on transmission electron microscopy (TEM) images as well as fine-probe energy dispersive spectroscopy (EDS) composition profiles.

9:30 AM

Finite Element Modeling of Creep Relaxation in Bolted Joints of Magnesium Castings: *Ken I. Johnson*[1]; Russell H. Jones[2]; Stan G. Pitman[3]; Eric A. Nyberg[3]; [1]Pacific Northwest National Laboratory, Energy Div./Appl. Mech. Grp., P.O. Box 999, Richland, WA 99352 USA; [2]Pacific Northwest National Laboratory, Matls. Resources/Structural Matls. Dev., P.O. Box 999, Richland, WA 99352 USA; [3]Pacific Northwest National Laboratory, Matls. Resources/Matls. Processing, P.O. Box 999, Richland, WA 99352 USA

Magnesium die-castings are of interest for automotive transmission housings because of the potential to save up to 30% of the weight of traditional aluminum castings. However, a major drawback is the poor creep resistance of magnesium casting alloys at elevated-temperatures. This can result in loss of bolt load retention (BLR) and subsequent leakage of bolted flange joints. There is a current need to develop modeling tools that correlate fundamental creep data with the BLR of actual joint designs. This stems from the ease of creep testing and the relative abundance of creep data compared with BLR tests. Both constant load tensile creep tests and simplified BLR tests are currently performed. This paper describes material testing and finite element modeling that was performed to demonstrate the correlation between these different forms of creep data for ZAC 8506. The goal of this research is to develop an analytical tool with which to optimize bolted joints in magnesium castings. Material constitutive models based on constant load creep tests where input to the MARC finite element code to simulate actual bolt load retention tests. The models include the effects of bolt preload, surface contact, creep relaxation, thermal expansion, and yielding due to thermal softening. Results are presented that compare the model predictions with the results of actual bolt load retention tests. The models are also used to show the relative importance of initial preload, creep, and yielding on the retained bolt load.

9:50 AM

High Temperature-Creep Resistant Magnesium Alloys: Advances in the Use of Thixomolding for Automotive Components: *Eric Arthur Nyberg*[1]; Russell H. Jones[1]; Stan G. Pitman[1]; Daniel J. Edwards[1]; Robert D. Carnahan[2]; Raymond F. Decker[2]; [1]Pacific Northwest National Laboratory, Matls. Resources, Battelle Blvd., P.O. Box 999, Richland, WA 99352 USA; [2]Thixomat Inc., 620 Technology Dr., Ann Arbor, MI 48108 USA

High temperature ZAC alloys (Mg-Zn-Al-Ca) have attracted interest due to their reasonable combination of cost, forming/processing characteristics and mechanical properties. In particular, the improved high temperature creep resistance of these alloys makes them candidates for automotive applications where the creep strength of magnesium is limited because the operating temperatures exceed approximately 125°C. Most research on the ZAC alloys has focused on comparing the die cast properties of the ZAC alloys to other alloys currently used in die cast production (AZ91D, AM50A, AM60B, AS21 and AS41). This project is part of the Northwest Alliance for Transportation Technologies (NATT) program which is funded through DOE's Partnership for New Generaton Vehicles (PNGV) program. The aim of the NATT program is to address specific transportation goals through cooperative R&D with suppliers. In this study, Pacific Northwest National Laboratory and Thixomat Inc. have partnered to investigate the high temperature properties of ZAC8506 (8%Zn-5%Al-0.06%Ca) comparing standard die casting and the semi-solid forming process referred to as Thixomolding. The objective of the work was to better understand the influence of lower part forming temperatures, from the semi-solid, laminar flow process (Thixomolding), on void/pore fraction, tensile strength and high temperature creep behavior as compared to die casting. As has been shown for other alloy systems, the fraction of pores is reduced and the mechanical properties are improved when formed by Thixomolding. This work is continuing to evaluate alloys produced by Thixomolding that are traditionally not die cast, such as magnesium metal matrix composites and the ZK alloys.

10:10 AM Break

10:20 AM

The Influence of Reversed Bending on the Formability of Aluminum Sheet: *Karole J. Lian*[1]; Armand J. Beaudoin[1]; Peter A. Friedman[2]; [1]University of Illinois at Urbana-Champaign, Dept. of Mech. and Industrial Eng., 1206 W. Green St., Urbana, IL 61801 USA; [2]Ford Research Laboratory, Dept. of Manufacturing Sys., P.O. Box 2053, MD 3135/SRL, Dearborn, MI 48121-2053 USA

The forming limit diagram (FLD) provides a useful engineering tool for the development and analysis of stamping operations. It is predicated on the notion that the deformation history will follow a proportional strain path. To extend the utility of the FLD, recent efforts have studied the effect of pre-straining the sheet in a specific mode of deformation prior to FLD determination. In sheet forming practice there is often significant deformation imparted on the sheet before contact with the forming punch. Two specific examples of this type of deformation are reversed bending occurring during tension-leveling operations and in material traversing a drawbead. The objective of this current research is to study the effect of through-thickness straining on the subsequent formability of aluminum sheet. A test apparatus has been designed to impart reversed bending to sheet of width sufficient for extended limiting dome height tests. Experiments were performed on both AA5754 and AA6111 sheets after bending, and the results were compared to the original sheet stock to detect changes in formability. The magnitude of through-thickness plastic pre-strain is assessed through X-ray measurement of crystallographic texture and residual stress.

10:40 AM

Formability of Aluminum Extrusions during Hydroforming Applications: *Mark Tower Smith*[1]; Richard William Davies[1]; Mohammad A. Khaleel[1]; [1]Battelle PNNL, Matls. Processing, 902 Battelle Blvd., P.O. Box 999 MSIN:P8-35, Richland, WA 99352 USA

Tubular hydroforming of automotive components is receiving significant attention as a manufacturing method capable of reducing part count, component weight, and overall manufacturing costs. Hydroforming is extremely attractive as a method to produce complex shaped components from tradition tube and extrusion materials. U.S. automotive companies and suppliers are currently manufacturing many steel tubular hydroformed components. However introducing aluminum alloys to hydroforming applications is consistent with the current automotive company and government initiatives to reduce the overall weight and emissions of automobiles. The current work evaluates and presents the formability of aluminum alloys during hydroforming applications, including studies conducted to systematically vary the application of axial end feed and internal pressure during the hydroforming experiments. The current work also experimentally investigates the anisotropic yield locus of the extruded aluminum tubing, and theoretically evaluates the effects the manufacturing inherent anisotropy has on the formability of aluminum alloys extrusions.

11:00 AM

Thermo-Mechanical Fatigue of a Cast 319 Aluminum: *John Victor Lasecki*[1]; Huseyin Sehitoglu[2]; John E. Allison[1]; [1]Ford Motor Company, Matl. Sci. Dept., Scientific Rsch. Lab., MD 3182 SRL, Dearborn, MI 48121-2053 USA; [2]University of Illinois, Mech. and Industrial Eng., 144 Mech. Eng. Bldg., 1206 W. Green St., Urbana, IL 61801 USA

The majority of automotive engine cylinder heads are cast aluminum. The trend is for aluminum cylinder heads to be used in increasingly more demanding operational environments which necessitates a knowledge of the material behavior under cyclic thermo-mechanical loading conditions. The purpose of this study is to determine the extent to which existing constitutive and life models can be extended to loading situations in which the strains are predominantly compressive. TMF experiments were carried out under mechanical strain control on solid cylindrical specimens of 319 Al. The TMF cycle was selected so as to simulate the critical conditions that exist in the surface volume of a cylinder head during a start-up and shut down

operation. Out of phase loading dominates the cylinder head duty cycle, therefore experiments were conducted with the maximum temperature coinciding with the maximum compressive strain. Tests were performed in air with the temperature cycled between 50 and 250°C. In this study the role of selected mean stresses and mechanical strain amplitudes were used to characterize the TMF behavior of cast aluminum. Results will be compared with recently developed constitutive and durability models for cast 319 Al. Fractography of failed samples was conducted to understand the crack initiation mechanism.

11:20 AM
Hot-Tearing Analysis of a Solidifying Die Cast Part: *Yimin Ruan*[1]; Jamal Righi[1]; [1]Alcoa Inc., Shaped Casting Platform, Alcoa Tech. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA

Aluminum alloys are susceptible to cracking during solidification (hot-tearing). The susceptibility largely depends on the alloy composition. Process conditions and the geometry of a casting also have significant influence on hot-tearing. Hot-tearing occurs in all castings, such as wrought ingot, die cast part, foundry cast part, etc. This paper presents a hot-tearing analysis of a solidifying die-cast part for an alloy which has a higher strength and more susceptible for hot-tearing. Due to the characteristics of the alloy, higher inelastic strains and stresses develop in the cast part and cause the cast material to crack. In this paper, the thermomechanical behavior of the alloy during solidification after the molten metal is injected into the die cavity is studied and the conditions to prevent cracking at the casting surface are determined. A hot-tearing criterion is also developed in this paper in order to reliably predict and prevent casting cracks during solidification.

11:40 AM
Casting Simulations for Semi-Solid Metal Forming Process: Yie Zhao[1]; *Jamal Righi*[1]; [1]Alcoa Inc., Shaped Casting Platform, Alcoa Tech. Ctr., 100 Technical Dr., Alcoa Center, PA 15069 USA

Forming alloys in the semi-solid state is a relatively new but potentially high rewarding method for producing near net-shaped castings. The production of high quality components requires detailed understanding of die filling and solidification pattern. To achieve this, numerical simulations have been adopted as a process design, development and optimization tool. This paper presents a filling analysis of a hat-shaped die, which is a demonstration part chosen by an Alcoa development program. Two software packages Flow-3D and APECS, using single phase simulation approach which employ governing equations for a Newtonian fluid and introduce rheological behavior for semi-solid slurries thought viscosity models, were used to perform the casting simulations. Simulation results showed the hat-shaped die could be filled without any difficulties from A356 slurry. The initial casting trials confirmed the simulation predictions. In addition, the effects of thixotropic behavior of material and process conditions on filling patterns were studied thoroughly.

Cast Shop Technology: Casting and Solidification

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

Tuesday AM — Room: Mississippi
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Martin J. Ekenes, Hycast, Otis Orchards, WA 99027-0603 USA

8:30 AM Introductory Remarks

8:35 AM
Automation & Data Acquisition: Tools for Safer & Easier Casting: *Patrick Pouly*[1]; Etienne Caloz[1]; [1]Alusuisse Technology & Management Limited, Tech. Ctr., Chippis 3965 Switzerland

Automation has proven to be a valuable tool since its introduction in the casthouse in the 1980's. One of the main arguments justifying its introduction is the drastic reduction in safety hazard, as most parameters can be kept under tight control. After a rapid presentation of the structure of a modern automation, this paper will show the potential lying within a powerful data acquisition system. Such a system can be linked to the automation and used for analyzing casting conditions. A series of practical examples will be given to illustrate how a data acquisition system can be used to localize the phenomenon that lead to a premature end of a cast.

9:00 AM
Chemical, Physical & Mechanical Properties of Today's Casting Tips for Aluminum Sheet Casting: *Jason M. Canon*[1]; [1]Thermatex Corporation, Rsch. & Dev., P.O. Box 125, Newton Falls, OH 44444 USA

A program has been instituted to measure pertinent properties of asbestos free casting tips currently used in the twin roll casting process and are now available in the global market. Casting tips are an integral part of the casting process and can directly affect the quality of the aluminum and impact the continuous casting operation. Measured properties included typical chemistry, shot content, thermal conductivity, density, modulus of rupture, compressive strength, flexural strength, shrinkage, water absorption and the measurement of organic/inorganic binders. Properties were also measured throughout the tip to determine the consistency of the product and it was determined variability exists within the casting tip. A method of improving the inconsistencies were evaluated and documented. Efforts were made to correlate calculated laboratory test results to product performance in the field.

9:25 AM
A Potential Casting Pit Coating Material: *George J. Binczewski*[1]; [1]S.C. Systems, P.O. Box 6154, Moraga, CA 94570 USA

Finding an acceptable replacement for the familiar, but environmentally unfriendly Tarset coating has been the object of an aluminum industry funded, and ongoing research project for several years. The need to enhance the safety performance of DC casting pits involving potential contact surfaces of molten aluminum and water is continuous. There are many favorable attributes associated with utilizing rubber sheeting, which is organic, as the coating material. These involve low costs, multiple availability, ease of application, exclusion of cure times, simplicity of repair, long service life, and vibration dampening characteristics. Experiences and performance evaluations of rubber sheeting involving situations of contact with molten aluminum and water are described and reported.

9:50 AM
Study of Hot-Tearing in Solidification of Aluminum Alloys Via an Acoustic Emission Technique: *Xiaojin Li*[1]; *Celil A. Aliravci*[1]; Mihriban O. Pekguleryuz[1]; Michel Bouchard[1]; [1]University of Quebec in Chicoutimi, Dept. Applied Sci., Alcan-UQAC Chair in Solidification and Metallu. of Al, 555 Univ. St., Chicoutimi, Quebec G7H 2B1 Canada

The hot-tearing tendency of AA1050 aluminum alloy solidifying in a ring-shaped test-casting mold was investigated in real-time and in situ via an acoustic emission (AE) and a temperature-data acquisition and analysis (TDA) techniques. In lab-scale experiments, AE signals were acquired simultaneously with temperature monitoring by the use of an inserted steel wave guide and a K-type thermocouple placed in the final freezing zone. These measurements have provided a time definition for AE signal characteristics and a definite time-temperature reference-frame for solidification and defect formation events. Hence, a technique of AE signal analysis combined with computer-aided cooling-curve analysis (CA-CCA) was developed. It was shown that the AE technique is effective in detecting hot-tearing occurrences, and can be further developed for mechanistic studies of hot-tearing, and possibly as a quality control tool for the casting industry. Grain refinement was carried out in AA1050 Al alloy to verify the effect of grain refinement on hot-tearing tendency. The experimental results showed that grain refinement can considerably reduce the hot-

tearing tendency in the AA1050 alloy. Fracture surface analysis was conducted for all samples under a scanning electron microscope. Typical exposed hot tear surfaces with free dendritic structure were observed and analyzed.

10:15 Break

10:20 AM

Laboratory Study of Cast Surface Structure Evolution: I. Mold Contact Stage: *Douglas A. Weirauch*[1]; Lawrence J. Martonik[1]; Alvaro Giron[1]; Donald P. Ziegler[1]; Men Glenn Chu[1]; [1]Alcoa, Alcoa Tech. Ctr., 100 Tech. Dr., Alcoa Center, PA 15069-0001 USA

A small-scale, aluminum caster is described which permits the careful control of many of the important continuous casting process parameters which affect the early stages of solidification. The immersion caster is applied to high-purity aluminum as a first step in quantifying the effect of key process variables on cast surface features and subsurface microstructure. The observed characteristics of surface features are interpreted on the basis of heat transfer and shell growth kinetics. The effects of casting speed, melt superheat, mold surface roughness, and aluminum alloy solidification range are discussed.

10:45 AM

Laboratory Study of Cast Surface Structure Evolution II. Macro Air Gap Stage: *Makoto Morishita*[1]; Kiminori Nakayama[2]; Kenji Tokuda[3]; Katsuyuki Yoshikawa[1]; [1]Kobe Steel Limited, Process Tech. Rsch. Lab., 5-5, Takatsukadai 1-Chome, Nishi-ku, Kobe, Hyogo 651-2271 Japan; [2]Kobe Steel Limited, Mech. Eng. Rsch. Lab., 5-5, Takatsukadai 1-Chome, Nishi-ku, Kobe, Hyogo 651-2271 Japan; [3]Kobe Steel Limited, Aluminum Rsch. Dept., Moka Plant, 15 Kinugaoka, Moka, Tochigi 321-4367 Japan

At macro air gap stage of aluminum casting process, an exudation is occurred by surface remelting because of high thermal resistance of the air gap between solidification shell and mold. This exudation causes some problems such as segregation, uneven structure and so forth. The air gap tester, which is possible to create the air gap and the exudation on the surface experimentally, is applied to molten aluminum alloy in order to clarify the effect of casting variables on the exudation during solidification. The effects of grain size, melt superheat, mold surface roughness and aluminum alloy solidification range are discussed.

11:10 AM

Computer Simulation of Metal Feeding System Used in Twin Roll Casting: *Kemal Sarioglu*[1]; *Murat Dundar*[1]; *Gungor Yildizbayrak*[1]; [1]ASSAN Aluminum Plant, Tech. Coordination Div., Tuzla, Istanbul 81700 Turkey

Twin roll casting has been accepted worldwide as a cost-effective method of producing wide variety of Al products. This process converts molten aluminium alloys into coiled sheet. The uniform distribution of metal to the water cooled roll is accomplished with a ceramic fiber tip. Internal buffling configuration affects this uniform distribution causing defect free sheet production. In this study, the placement of the buffles in the tip having different widths has been investigated by using computer simulation. Uniform flow distribution at the exit of the tip was attained with proper arrangement of the buffles within the tip body. Practical applications verified that unperturbed flow distribution over the whole width of the tip prevented void formation at certain location of the sheet and insufficient crown formation. The goal of this research is also to find sufficient tip geometry that enables continuous metal feeding for thinner gauges.

11:35 AM

The Effect of Casting Parameters on Twin Roll Cast Strip Microstructure: A. Soner Akkurt[1]; *Murat Dundar*[1]; Seda Ertan[1]; Erol Ozden[1]; Kemal Sarioglu[1]; Gungor Yildizbayrak[2]; Shaun Hamer[3]; Chris Romanowski[3]; [1]ASSAN Aluminum Works, E-5 Karayolu 32.Km, Tuzla, Istanbul 81700 Turkey; [2]Kibar Holding A.S., E-5 Karayolu 32.Km, Tuzlal, Istanbul 81700 Turkey; [3]FATA Hunter Inc., 6147 River Crest Dr., Riverside, CA 92507 USA

Twin roll casting is used throughout the aluminum industry to produce reroll for a variety of fin and foil products. The recent trend has been to reduce the gauge at which these casters operate to <3mm. Difficulties in industrially processing this thin gauge cast strip have been reported; in particular, maintaining satisfactory final fin and foil product ductility. Assan Aluminum, in cooperation with FATA Hunter, recently completed an extensive series of casting trials to characterize the effects of casting parameters, including casting gauge, speed and tip setback on the microstructure of various fin and foil alloys. The samples were examined using optical and electron microscopy, in combination with mechanical properties, micro-hardness and resistivity measurements. This analysis was used to optimize the casting, rolling and annealing practice for each alloy. This paper presents the initial results from this study and describes general trends correlating casting parameters to microstructure.

Cyclic Deformation and Fatigue of Materials; A Symposium in Honor of Professor Campbell Laird: Cyclic Deformation and Mechanism (II)

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

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

Tuesday AM — Room: Canal A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: J. D. Embury, McMaster University, Matls. Sci. & Eng., Hamilton, Ontario Canada; P. Peralta, Arizona State University, Dept. of Mech. & Aerospace Eng., Tempe, AZ 85287-6106 USA

8:30 AM

Constriction Energy in the Presence of an Interstitial Field: *Huseyin Sehitoglu*[1]; Scott Andrews[1]; Ibrahim Karaman[1]; [1]University of Illinois, Dept. of Mech. and Industrial Eng., Urbana, IL 61801 USA

It has been widely known that both solute concentration, ie. frictional effects, and stacking fault energy influence the degree of cross-slip and slip planarity in fcc alloys (for details see Hong and Laird, 1990). Cross-slip is preceded by constriction of two partial dislocations. A model is proposed for the energy required to form a constriction from two parallel partial dislocations as a function of stacking fault energy, interstitial concentration and atomic size misfit. In the analysis, the local stress fields are calculated due to either interstitial or substitutional solute concentrations around the partial dislocations. The cross-slip is curtailed due to interaction of interstitials with the edge components of the partials. The atomic size misfit influences the local interstitial concentration, local stresses which in turn decide the energy to form the constriction. The shape of partials and the energy to form the constriction was established for stacking fault energies in the range 10-100 mJ/m^2, misfit strains in the range of 0.1 to 0.5, and nominal interstitial concentration varying from 0 to 10 atomic %. In extreme cases, the constriction energy has been found to increase by four-fold compared to the interstitial-free case. The results are readily applicable to iron alloys with carbon and nitrogen interstitials, and copper alloys with manganese, aluminum or zinc as substitutional solute atoms. The results converge to the well known solution of Stroh in the limit of zero interstitial concentration.

8:55 AM

Stacking Faults on (001) in $MoSi_2$-WSi_2 Solid-Solutions with the $C11_b$ Structure and Their Influence on the Fracture Behavior: *H. Inui*[1]; K. Ito[1]; T. Nakamoto[1]; M. Yamaguchi[1]; [1]Kyoto University, Dept. of Matls. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan

Stacking faults on (001) in $MoSi_2$-WSi_2 solid-solutions with the $C11_b$ structure have been characterized by transmission electron microscopy (TEM), using their single crystals grown by the floating-zone method. Although binary WSi_2 contains a high density of stacking faults, only a few faults are observed in binary $MoSi_2$. The density of these stacking faults increases with the increase in the WSi_2 content in the solid-solutions with the drastic increase occurring only near the WSi_2- end side. These (001) faults are characterized to be of the Frank-type in which two successive (001) Si layers are removed from the lattice, giving rise to a displacement vector parallel to [001]. When the displacement vector of faults is expressed in the form of R=1/n[001], however, their n values are slightly deviated from the exact value of 3, because of dilatation of the lattice in the direction perpendicular to the fault, which is caused by the repulsive interaction between Mo (W) layers above and below the fault. Matching of experimental high-resolution TEM images with calculated ones indicates Π values to be 3.12 + 0.10 and 3.34 + 0.10 for $MoSi_2$ and WSi_2 respectively. Fracture toughness has also been measured as a function of WSi_2 content with notched specimens by three point bending. When a crack propagates parallel to (001) fault planes, the value of fracture toughness is higher for $MoSi_2$ than for WSi_2 when a crack propagates parallel to (110) so that a crack is arrested by (001) faults if they exist. This may indicate that (001) faults are beneficial for increasing the fracture toughness of $MoSi_2$-WSi_2 solid-solutions.

9:20 AM

Cyclic Deformation and Strain Burst Behavior of Cu-7 at%Al and Cu-16at%Al Single Crystals with Different Orientations: *Zhong Guang Wang*[1]; Xi Mao Wu[1]; Guang Yi Li[1]; [1]Chinese Academy of Science, State Key Lab. for Fatigue and Fracture of Matls., Shenyang 110015 China

Compared to the cyclic deformation of wavy slip materials, our knowledge about the cyclic deformation of planar slip materials has been much less. The available reports up to date are not consistent and sometimes even controversial with each other. The materials used for these studies were usually copper alloys containing alloying elements such as Al or Zn in order to reduce the value of stacking fault energy (SFE). Abel et al. [1] studied the effect of aluminum content (2-16 at%) on the fatigue behavior of Cu-Al single crystals oriented for single slip. They found that the cyclic hardening behavior of the alloys with Al content less than 4 at% was similar to that of pure Cu single crystals showing clear saturation behavior. Alloys containing more than 11 at% Al did not show saturation behavior before failure. By using multistep tests, Yan et al. [2] reported the occurrence of saturation behavior and a regular cyclic stress-strain curve with a plateau in Cu-16at% Al single crystal oriented for single slip. However, Hong and Laird [3-5] investigated the cyclic deformation of the same alloy again under total strain control mode and this time for the entire range of strain amplitudes investigated stress saturation in cyclic hardening curves was not detected until the final failure.

9:45 AM

Internal Stresses in Heavily Deformed Materials and Their Influence on Mechanical Response: *Ke Han*[1]; J. D. Embury[2]; [1]Florida State University, Nat. High Mag. Field Lab., Tallahassee, FL 32310 USA; [2]McMaster University, Matls. Sci. & Eng., Hamilton, Canada

The deformation of two phase materials represents one very valuable method of producing extremely high strength materials. One aspect of these materials which is poorly understood is the pattern of residual stresses produced by codeformation. The current work examines a variety of methods of estimating these stresses and the influence of these stresses on both monotonic and cyclic deformation.

10:10 AM Break

10:35 AM

Influence of Thermo-Mechanical Treatment and Loading Frequency on the High Cycle Fatigue Properties of AlZnMgCul, 5 Aluminum Alloy: *H. Mayer*[1]; M. Papakyriacou[1]; R. Pippan[1]; S. Stanzl-Tschegg[1]; [1]University of Agricultural Sciences, Instit. of Meteorology & Physics, Vienna, Austria

Fatigue properties of age hardened AlZnMgCul, 5 aluminum alloy have been investigated in the high cycle/low crack growth rate regime. AlZnMgCul, 5 was tested in three different thermo-mechanical conditions: artificially aged (T6), cold worked and artificially aged to increase static strength (T66), and cold worked and artificially aged to increase ductility (T64). To observe an eventual influence of cycling frequency on the fatigue behaviour, S-N experiments were performed using conventional and ultrasonic fatigue testing equipment. The best fatigue properties in the S-N experiments were found for T6 hardening condition, whereas T66 as well as T64 thermo-mechanical treatment deteriorated the fatigue properties, especially at very high numbers of cycles. Similarly, the best fatigue crack growth properties were found for T6 condition. A threshold cyclic stress intensity to propagate a fatigue crack could be detected, whereas no endurance limit could be found in S-N experiments.

11:00 AM

Interfacial Fatigue Fracture in Copper-Sapphire Bicrystals: *P. Peralta*[1]; U. Ramamurty[2]; S. Suresh[3]; G. H. Campbell[4]; W. E. King[4]; T. E. Mitchell[5]; [1]Arizona State University, Dept. of Mech. and Aerospace Eng., P.O. Box 876106, Tempe, AZ 85287-6106 USA; [2]Nanyang Technological University, School of Mech. and Prod. Eng., 639798 Singapore; [3]Massachusetts Institute of Technology, Dept. of Matls. Sci. and Eng., Cambridge, MA 02139-4307 USA; [4]Lawrence Livermore National Laboratory, MSL-356, Livermore, CA 94550 USA; [5]Los Alamos National Laboratory, MST-CMS, MSK765, Los Alamos, NM 87545 USA

Interfacial fatigue cracks were propagated in copper/sapphire bicrystals with the boundary perpendicular to the load axis and $(110)_{Cu}(1010)_{Al2O3}$/[001] Cu$[0001]_{Al2O3}$ to study the effect of crystallography and slip geometry in the fracture process. Compact tension (CT) specimens with two different crystallographic directions for crack growth were loaded in tension-tension with ΔK applied ≈2MPa.$m^{1/2}$. Cracks grew preferentially when the growth direction was $<110>_{Cu}$ and less favorably when the growth direction was along $<001>_{Cu}$, which also resulted in an inclined crack front. Striations, which did not coincide macroscopically with traces of available slip planes, could be observed on the copper fracture surface; however, large areas were also relatively free of features. Elastic analysis of the anistropic near-tip stress fields for the interfacial crack revealed that the preferential crack growth direction had the highest energy release rate, whereas the second crack direction had the minimum mode II mix. A model to account for the non-crystallographic striations observed is proposed.

11:25 AM

Cyclic Deformation Mechanisms in NiTi Alloys: *Huseyin Sehitoglu*[1]; Ken Gall[2]; I. Karaman[1]; R. Anderson[1]; Y. I. Chumlyakov[3]; [1]University of Illinois, Dept. of Mech. and Industrial Eng., Urbana, IL 61801 USA; [2]University of Colorado, Dept. of Mech. Eng., Boulder, CO 80309 USA; [3]Siberian Physical and Technical Institute, Physics of Plasticity and Strength of Matls. Lab., Tomsk 634050 Russia

Single crystal NiTi shape memory alloys exhibit considerable cyclic hardening under zero-tension and zero-compression strain control experiments. The stress amplitude under strain control can increase by as much as factor of 1.2 in tension and a factor of 2 in compression. The increase in stress amplitude is primarily from the increasing strain hardening modulus. The deformation is controlled by transformation from a parent to martensitic phase, and detwinning of the martensite. Both the transformation strains and detwinning strains are orientation and stress direction dependent. In the past, the cyclic deformation characteristics of NiTi have been primarily studied in polycrystalline alloys. In this presentation, the focus will be on single crystals. The strong advantage of single crystals is that the characteristics of the deformation mechanisms (martensite plate variants and slip systems) are a known function of the loading axis of the crystal. In addition, these materials are almost always used in aged condition to produce pseudoelastic stress-strain response. Consequently, the precipitate coherency and precipitate size influences the deformation resistance. The presentation will highlight some of the micro-mechanical modeling to predict the role of precipitates on the deformation resistance

and explain the strong dependence of orientation and stress direction on the fatigue response.

Fundamentals of Lead and Zinc Extraction and Recycling: Session I

Sponsored by: Extraction & Processing Division, Lead, Zinc, and Tin Committee

Program Organizers: Markus Reuter, Delft University of Technology, Applied Earth Sciences, Delft 2628 RX The Netherlands; Eric Allain, University of Missouri, Rolla, MO 65409-1460 USA

Tuesday AM — Room: Bayou D
March 14, 2000 — Location: Opryland Convention Center

Session Chair: M. A. Reuter, Delft University of Technology, Appl. Earth Sci., Delft, RX 2628 The Netherlands

TUESDAY AM

8:30 AM

Modeling of the Lead Blast Furnace: *Bart Blanpain*[1]; P. Verguts[1]; P. Wollants[1]; S. Brouwer[2]; Frank De Buyck[3]; [1]KU Leuven, Dept. of Metallu. and Matls. Eng., Leuven Belgium; [2]Union Minière, B.U. Precious Metals, A. Greinerstraat 14, Hoboken B-2660 Belgium; [3]Union Minière, Research, Kasteelstraat 7, Olen B-2250 Belgium

Union Minière, one of the major non-ferrous metal producers in the world with several industrial sites in Belgium, recently changed its copper-lead metallurgy flow sheet substantially. Before the change, lead-copper ore sinter and secondary materials were fed into a blast furnace. Now only secondary raw materials are smelted in the blast furnace. Since the porosity of this feed is very low in comparison with the former sintered ores, the gas-solid reactions occur very slowly. Because the process of the blast furnace has changed considerably, a mathematical model of the furnace is being constructed as a tool to study the phenomena occurring in the furnace. The model uses general Computational Fluid Dynamics together with models for porous zones, combustion and so on. The model will be used to study the influence of operating parameters on blast furnace phenomena in order to optimize blast furnace performance. An outline of the model, together with first results will be discussed.

8:50 AM

Production of an Ultra-Pure Fraction of ZnO by the Recycling of EAF Dust: Fernando García-Carcedo[1]; Nancy Ayala[1]; N. Goicoechea[2]; A. Hernández[1]; Enrique Ruiz-Ayúcar[1]; E. García-Ventosa[1]; I. Dañobeitia[2]; N. Cornejo[1]; *Eric G. Allain*[3]; [1]CENIM, Centro Nacional de Investigaciones Metalúrgicas, C/. Gregorio del Amo 8, Madrid 28040 Spain; [2]Compañía Industrial ASER, S.A. Carretera Bilbao-Plencia, Asúa-Erandio, Bilbao, Vizcaya 48950 Spain; [3]UMR, Dept. of Metallu., 210 Fulton Hall, Rolla, MO 65401 USA

This research aimed to develop a clean technology for the production of ZnO from residues generated by the steel making industry. The scientific and technologic parameters were both investigated in order to define an industrial methodology allowing the integral recycling of EAF dusts. A reengineering of the Wáelz process was performed, aiming to produce a clean fraction of ZnO, free of fluorine, as well as a slag enriched in metallic iron, which can be recycled to the electric arc furnace. The technical conditions used to produce the ultra pure fraction of ZnO are described. The magnetic separation of the EAF dust allowing the production of a phase high in iron and directly recyclable to the electric arc furnace is also discussed. Thermodynamic of the volatilization of all the elements contained in the EAF dust was performed in order to allow a better control of the thermal process in the rotary kiln. Tests were run using different reducing agents, weakly or highly reactive, to determine their specific efficiency.

9:10 AM

Zincproblem Integrated Steel Industries Gradually Alters to Source of Lead and Zinc with the Aid of Metallurgy: *Simon Honingh*[1]; [1]Hoogovens Staal BV, Environmental Control & Mgmt. Sys., P.O. Box 1000, IJmuiden 1970CA The Netherlands

Hoogovens has been investigating different options for the processing of their zinc/lead containing flue dust. Four methods have been applied on laboratory and/or pilot scale to process this flue dust viz. (i) pressure leaching and subsequent hydrometallurgical processing, (ii) processing of the flue dust together with spent pickling acid in a pyrohydrolyzer, (iii) pyrometallurgical treatment of the flue dust in an electric arc furnace/plasma furnace and (iv) processing of the flue dust in a cupola furnace. Various results obtained will be discussed and evaluated to establish which process is most suitable for processing of this flue dust.

9:30 AM

Comparative Study on Zinc Electrowinning from Sulfate and Chloride Solutions: Carla Lupi[1]; *Daniela Pilone*[1]; [1]University of Rome "La Sapienza", Dept. ICMMPM, Via Eudossiana 18, Roma 00184 Italy

Zinc electrowinning from sulfate solutions is the traditional industrial method to produce SHG zinc. Over the past few years several efforts have been done to improve process efficiency in order to reach high productivity with low energy requirement. The latter aim can be pursued both by a hard purification of the electrolyte and by a reduction in the anodic voltage. In this work electrowinning tests on industrial electrolyte have been carried out by using Pb-Ag anode and by adding organic depolarizers such as ethanol, acetic acid or ethylene glycol. At the same time tests were performed on chloride solutions obtained from purification of zinc exhausted electrolyte: these experiments have been done in different operative conditions by using DSA anodes and by testing the effect of various additives such TEACl and TBACl. Comparing the best results obtained with the two methods it can be highlighted that a high quality zinc is electrowon in both cases, but the energy requirement is lower in the case of zinc chloride electrowinning.

9:50 AM

The Use of Data Reconciliation to Optimise Metallurgical Plants-Case Study Zn Plant: *Markus Andreas Reuter*[1]; Sabina Grund[2]; Thomas Auping[1]; [1]TU Delft, Raw Matls. Processing, 120 Mijnbouwstraat, Delft 2628RX The Netherlands; [2]Consultant, Alter Postweg 12, Dorsten 46282 Germany

This paper discusses data reconciliation as a tool to assist in the modeling and optimisation of metallurgical plants. After discussing the theory the methodology is illustrated using an industrial hydrometallurgical zinc plant as a case study. Various sections of the plant are mass balanced and various fundamental and practical useful relationships are derived. These are discussed in terms of among others process control, metallurgical control, accounting and environmental monitoring.

10:10 AM

Spouted Bed Electrowinning of Zinc from Zinc Chloride Electrolytes: *A. Roy*[1]; J. W. Evans[1]; C. Allen[2]; [1]University of California, Dept. of Matls. Sci. and Eng., Berkeley, CA 94720 USA; [2]Noranda Inc., Tech. Ctr., 240 Hymus Blvd., Point-Claire, Quebec H9R1G5 Canada

The spouted bed electrode (SBE) is a particulate electrode in which metal particles may be grown from a small "seed" to particles that are a few mm across. In prior work at UC Berkeley the SBE has been studied to determine its suitability for use in electrowinning zinc (from both conventional acid sulfate electrolytes and from alkaline electrolytes) and copper. The paper describes the results of a laboratory investigation into the application of this electrode for electrowinning from zinc chloride electrolytes. The anodic reaction was the evolution of chlorine and, when steps were taken to minimize attack of the zinc particles by this chlorine, high current efficiencies (as high as 93%) were obtained. The dependence of cell performance on some operating/design parameters such as current density (up to 4381A/m2) was examined. Reasonable electrical energy consumptions (as low as 3kWh/

kg Zn) were found under many circumstances. Zinc deposits appeared metallic to the naked eye and dense under the SEM.

General Non-Ferrous Pyrometallurgy: Pyrometallurgical Processing of Minerals and Metals

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee

Program Organizers: Robert L. Stephens, Cominco Research, Trail, British Columbia V1R 4S4 Canada; Pekka Taskinen, Outokumpu Research Oy, Pori FIN-28101 Finland

Tuesday AM — Room: Bayou B
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Adrian C. Deneys, Praxair, Tarrytown, NY 10591-6714 USA

8:30 AM

Effect of Temperature and Oxygen Partial Pressure on Phase Equilibria of Natural Chromite Minerals: *Vilas D. Tathavadkar*[1]; *Clair C. Calvert*[1]; Animesh Jha[1]; M. P. Antony[1]; Martin Wilkinson[2]; [1]University of Leeds, Dept. of Matls., Leeds, West Yorkshire LS29JT UK; [2]Elementis Chromium, Egalescliff, Stockton-on-Tees, UK

Chromite ore is of immense importance to the extraction of chromium metal and chemicals. The mineralogy and crystal structure of chromite phase are therefore important part of our investigation on the extraction chemistry of chromium species. In this paper, we have investigated the effect of heat treatment on mineral composition and structure as a function of temperature and gaseous atmosphere. The effect the processing parameters i.e. temperature and oxygen potential on the microstructure and composition of mineral phases formed have been systematically investigated in view of the phase equilibria in the Cr_2O_3-Al_2O_3-Fe_xO_y-MgO system and spinoidal decomposition reactions. Experiments were carried out in order to study the differences in the crystallography of different chromite minerals. Phase equilibria in natural chromite minerals has been investigated by calcining chromite mineral in air, argon and 5% hydrogen + argon atmospheres over a temperature range from 200 to 1200°C. The effect of the oxygen partial pressure and temperature on the phase constituents of the calcined product has been discussed. The microstructural changes were examined using scanning electron microscope (SEM) and the changes in elemental composition in different phases were analysed by electron probe micro analyser (EPMA). The results obtained from EPMA studies were used to calculate lattice parameter of the spinel lattice and these values were compared with the values obtained from X-ray powder diffraction data. It is evident from the experimental results that spinoidal decomposition of complex spinel phases has occurred under the influence of different oxygen partial pressures and temperatures.

8:55 AM

Chlorination Kinetics of Xenotime with Chlorine in Presence of Carbon: Marco Antonio Gimenes[1]; *Herenilton Paulino Oliveira*[1]; [1]Faculdade de Filosofia, Ciências e Letras de Rib. Preto-USP, Chem., Av. Bandeirantes 3900, Ribeirão Preto, São Paulo 14040-901 Brazil

The utilization of chlorination in extractive metallurgy and advanced ceramics areas has been widely investigated as a preparative route in order to obtain intermetallic compounds, which are used as precursors in the development of new materials and process. The major processes of decomposition of rare-earth ores use sulfuric acid and sodium hydroxide at high temperatures; however, there is not much work concerning chlorination. In this work, a systematic study of the reaction between xenotime ($REPO_4$), chlorine, and carbon has been performed. Particular emphasis was on kinetics studies to establish optimized conditions for the reaction. The kinetics of chlorination of xenotime raw material by rare-earth elements/compounds has been studied over a temperature range from 600°C to 1100°C. The influence on the rate of conversion of xenotime to $RECl_3$ of temperature, partial pressure of chlorine, carbon content, and particle size were investigated. A global rate equation that includes these parameters has been developed. The results show that the process follows the unreacted core shrinking model with a formation of a product layer. The powder X-ray diffraction technique corroborated the model showing clearly the patterns related to the formation of yttrium oxychloride (YOCl), indicating that the reaction mechanism involves the presence of an intermediate step before the formation of lanthanide chloride. We thank FAPESP for financial support (proc.:1997/05779-1).

9:20 AM +

Reaction Sequences in Sulphide Particle Oxidation: *Esa J. Peuraniemi*[1]; Ari Jokilaakso[1]; [1]Helsinki University of Technology, Lab. of Matls. Proc. and Powder Metallu., P.O. Box 6200, Espoo 02015 Finland

Oxidation of chalcopyrite concentrate and two low-iron copper mattes were studied using a laboratory scale laminar-flow furnace simulating the phenomena taking place in the reaction shaft of a flash furnace. In the experiments, screened fractions were fed into the furnace and sampled after short reaction time intervals by quenching them into a water film. Experimental conditions included temperatures of 1100 and 1300°C with reaction gas oxygen contents from 21 to 75 vol%. Samples were analysed chemically for Cu, Fe and S to define their respective removal rates. Optical and scanning electron microscopy with EDS-analyser were used to examine the phenomena occurring during reactions. Oxidation kinetics as well as ignition of particles are discussed. Changes in particle morphology, size, and composition are viewed to closely follow the development of the oxidation phenomenon and, consequently, to compose a detailed model of dust formation and reaction mechanisms. Data obtained in the experiments enlarges the knowledge of physical and chemical phenomena in flash reactions and serves as reference for computer simulations.

9:45 AM

Thermodynamics of Deoxidation of Molten Titanium and Zirconium: *Yoshinao Kobayashi*[1]; Fumitaka Tsukihashi[2]; [1]National Research Institute for Metals, Matls. Creation Rsch. Station, 1-2-1, Sengen, Tsukuba-shi, Ibaraki 305-0047 Japan; [2]The University of Tokyo, Dept. of Advanced Matls. Sci., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-0033 Japan

The removal of oxygen from titanium and zirconium is important for use in application because it significantly affects the mechanical and physical properties such as ductility and toughness. However, thermodynamic properties of oxygen in molten titanium and zirconium have not been clarified and the effective deoxidation process of molten titanium and zirconium has not been established. In this study, thermodynamic properties of oxygen and yttrium in molten titanium and zirconium have been investigated by a chemical equilibrium technique. Molten titanium, zirconium, and their alloys with aluminum were equilibrated with a Y_2O_3, Al_2O_3, or ZrO_2 pellet in a cold crucible. The results are summarized as follows: $1/2O_2(g)=O(X_O$, in Ti), ΔG^0=-566,000+103T[J/mol](1673 to 1873K), $1/2O_2(g)=O(X_O$, in Zr), ΔG^0=-543,000+64.6T[J/mol](1848 to 1973K), $Y_2O_3(s)=2Y$(mass pct, in Ti)+3O(mass pct, in Ti), ΔG^0=601,000-262T[J/mol](1991 to 2093K), $Y_2O_3(s)=2Y$(mass pct, in Zr)+3O(mass pct, in Zr) and ΔG^0=736,000-317T[J/mol](2153 to 2173K). The possibilities of deoxidation by using yttrium-based fluxes are discussed based on the observed thermodynamic data.

10:10 Break

10:20 AM

Study on Electroslag Remelting of Cu-Cr-Zr Alloy: *Xiao Yu Shen*[2]; *Ji He Wei*[1]; [1]Shanghai University, Dept. of Metallic Matls., 149 Yan Chang Rd., Shanghai 200072 PRC; [2]Shanghai Electrical Apparatus Research Institute, 505 Wu Ning Rd., Shanghai 200063 PRC

Electroslag remelting (ESR) of Cu-Cr-Zr alloy has been experimentally investigated. The remelting experiments were carried out with different slags in the CaF_2+NaF, CaF_2+ZrO_2 and CaF_2+NaF+ZrO_2 systems in an ES unit of 25 kg capacity. The influence of slag on the losses of alloying elements (Zr and Cr) has been considered and exam-

ined. The technologies of the pressure working and heat treatment of the ER ingot have been described and discussed. Some physical properties of the slag in CaF_2+NaF+ZrO_2 system and the remelted Cu-Cr-Zr alloy have been determined. The results indicated that with the slag in a CaF_2+NaF+ZrO_2 system and other operation parameters employed, a high quality ingot of Cu-Cr-Zr alloy may be made with high yields of Zr and Cr by means of ESR. For the remelted Cu-Cr-Zr alloy with a specified composition of 0.5-mass% Cr and 0.1 mass-% Zr, the softening temperature, hardness and electrical conductivity are 550°C, HRB 75 and larger than 43 MS/m, respectively. The properties of Cu-Cr-Zr alloy products are in accordance with and superior to the requirements and specifications of ISO 5182-1991(E).

10:45 AM

Kinetic Study on the Carbon Thermal Reduction of V_2O_3: *Zhiyu Lu*[1]; Zhitong Sui[1]; Zhenqi Huang[1]; Jing Yang[1]; [1]Northeastern University, School of Matl. and Metallu., Shenyang 110006 PRC

Vanadium is an important alloy element in making high strength low alloy steel in which vanadium can play the role of dispersion strengthening to the steel. In recent years there has been great interest in vanadium carbide (VC), vanadium nitride (VN) and vanadium carbide nitride (V(C,N)), because all of the three compounds can exhibit excellent quality for the addition of vanadium to the molten steel. In making these compounds V_20_3 is an important raw material due to its low oxygen content in the molecule, so the kinetic study on reduction of V_2O_3 can provide valuable information to the production of VC, VN, V(C,N). In this paper the reduction process of V_2O_3 was studied by TG and XRD method, the study results reveal that the reduction rate was controlled by both interface chemical reaction step and mass transport step in the experimental temperature scope (1300K—1700K). The apparent activation energy for the interface chemical reaction was 34kJ/mole; the apparent activation energy for the mass transport step was 32kJ/mole. The study on the reduction mechanism can give the following conclusion. When the temperature is lower than 1300K, the carbon reduction mechanism of V_2O_3 give mainly VC, and there are enough clues indicates that the main reaction is a self-catalyzed reaction at this temperature range.

High-Temperature Superconductors: Coated Conductors

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

Program Organizers: U. Balu Balachandran, Argonne National Laboratory, Argonne, IL 60439 USA; Pradeep Haldar, Intermagnetics General Corporation, Latham, NY 12110-0461 USA; Chandra Pande, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5000 USA

Tuesday AM — Room: Canal D
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Chandra S Pande, Naval Research Laboratory, Matls. Sci. Div., Washington, DC 20375 USA

8:30 AM Invited

High-Jc, YBCO Conductors Fabricated by Epitaxial Deposition of YBCO on Rolling Assisted Biaxially Textured Substrates (RABiTS): *Amit Goyal*[1]; F. A. List[1]; D. F. Lee[1]; D. M. Kreoeger[1]; M. Paranthaman[1]; X. Cui[1]; R. Feenstra[1]; D. T. Verebelyi[1]; T. Autug[1]; C. Cantoni[1]; D. K. Christen[1]; P. M. Martin[1]; T. Chirayil[1]; C. Park[1]; D. P. Norton[1]; R. K. Williams[1]; E. D. Specht[1]; D. B. Beach[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics, P.O. Box 2008; MS 6116, Bldg. 4500S; Rm. B-248, Oak Ridge, TN 37831 USA

Advances in the fabrication of Rolling assisted biaxially textured substrates (RABiTS) and epitaxial deposition or formation of HTS on such substrates is reported. Significant progress has been made in the fabrication of non-magnetic, strengthened, biaxially textured metal templates, deposition of oxide and other buffer layers and in the fabrication of long length substrates and superconductors. High Jc's exceeding 1 MA/cm2 have been demonstrated on epitaxially grown YBCO films on RABiTS using Ni-Cr as the starting template. High Jc's have been demonstrated on a variety of new buffer layer configurations including conducting buffer layers. Efforts are underway to fabricate longer length superconductor samples exceeding 10cm and results obtained will also be summarized. Particular emphasis would be given to microstructural factors affecting Jc in coated conductors. Research sponsored by U. S. Department of Energy under contract DE-AC05-96OR22464 to Lockheed Martin Energy Research Corporation.

9:10 AM

Microstructure of YBCO Films Deposited on Oxide Buffered Rolling Assisted Biaxially Textured Ni Substrates: *Chau-Yun Yang*[1]; S. E. Babcock[1]; A. Goyal[2]; F. A. List[2]; J. E. Mathis[2]; C. Park[2]; M. Paranthaman[2]; D. F. Lee[2]; D. P. Norton[2]; D. M. Kroeger[2]; [1]University of Wisconsin-Madison, Appl. Superconductivity Ctr., 1500 Engineering Dr., Madison, WI 53706 USA; [2]Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831 USA

The microstructures of pulsed laser deposited YBCO films on different buffer layer materials including YSZ, CeO_2, and Yb_2O_3 were studied with a view toward identifying current limiting defects and elucidating the roles of the buffer layer material in YBCO coated conductors. The YBCO films deposited on YSZ possess an island microstructure, and the island size increases with increasing film thickness. The observed current limiting mechanisms are unlikely to be low angle grain boundaries between YBCO islands but pores, especially the columnar pores elongated through YBCO [001] direction when the films are over 1 mm thick, second phase particles, and 45° rotated grains. The YBCO film grown on CeO_2 does not show the same island microstructure, rather a single YBCO grain that contains twin boundaries on both sets of {110} planes extend over the entire 10 to 20 microns observable area of TEM specimens. Although some dislocations widely dispersed in the film, no clear domain structure is observed from YBCO on a given Ni grain. Two observed main current limiting factors are open pores and poor YBCO connection across Ni grain boundaries. The results is a films with a low Jc value of just ~ 0.2 MA/cm2. The high Jc (1.7MA/cm2) YBCO film grown on recently developed new buffer Yb_2O_3 has an island microstructure like that of YBCO on YSZ. These results suggest that microstructural details of length scale ranging from sub-micron to many tens of microns depend on the buffer layer. This work is supported by US-DOE through ORNL.

9:50 AM Break

10:00 AM Invited

Direct Measurements of Grain Boundary Transport Currents in YBa2Cu3Ox Coated Conductors: *Dean J. Miller*[1]; Peter Berghuis[1]; Ron Feenstra[2]; Dave K. Christen[2]; [1]Argonne National Laboratory, Matls. Sci. Div., 9500 S. Cass, MSD-223, Argonne, IL 60439 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

The impressive transport properties achieved in YBa2Cu3Ox coated conductors have led to an intense effort to understand the key factors that influence the critical current and current density on this type of conductor. In this work, we aim to understand the current path in these conductors and particularly how grain boundaries influence the critical current density. We use micropatterning techniques to isolate individual grain boundaries on coated conductors prepared using RABiTS substrates. The transport properties measured across these single grain boundaries are then compared to those across grain boundaries from thin film and bulk bicrystals as well as to the global transport properties of coated conductors. In this presentation, details of the patterning and measurement of critical currents will be presented. An emphasis will be placed on the comparison with global transport properties and the influence of the criterion used to establish the critical current. This work was partially supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences and Energy Efficiency and Renewable Energy, under contract #W-31-109-ENG-38.

10:40 AM
IBAD/MOCVD-Based YBCO-Coated Conductor Development: *U. Balachandran*[1]; M. P. Chudzik[1]; R. A. Erck[1]; C. R. Kannewurf[2]; V. Selvamanickam[3]; P. Haldar[3]; [1]Argonne National Laboratory, Energy Tech., 9700 S. Cass Ave., Argonne, IL 60440 USA; [2]Northwestern University, Dept. of Elect. Eng., Evanston, IL 60208 USA; [3]Intermagnetics General Corporation, Latham, NY 12110 USA

Biaxially aligned yttria-stabilized zirconia (YSZ) films were deposited on polished Hastelloy substrates via ion-beam-assisted deposition (IBAD). Atomic plumes were created by electron beam evaporation and an argon-ion gun aided texture development in the YSZ. Effects of ion-to-atom flux ratio and beam optics divergence on in-plane texture of YSZ were investigated. Epitaxial cap layers of cerium oxide were deposited on IBAD/YSZ films by electron beam evaporation. YBCO superconductors of ≈1 μm thickness were deposited on ceria cap layers by metal-organic chemical vapor deposition (MOCVD). The MOCVD processing parameters were optimized and critical currents of >50 A (critical current density >1 MA/cm^2) were obtained at 77K. The deposition conditions required to obtain the textured layers will be discussed. *Work at ANL and part of the work at IGC supported by the U.S. Department of Energy, Energy Efficiency and Renewable Energy, as part of a program to develop electric power technology, under Contract W-31-109-Eng-38. Work at NU supported by the National Science Foundation through the Science and Technology Center for Superconductivity (Grant No. DMR 91-2000).

11:20 AM Invited
A Study on the Grain Texturing Mechanism of YBCO Film on a Silver Alloy Substrate through Peritectic Solidification: *Donglu Shi*[1]; [1]University of Cincinnati, Matls. Sci. & Eng., 498 Rhodes Hall, Mail Location 0012, Cincinnati, OH 45221-0012 USA

Quenching experiments were carried out near the peritectic temperature for the thick films of YBa2Cu3Ox (YBCO) on the silver alloy substrate. The initial YBCO morphology exhibits a column-like grain structure as a result of rapid a-growth when quenched from 1000°C (the sample was pre-melted at 1030°C). A waffle-like structure was observed on the surface of the silver alloy substrate as the quenching temperature was lowered to 950°C providing a much greater driving force. We found that a grain-oriented substrate may not be required to achieve the grain texturing in the peritectic-reaction-controlled process. During solidification, the YBCO grains will nucleate on the surface of the silver alloy in a parallel fashion to minimize its surface energy, and grow along the a-axis rapidly resulting in a textured film.

Honorary Symposium for Professor Oleg D. Sherby: Creep Mechanisms and Behavior A

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

Tuesday AM — Room: Bayou E
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Alan Ardell, University of California, Matls. Sci. and Eng. Dept., Los Angeles, CA 90095-1595 USA

8:30 AM Opening Remarks

8:40 AM Keynote
Mechanisms of Time-Dependent Plasticity in Polycrystalline Thin Films on Substrates: *William D. Nix*[1]; [1]Stanford University, Matls. Sci. and Eng., 416 Escondido Mall, Bldg. 550, Stanford, CA 94087-2205 USA

Mechanisms of time-dependent plasticity in polycrystalline thin metal films (Al, Cu, Au) on silicon substrates are reviewed with particular reference to understanding the softening and hardening effects of dislocations and grain boundaries. Diffusional deformation contributes to plasticity in these fine-grained materials, though it can be inhibited by the presence of thin passivating layers. For unpassivated films it is shown that diffusional deformation involving mass transport between the free surface of the film and the grain boundaries is constrained by kinetic processes at the film/substrate interface and that classical Coble or Herring-Nabarro creep relations do not apply unless the film/substrate is free to slide. For the case of no sliding at the film/substrate interface, diffusional deformation alone cannot relax the stresses completely at high temperatures, with the consequence that dislocation plasticity must be active when full relaxation is observed. For passivated films, plasticity appears to be dominated by dislocation processes. Dislocation plasticity is largely athermal in passivated thin films because the storage of dislocations at the film/substrate and film/passivation interfaces dominates the deformation resistance. The hardening effects of grain boundaries are also explored and compared with the hardening processes that occur in single crystal films. Some time-dependent stress relaxation is observed in Au films at room temperature. This appears to be controlled by thermally activated dislocation cutting processes.

9:10 AM Invited
The Interpretation of Creep Mechanisms in High Temperature Flow: *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Matls. Sci. & Mech. Eng., Los Angeles, CA 90089-1453 USA

Several different flow mechanisms may occur in high temperature creep and an identification of the rate-controlling process is generally based on measurements of the stress exponent and the activation energy. This paper considers the different creep processes occurring in crystalline solids and examines procedures for their unambiguous identification.

9:30 AM Invited
Subgrain Strengthening Revisited II: *Michael Ernest Kassner*[1]; [1]Oregon State University, Dept. Mech. Eng., Rogers Hall, Corvallis, OR 97331 USA

This work is a sequel to earlier work with Prof. Oleg D. Sherby and the author on the effects of subgrain boundaries on elevated-temperature plasticity [1]. This work discusses some of the more recent work in this area, including investigations by the author, that attempted to discern the contributions of various substructural features, including subgrain boundaries, on the rate-controlling process for five power-law-creep in single-phase metals. Particular attention is devoted to recent developments regarding internal back-stresses. This will include discussions of recent in-situ TEM, x-ray diffraction and convergent beam electron diffraction experiments on metals to evaluate internal stresses in association with dislocation heterogeneities. [1]O.D. Sherby, A.K. Miller and M.E. Kassner, “Subgrain Strengthening Revisited”, Metals Forum, 4, 1981, pp. 53-56.

9:50 AM Invited
Constant Structure Creep of Aluminum: All the Data in the World: *Jeffrey C. Gibeling*[1]; [1]University of California, Dept. of Chem. Eng. and Matls. Sci., One Shields Ave., Davis, CA 95616-5294 USA

The analysis of creep under conditions of constant internal structure is reviewed, with the goal of using these results to develop a better understanding of the mechanisms of deformation. Data from both strain rate change experiments and stress change tests are considered. While both types of experiments give similar results, greater emphasis is given to the latter in the present discussion. Data for aluminum from numerous investigations present a consistent picture over a wide range of temperatures and stresses. These results show that constant structure creep after stress reductions occurs by parallel processes of dislo-

cation glide within subgrain interiors and dynamic recovery associated with subgrain boundaries. After relatively small changes in stress, thermally activated motion of dislocations within subgrain interiors is the predominant mechanism of deformation. In this regime, a thermally activated rate law can describe the creep transients, thereby enabling various activation parameters to be evaluated from the data. In particular, the true activation areas are consistent with thermally activated cutting of forest dislocations. In contrast, dynamic recovery processes dominate the constant structure deformation following relatively large stress reductions. Selected results for other FCC metals and related materials are shown to follow the trends established for pure aluminum. In particular, it is demonstrated that constant structure creep of pure copper and LiF at high temperatures and after small stress changes is also consistent with a description based on thermally activated glide. The differences in behavior between these materials are attributed to differences in stacking fault energy.

10:10 AM Break

10:20 AM Keynote

Unnatural Power-Law Creep Exponents, Nonlinear Monotonic and Cyclic Stress-Strain Curves and Similitude Breaking: *Johannes Weertman*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

Many alloys have a “natural” power law exponent of about 3 at the higher temperatures. Pure metals and some alloys have an “unnatural” power law exponent of about 5 at the higher temperatures and an unnatural power law exponent of about 7 (instead of 5) at moderately high temperatures. It is argued in this talk that the discrepancy of the exponent in high temperature creep, as well as the occurrence of nonlinear elastic-plastic stress strain curves, is a consequence of similitude breaking in the underlining dislocation mechanics. (Similitude breaking is a concept introduced by Kuhlmann-Wisdorf Met. Trans. 16A, 2091 (1985) to dislocation mechanics.)

10:50 AM Invited

New Predictions of the Dislocation Network Theory of Harper-Dorn Creep: *Alan J. Ardell*[1]; Marek A. Przystupa[1]; [1]UCLA, Matls. Sci. and Eng., 6531-G Boelter Hall, Los Angeles, CA 90095-1595 USA

The dislocation network theory of high-temperature deformation explains many features of Harper-Dorn (H-D) creep that other theories do not. One is that the dislocation density in the H-D creep regime is independent of the applied stress. Frustration of dislocation network coarsening, arising because Frank’s rule cannot be satisfied at the nodes when the network coarsens and dislocations are eliminated, is responsible. The reduction in dislocation density during primary creep in the H-D regime is also satisfactorily explained. Previous equations of the network theory involved formation of only one kind of node resulting from dislocation collisions and annihilation. In real f.c.c. crystals several kinds of interactions are possible, leading to different configurations at dislocation nodes. In the present work we take these into account. We apply the new equations to describe the experimentally measured distributions of dislocation link lengths in Al. We also attempt to provide a self-consistent prediction of creep curves in Al deformed in the H-D regime.

11:10 AM Invited

Effect of Stress-Ratio on Biaxial Creep and Dislocation Microstructures in Recrystallized Ti_3Al_2.5V Tubing: *K. Linga Murty*[1]; S. Nangalia[2]; A. Paradkar[3]; [1]North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA; [2]MCNC Electronic and Information Technologies, RTP, NC 27709-2889 USA; [3]Defence Metallurgical Research Laboratory, Hyderabad, India

Biaxial creep characteristics were investigated on recrystallized Ti_3Al_2.5V tubing by varying the internal pressurization superimposed with axial load at 673K. The stress ratio () of the hoop to axial was varied from 0 to while the hoop and axial creep strains were monitored using a telemetric laser extensometer and an LVDT respectively. The creep locus defined at a constant energy dissipation rate deviated from isotropy with relatively large hardening towards the axial stress axis. The results were fit to the modified Hill’s equation from which the creep anisotropy parameters, R and P, were derived and these parameters deviated from unity with R quite large (~6) and P relatively smaller (~0.5). The stress-state dependence of the strain-rate ratio predicted using these anisotropy parameters was in reasonable agreement with the experimental results albeit relatively large scatter was noted in the data. The crystallographic texture was determined using x-ray diffraction via inverse and direct pole figures from which crystallite orientation distribution functions (CODF) were derived. Predictions based on crystal slip plasticity in conjunction with CODF exhibited large deviations, which were initially thought to arise from contributions from twinning and/or second phase. Dislocation microstructures were investigated as a function of the stress level following creep tests at uniaxial (=) and equi-biaxial (=1) loading. At stresses below about 180 MPa, dislocation arrangement was generally random while at higher stresses (at ~211 MPa) distinct subgrain formation was noted. Whereas, uniaxially loaded samples exhibited random distribution of dislocations even at very high stresses (~351 MPa). In all cases, the dislocations are predominantly <11 0> type lying on {10 0} planes. Some <c+a> type dislocations were observed in few low angle grain boundaries, and these are relatively widely separated. No twins were noted while only small amounts of  phase were observed confined mainly to grain boundaries.

11:30 AM Invited

An Evaluation of Power Law Breakdown in Metals, Alloys and Compounds: *Donald R. Lesuer*[1]; Chol K. Syn[1]; Oleg D. Sherby[2]; [1]Lawrence Livermore National Laboratory, L-342, P.O. Box 808, Livermore, CA USA; [2]Stanford University, Dept. of Matls. Sci. and Eng., Stanford, CA 94305 USA

Creep at high stresses often produces strain rates that do not follow a power law relationship between strain rate and stress. At low stress, a power law relationship is observed while, at high stress, greater strain rates are observed than would be predicted by this relationship. This phenomena is referred to as Power Law Breakdown (PLB). In this paper, we examine the available creep data at high stress in pure metals, solid solution alloys, dispersion strengthened alloys and compounds to identify materials characteristics and experimental conditions (stress, temperature and strain rate) that result in PLB. The results are analyzed by assessing the influence of these higher stresses and strain rates on the diffusion coefficient. For a number of Fe-C alloys, PLB can be explained by the increase in dislocation density with increasing stress and the resulting increase in diffusivity. When creep results in the Fe-C alloys are analyzed in terms of an effective diffusivity, which includes lattice and pipe diffusion, PLB disappears. Power Law Breakdown has also been studied in terms of the production of excess vacancies and the influence of excess vacancies on the diffusion coefficient. A simple model has been constructed to study PLB based on the rates of production and annihilation of vacancies. The results provide insight into the physical basis for PLB in metals alloys and compounds. Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under contract W-7405-ENG-48.

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

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

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

Tuesday AM — Room: Johnson A/B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: David E. Laughlin, Carnegie Mellon University, Matls. Sci. and Eng., Pittsburgh, PA 15213-3890 USA; Alan J. Ardell, UCLA, Matls. Sci. and Eng., Los Angeles, CA 90024 USA

8:30 AM Invited
Some Aspects of Ordering Energy: *Robert W. Cahn*[1]; [1]Cambridge University, Matls. Sci. & Metallu., Pembroke St., Cambridge CB2 3QZ England

The ordering energy of an alloy with a superlattice determines a number of its properties, including the temperature (actual or virtual) of the order-disorder transition, the specific energy of antiphase domain boundaries, the change in lattice parameter when order is lost, the width of superdislocations, resistance to creep under stress, and some aspects of recrystallization. In spite of the fact that several distinct methods exist to estimate the ordering energy, both experimental and theoretical, not much attention has as yet been paid to this variable. This discursive survey will look at a number of these aspects.

9:00 AM Invited
Dynamical Evolution of Ordering in Alloys: Phase Field and Monte Carlo Simulations: *Alphonse Finel*[1]; [1]Onera/CNRS, LEM, BP72, Chatillon, Cedex 92322 France

Structural or ordering transformations in alloys lead generally to the coexistence of many different phases or domains which form complex microstructures. At a mesoscopic time and space scale, most of the characteristics of these microstructures are determined by elastic effects, in particular growth laws of particle sizes and scaling properties. We will discuss these aspects in a continuous phase field approach valid for coherent systems. We will also present a general atomistic approach, based on Monte Carlo simulations, which, in simple cases, reproduces well-know properties (particle shape transitions, precipitates interactions) and which is able to deal with arbitrary local deformations, coherent or not, such as semi-coherent or incoherent interfaces, dislocations.

9:30 AM Invited
First Principles Concentration Functional Approach to Alloy Phase Stability: *George Malcolm Stocks*[1]; [1]Oak Ridge National Laboratory, Met. and Cer. Div., P.O. Box 2008-6114, Oak Ridge, TN 37831-6114 USA

In this paper I review the origins, successes, and limitations of the first principles concentration functional theory of ordering and phase stability developed in the early 80's [B. L. Gyorffy and G. M. Stocks, Phys. Rev. Letters {\bf 56}, 374 (1983)]. When evaluated in the mean field approximation and combined with the first principles local density approximation Korringa-Kohn-Rostoker coherent-potential-approximation (LDA-KKR-CPA) the, in principle exact, concentration functional approach provides a direct way of relating clustering and short range order to the underlying electronic structure of the disordered phase. I will review applications of the LDA-KKR-CPA concentration functional method to a number of classical problems in alloy theory and to the magnetic phase transition in ferromagnetic transition metals. In each case stress will be placed on understanding the underlying electronic mechanisms (Fermi surface nesting, band filling, charge transfer, ...) that give rise to the observed ordering behavior. I will place particular emphasis on what as been learned in recent years regarding screening in substitutionally disordered alloys and its affect on alloy phase stability based on large cell (100-1000 atom) simulations. Work supported by Office of Basic Energy Sciences, Division of Materials Sciences, US Department of Energy, under subcontract DEAC05—960R22464 with Lockheed-Martin Energy Research Corporation.

10:00 AM Invited
Master Equation Approach to Configurational Kinetics of Non-Equilibrium Alloys and Its Applications to Studies of Phase Transformations: Kirill Belashchenko[1]; Vladimir Dobretsov[1]; German Samolyuk[1]; *Valentin Vaks*[1]; [1]Russian Research Center, Kurchatov Instit., Moscow 123182 Russia

We review a series of works where we use the fundamental master equation to develop a consistent theoretical description of the evolution of non-equilibrium atomic distributions in an alloy. We derive exact equations for the temporal evolution of local concentrations and correlators of their fluctuations, as well as for the free energy of a non-equilibrium alloy. To solve these equations we employ the approximate methods analogous to those used in the equilibrium statistical physics including the kinetic mean-field method and the kinetic cluster methods. In particular, we develop a kinetic cluster field method being a kinetic analogue of the known cluster variation method which combines high accuracy in the description of thermodynamics with great simplification of the calculations. We also suggest a microscopic model to describe the influence of elastic forces on the phase transformation with a lattice symmetry change, such as the tetragonal distortion under $L1_0$ ordering. The developed methods are used for extensive studies of the microstructural evolution under various phase transitions including the decomposition of disordered alloys and orderings of B2, $D0_3$, $L1_2$ and $L1_0$ type both without and with phase separation as well as without and with elastic effects. These studies reveal a number of new and interesting microstructural effects, many of them agreeing well with experimental observations.

10:30 AM Break

10:45 AM Invited
Computational Investigations on the Microstructure Formation in Real Alloy Systems Based on the Phase Field Method: *Toru Miyazaki*[1]; Toshiyuki Koyama[1]; [1]Nagoya Institute of Technology, Dept. of Matls. Sci. & Eng., Gokiso-cho, Showa-ku, Nagoya 466-8555 Japan

The kinetic simulation based on the non-linear diffusion equation become very powerful method in fundamental understanding the dynamics of phase transformation with the recent remarkable development of computer. In the present study, we calculate the dynamics of microstructure changes in real alloy systems, such as Fe-Mo, Al-Zn, Fe-Al-Co and GaAs-InP based on the phase field method. The composition dependencies of atomic interchange energy are taken into account so as to be applicable for the phase diagram of the real alloy systems. The elasticity and mobility of atoms are assumed to depend on the local order parameters such as composition, degree of order, etc. Time dependent morphological changes of the microstructure such as formation of modulated structure by spinodal decomposition, strain induced morphological changes of precipitates, the order-disorder phase transition with phase decomposition, discontinuous precipitation will be demonstrated. The results simulated are quantitatively in good agreement with the experimental results in the real alloy systems.

11:15 AM Invited
Evolution of Microstructure and Defect Structure in Polytwinned Ferromagnets: *William A. Soffa*[1]; [1]University of Pittsburgh, Matls. Sci. and Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

The L10 family of ferromagnets are interesting magnetic materials from both a scientific and technical point of view. These intermetallics, which include CoPt, FePt, FePd and MnAl, form as stable or metastable phases in the vicinity of the equiatomic alloy compositions. The tetragonal structures are characterized by a high magnetocrystalline anisotropy with an "easy" c-axis and exhibit anisotropy constants in the range K1 ~ 107-108 ergs/cm3. The formation of the L10 phase from the parent phase in these alloy systems generates a characteristic polytwinned structure and defect structure which have important implications regarding the transformation mechanisms involved and the resultant structure-property relationships. In this paper, the development of the microstructure and defect structure characterizing the L10 polytwinned structures will be discussed and related to the nature of the disorder -> order transformation giving rise to these polytwinned ferromagnets. This work has been supported by NSF and DOE.

11:45 AM Invited

The Chessboard and Saw-Tooth-Like Morphologies in Decomposing Alloys: *Yann M. Le Bouar*[1]; Armen G. Khachaturyan[2]; [1]Commissariat a l 'Energie Atomique, DTA/SRMP, Cea-Saclay, Gif-Sur-Yvette 91191 France; [2]Rutgers University, Cer. and Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08855-0909 USA

The understanding and control of the microstructure evolution of multiphase alloys is of critical importance to synthesize advanced materials with given properties. During the last decades, the use of Transmission Electron Microscopy (TEM) has enabled a detailed description of the microstructure of numerous multiphase alloys. We focused here on puzzling microstructures obtained during ordering in the platinum-rich $L1_0$+$L1_2$ two phase region of the Co-Pt system. First, we present a series of TEM images (Dark Field and High Resolution images) to describe the complex microstructures of our Co_x-Pt_{1-x} alloys. Depending of the average concentration, our TEM images show that the microstructure may evolve either towards a chessboard or a platelet-like microstructure. Then, we present two computational methods, based on the continuum stochastic field kinetic equations or the microscopic Master Equation, able to describe a first order phase transition with a cubic -> tetragonal symmetry reduction. No a priori constraints are made on the possible configurations and the sequence of structural pattern. Finally, 2D computer simulations are performed for an elastically isotropic and homogeneous crystal. When the concentration of the alloy is chosen close to the middle of the $L1_0$+$L1_2$ two phase region, our continuous simulations predict the formation of a chessboard microstructure, whose edges are aligned in the elastically soft directions, and show that the coarsening of such a microstructure is only possible with the disappearance of an entire band of the pattern. When the concentration is close to the $L1_2$ stability region, our microscopic simulation explain the formation of the platelet or saw-tooth-like morphologies. All the simulation results are in excellent agreement with our experimental observation in the Co-Pt system.

International Symposium on Global Innovations in Materials Processing and Manufacturing: Stereolithography and Selective Laser Sintering

Sponsored by: Materials Processing and Manufacturing Division,

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

Tuesday AM — Room: Canal E
March 14, 2000 — Location: Opryland Convention Center

Session Chair: David L. Bourell, University of Texas-Austin, Austin, TX 78745 USA

8:30 AM

Solid Freeform Fabrication: A United States Navy Perspective: *Khershed P. Cooper*[1]; George Spanos[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech., Code 6324, 4555 Overlook Ave. S.W., Washington, DC 20375 USA

ONR in cooperation with other federal government agencies such as DARPA, OSD and NSF has been sponsoring research in Solid Freeform Fabrication (SFF) technology since it's early days. These agencies have played a major role in promoting the understanding and development of manufacturing processes such as Fused Deposition Modeling (FDM), 3-D Printing (3DP), Selective Laser Sintering (SLS), Shape Deposition Modeling (SDM), Stereolithography (SLA), Selected Area Laser Deposition (SALD), Direct Photo Shaping and others. SFF projects have been pushed to experiment with various homogeneous materials and combination of materials (functionally graded materials). Technologies are finding their way into industry for customized prototypes and even for functional components. But theoretical and experimental work remains to be done and new opportunities in diverse areas such as electronic packaging and biomedical engineering are emerging. This talk will survey some of the projects supported by ONR and other agencies and discuss some possible Navy applications.

9:00 AM

Effect of Build-Plane Orientation on the Mechanical Properties of Parts Made by Fused Deposition Modeling (FDM) and by Stereolithography (SLA): *Nancy S. Losure*[1]; Ward Jensen[2]; [1]Mississippi State University, Swalm Schl. of Chem. Eng., Mississippi State, MS 39762 USA; [2]Oreck Manufacturing, 21180 Oreck Blvd., Long Beach, MS 39560 USA

Rapid prototyping techniques have traditionally been used to produce parts to validate design dimensions and assembly procedures. Stereolithographic (SLA) techniques produce parts with excellent surface finish and detail, but limited strength and problems with dimensional stability. On the other hand, a relatively new process, Fused Deposition Modeling (FDM), produces parts from an engineering resin, ABS, with good surface finish, much higher strength and excellent dimensional stability. It is important that the mechanical properties

of prototype parts be understood, particularly with respect to the direction of build so that users can maximize the benefit from a rapid prototyping program. In this work, we will produce tensile, bending and impact specimens in which the build-plane is perpendicular to each of the dimensional axes, in order to determine how the build-plane orientation affects the strength and stiffness of prototype parts produced by FDM and SLA.

9:20 AM

Mechanical Alloying Polymer Blends for Selective Laser Sintering: *Julie Patricia Martin*[1]; Ronald George Kander[1]; [1]Virginia Tech, Matls. Sci. & Eng. Dept., 213 Holden Hall, Blacksburg, VA 24061-0237 USA

In this work, mechanical alloying (MA) is presented as an effective alternative to traditional coating methods for creating co-continuous phases during selective laser sintering (SLS). Specifically, the morphology of mechanically alloyed polymer blends for use in the SLS process is investigated in this research. By varying the charge ratio, time, and temperature of the MA process, the phase domain size of the resulting composite powder can be manipulated and the physical, mechanical, and electronic properties of the blend altered. The mechanically alloyed powder can then be selectively laser sintered into parts containing co-continuous phases. Although polymer/polymer composite morphologies are studied here, the MA process is also a viable technique for creating SLS powders using ceramics or metals.

9:40 AM

Selective Laser Sintering of Polymer-Polymer, Polymer-Metal, and Polymer-Ceramic Composite Powders Made by Cryogenic Mechanical Alloying: *Jeffrey Patrick Schultz*[1]; Ronald George Kander[1]; Carlos Tres Ayala Suchicital[1]; [1]Virginia Tech, Matls. Sci. and Eng., 213 Holden Hall, Blacksburg, VA 24061-0237 USA

Cryogenic mechanical alloying (CMA) offers a new means of producing composite powders for selective laser sintering (SLS). Unlike composite particles made by a coating process, both materials are continuous throughout the particle. Consolidation of these composite particles via SLS offers the possibility of forming a co-continuous microstructure in parts produced by SLS. The work presented is an initial investigation into the mechanical properties and microstructural characteristics of three SLS material systems formed by CMA: a polymer-polymer, polymer-ceramic, and polymer-metal system. The polymer-polymer composite has applications in fabrication of functional prototypes and direct manufacturing, and the polymer-metal polymer-ceramic systems are both used for fabrication of green structures and direct manufacturing.

10:00 AM

Direct Laser Fabrication of High Performance Metal Components via SLS/HIP: Suman Das[1]; *Martin Wohlert*[1]; Joseph J. Beaman[1]; David L. Bourell[1]; [1]University of Texas at Austin, Mech. Eng., MC C2200, Austin, TX 78712-1063 USA

This paper focuses on recent advances in direct freeform fabrication of high performance metal components via selective laser sintering (SLS). The application, known as SLS/HIP, is a low cost manufacturing technique that combines the strengths of selective laser sintering and hot isostatic processing (HIP) to rapidly produce low volume or "one of a kind" high performance metal components. The advantages of in-situ encapsulation include elimination of a secondary canning step and container material, no container-powder interaction, reduced pre-processing time, and reduction in post-processing steps compared to HIP of canned parts. SLS/HIP is currently being developed for superalloy 625 and Ti-6Al-4V. Microstructure and mechanical properties of material processed by SLS/HIP are comparable to conventionally processed material. Results of SLS/HIP development for superalloy 625 are presented.

10:20 AM Break

10:40 AM

Selective Laser Sintering with Meso-Scale Features: *Nicole Harlan*[1]; Seok-Min Park[1]; Joseph J. Beaman[1]; David L. Bourell[1]; [1]University of Texas at Austin, Mech. Eng., MC C2200, Austin, TX 78712-1063 USA

Recent work in Selective Laser Sintering of materials at the University of Texas at Austin demonstrates the capability to produce parts with fine features of the order of 100 µm to 200 µm. Zirconia powder was pre-processed into spherical particles, laser sintered with a sacrificial polymer binder, infiltrated and post-sintered to higher density. Optical micrographs show that hole sizes of 180 µm are possible in fully ceramic components. Several examples illustrating the potential of Selective Laser Sintering of parts with meso-scale features will be presented.

11:00 AM

Zirconia Molds for Titanium Casting: *Nicole Harlan*[1]; Seok-Min Park[1]; David L. Bourell[1]; Joseph J. Beaman[1]; [1]University of Texas at Austin, Mech. Eng., MC C2200, Austin, TX 78712-1063 USA

A combination of selective laser sintering and colloidal infiltration has been used to create "partially stabilized" zirconia molds for titanium casting. The mold material system was chosen for its low reactivity with molten titanium and thermal shock resistance. The base material, stabilized zirconia mixed with a copolymer binder, is laser sintered into the desired green shape. The binder is removed and replaced by zirconia. The fired parts show graded porosity from a dense surface to a porous interior. The average density of the fired parts can be increased to twice that of the green density. Surface roughness (Ra) is less than 10 µm and flexural strength is sufficient for high temperature casting. A half-scale casting mold for the head of a human femur bone was produced using laser scanned data.

11:20 AM

Solid Free-Form Fabrication of Refractory Metal Components: *Gary K. Lewis*[1]; Joe C. Fonseca[1]; Ron B. Nemec[1]; Tom N. Taylor[1]; Paul Burghardt[1]; [1]Los Alamos National Laboratory, Mail Stop G770, Los Alamos, NM 87545 USA

Directed Light Fabrication, a solid free-form fabrication process, has been shown feasible to build layered components from almost any metal in a single step. This process is viewed as particularly beneficial to the fabrication of refractory metal components by saving multiple powder consolidation, thermo-mechanical processing, joining, and machining steps used in conventional processing. However, refractory metal powders, in contrast to many lower melting point powders that are typically gas or water atomized, are produced by a chemical reduction, precipitation process. DLF processing of these refractory metal precipitate powders results in porosity in the solidified product that is being traced back to trace elements in the powder. Additional powder processing prior to deposition to remove or reduce undesired elements has proven effective in eliminating porosity. Characterization of powders and deposits and optimization of DLF process parameters is leading to successful fabrication of refractory metal components.

11:40 AM

SFF Using Inverted Projection of Liquid Metal Droplets: *Dawn R. White*[1]; Sankaran Subramaniam[1]; Larry Jepson[2]; [1]Ford Research Laboratory, P.O. Box 2053, MD 3135 SRL Bldg., Dearborn, MI 48121-2053 USA; [2]University of Texas-Austin, SFF Lab., Austin, TX 78712-1063 USA

A number of droplet based free form fabrication techniques have been developed. Without exception, these involve the formation and transfer of a droplet to a substrate beneath the droplet origination site. Under these conditions, gravity has the effect of causing droplet spreading upon impingement. Factors such as surface tension, viscosity of the droplet, droplet liquid fraction, etc. affect the degree of spreading and wetting involved, however, the result is that part geometry is difficult to control precisely. Thus, SFF parts made using droplets often have fairly rough or irregular surfaces, which require finish machining for many applications. If however, the droplet is projected upwards, and allowed to impinge and hang on a "superstrate," gravity and surface tension interact to produce an elongated droplet, with a smooth side. As these hanging droplets accumulate to for an SFF object, our experiments show that it has a much smoother, and dimensionally controllable sidewall, than an object produced in the conventional manner. This paper presents a numerical analysis of the hanging droplet approach to SFF. In addition, experiments were performed using projected aluminum and steel droplets. The results of experiments conducted on single and multiple droplets, and bulk features are also presented.

International Symposium on Iridium: Applications

Sponsored by: Structural Materials Division, Refractory Metals Committee

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

Tuesday AM — Room: Jackson A/B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Richard D. Lanam, Engelhard-CLAL LP, Carteret, NJ 07008 USA; N. I. Timofeev, Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Research Center, Ekaterinburg 620014 Russia

8:30 AM

Characterization of Sputter-Deposited Iridium Oxide Coatings for Medical Implants: *T. Loose*[1]; M. Frericks[1]; T. Giesel[1]; G. Herklotz[1]; [1]W.C. Heraeus GmbH & Company KG, Heraeusstrasse 12-14, Hanau D-63450 Germany

Iridium oxide coatings were produced by d.c. reactive magnetron sputtering of a metallic iridium target in an Ar/O_2 atmosphere. The properties of the deposit depend on the sputter parameters (time, power, oxygen flow, temperature) as well as the substrate material (titanium, stainless steel). Cyclo-voltametry was used to investigate electrochemical properties. Corrosion in 0.9% NaCl-solution was measured by potentiostatic voltametry at the corrosion potential of the uncoated surface. The composition of the iridium oxide was determined by auger electron spectroscopy, the surface morphology by scanning electron microscopy. Adherent, crack free coatings with good mechanical and corrosion stability can be achieved using a metallic interlayer.

8:50 AM

Production of Iridium Crucibles by Electrolysis of Molten Salts: *N. I. Timofeev*[1]; V. E. Baraboshkin[1]; N. Saltykova[2]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia; [2]Institute of High Temperature Electrochemistry, Ekaterinburg, Russia

Iridium has the high melting point and unique resistance to aggressive oxidation environments at high temperatures. It is used as container material for growing of high temperature oxide crystals. The mechanical treatment of iridium has essential difficulties because of its considerable hardness. In the Ekaterinburg Non-Ferrous Metals Processing Plant, the electrolysis was performed in the hermetic electrolyzer in argon atmosphere. The electrodeposition of iridium was carried out in the melt of eutectics of NaCl-KCl-CsCl containing iridium chlorides at 500-600°C. The most optimum method can be considered as the combination of the electrolytic iridium refining of metal- and non-metal impurities with the preparation of iridium articles using the method of galvanoplastics. Therefore, the dissoluble anodic material can be used not only in form of pure iridium but also as its scrap. The cathodic matrices were made of graphite having the form of articles. The anodic current density should be as low as to avoid salt passivation of iridium anode. The reversal current and the rotation of the cathode during electrolysis decreased the grain size of iridium compact layer (coating) and smoothed its surface. To avoid the appearance of the structure coating defects (excrescencies, pores) several technological procedures were used; one of which was the preliminary thermal treatment of graphite matrixes in aim to remove the gases from them. After the electrodeposition of iridium coatings onto matrix the latter was destroyed. The roughness of the internal surface of crucible corresponded to the degree of mechanical treatment of the graphite matrix. The electrodeposited iridium has high plasticity and the same density as iridium castings. Microhardness of electrolytic iridium is 320-340 kg/mm^{2}and Vickers hardness is 240-280 kg/mm^2. The purity of iridium is 99.98-99.99% and the main impurities are the metals of platinum group (Pt, Rh). The method of galvanoplastics allows producing various articles (tubes, rings, and disks). It is an ecologically pure and practically waste-free technology.

9:10 AM

Experimental Technology for Production of Disks from High Strength Iridium: *A. V. Ermakov*[1]; A. V. Sedavnykh[1]; L. G. Grokhovskaya[1]; S. G. Tretiakova[1]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia

The sources of radioisotope Ir^{192} are manufactured by radiation treatment of iridium targets in reactor. Iridium discs for this purpose should meet hard conditions, such as (1) no contaminants should be contained in the metal; (2) concavity of discs is about 10^{-2} mm for diameter of 1.5-3.0 mm and thickness of 0.1-0.3 mm; and (3) their edges should not crumble after processing. Technology for manufacture of these discs has been elaborated. Massive iridium crystals, whose purity is 99.9%, are used as workpieces. Mechanical treatment of crystals at high temperatures allows obtaining sheets with grain size of 5 x 10^{-2} mm. Fine grain iridium can be stamped to discs and calibrated during one technological operation. Besides, these discs do not crumble. Another way for suppressing the crumble is the coating of disc edges by aluminum.

9:30 AM

Radioactive ^{192}Ir Gamma-Sources Made from Enriched ^{191}Ir: W. Borneman[1]; M. Frericks[2]; *D. F. Lupton*[2]; H. Rakhorst[3]; [1]Malinckrodt Medical B.V., Petten Netherlands; [2]W.C. Heraeus GmbH & Company KG, Heraeusstrasse 12-14, Hanau D-63450 Germany; [3]Urenco Nederland B.V., Almelo, Netherlands

Radioactive ^{192}Ir sources play an important role in medical applications, especially brachytherapy, and in industrial non-destructive testing (NDT). Due to the nature of nuclear reactors with variable neutron fluxes and sometimes unplanned shutdowns, the supply of iridium sources has not only been under stress, but also the guarantee of a minimum yield (Ci/gram) has been difficult. Current sources are made by irradiating natural iridium, in solid metal form, in a nuclear reactor. Natural iridium, however, contains only 37% ^{191}Ir that is activated to ^{192}Ir, the remainder being ^{193}Ir which is not activated. Urenco has a proprietary gas centrifuge technology that is extensively used for enriching ^{235}U for commercial nuclear power plants. A successful R&D programme has adapted this technology to enrich ^{191}Ir to at least 85% using the process gas IrF_6. By means of a hydrogen reduction treatment, this compound is converted to high purity iridium black. The ultra-fine iridium metal powder is consolidated to compact products by Heraeus. The iridium black is induction melted and then remelted under high vacuum by an electron beam process, to ensure ultra-high purity. The fine powder can also be consolidated by powder metallurgical techniques, but experience has shown this to be a less satisfactory route. As a result of the extremely high purity, forming operations can be carried out more readily than with conventional grades of iridium. The advantage of enriched ^{191}Ir is a much more efficient use of reactor neutrons, resulting in much smaller sources, or sources with much higher specific activity. These efficiency gains outweigh the higher costs of enrichment and pellet manufacturing. Irradiation results have verified a near doubling of specific activity of irradiated 85% ^{191}Ir versus natural iridium giving the following advantages: (1) no minimum yield problem, (2) unplanned shutdowns of reactors do not jeopardize the continuity of supply, and (3) longer shutdown periods of reactors can be overlapped.

9:50 AM

Iridium and Iridium Alloy Utilization in Ignition Devices: *L. F. Toth*[1]; D. A. Toenshoff[1]; [1]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

The high melting and boiling points, corrosion and spark erosion resistances of iridium and iridium alloys make these ideal candidates for electrodes in ignition devices for use in the hostile environment of a typical combustion chamber. The demands for ever increasing envi-

ronmental and combustion efficiency and longer service life in automobile engines strongly suggest the utilization of iridium and/or its alloys as spark plug electrodes. This paper will examine some of the thermomechanical properties and processing of iridium materials, their incorporation and behavior in various ignition devices, and an insight into some of the obstacles encountered in the use of iridium and potential methods of overcoming them.

10:10 AM Break

10:20 AM
The Use of Iridium in High Temperature Thermometry: *J. Grossi*[1]; [1]Engelhard-CLAL, 4025 Clipper Ct., Fremont, CA 94538 USA

One of the lessor known industrial uses of iridium is the application of it and its alloys in high temperature thermometry. This use is becoming increasingly important with the need to accurately measure higher temperatures especially under oxidizing conditions. This paper will focus on current applications such as gas turbine instrumentation, temperature measurement for crystal growth and industrial furnace control. In addition, practical error considerations; such as utilization of proper extension wires and data acquisition recommendations will be discussed.

10:40 AM
Iridium/Rhenium Combustion Chambers for Chemical Propulsion: *A. J. Fortini*[1]; R. H. Tuffias[1]; R. B. Kaplan[1]; A. J. Duffy[1]; B. E. Williams[1]; J. W. Brockmeyer[1]; [1]Ultramet, 12173 Montague St., Pacoima, CA 91331 USA

Most satellites placed in geosynchronous orbit rely on silicide-coated niobium combustion chambers for apogee topping maneuvers. These chambers typically operate at temperatures between 1300 and 1400°C. At higher temperatures, rapid oxidation of the silicide takes place and catastrophic failure occurs. Current state-of-the-art combustion chambers now comprise a rhenium structural shell protected by a thin film (typically 0.003") of chemical vapor deposited (CVD) iridium on the interior. Iridium is ideal for this application due to its excellent oxidation resistance, high melting point, and thermal expansion match with rhenium, and CVD is the preferred processing technique because the resulting coating is very pure and free of cracks, porosity, pinholes, and other defects. Using this architecture, combustion chambers can now be operated at temperatures well in excess of 1900°C, which yields a dramatic increase in performance.

11:00 AM
The Use of Iridium for Jewelry: *C. Volpe*[1]; K. Vaithinathan[2]; R. Lanam[2]; [1]Tiffany & Company, 143 Sparks Ave., Pelham, NY 10803 USA; [2]Engelhard-CLAL, 700 Blair Rd., Carteret, NJ 07008 USA

The principle use of iridium for jewelry has been as an alloying addition to platinum to increase hardness and improve resistance to wear. The compositions and application of the platinum-iridium alloys will be discussed. A recent development is the use of iridium itself in jewelry. Iridium's properties of being very hard and corrosion resistant make it a desirable material for jewelry. Through near-net shape and surface enhancing processes unique designs can be achieved. Examples of the traditional Pt-Ir and new iridium uses will be shown.

11:20 AM
Iridium as Container Material for Oxide Crystal Growing: N. I. Timofeev[1]; B. Dorogovin[2]; *A. V. Yermakov*[1]; S. Yu. Stepanov[2]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia; [2]VNIISIMS, Alaxandrov, Russia

Iridium is the sole material for containers, where large single crystals of aluminum-yttrium garnet AYG ($Y_3Al_5O_{12}$), gallium-gadolinium garnet GGG ($Gd_3Ga_5O_{12}$), gallium-lanthanum silicate ($La_3Ga_5SiO_{14}$), and other oxides could be grown. Experience has shown that crucible is the key element of growing technology, which determines purity and quality of crystals. Iridium containers with size up to 200 mm in diameter, 200 mm in height, thickness of walls is 2 mm could be manufactured either traditional technology (rolling of sheets, stamping of cans and welding of crucibles) or galvanoplastic means (production of jointless crucibles). Welding crucibles from single crystal workpieces (wall thick of 2 mm) possess highest working resource for iridium containers: their life-time in GGG is 2000 hours and in AYG is 800 hours, while lifetimes of galvanoplastic ones are 1000 hours for GGG and 120 hours for AYG. Resource of welding containers from electron beam melted iridium is less than for "single crystal" crucibles, but higher than for ones.

11:40 AM
Iridium and Its Application-Present Status in Japan: *T. Maruko*[1]; [1]Furuyametals Company Limited, 1915 Morizoesima, Shimodateshi, Ibaraki 308-0861 Japan

Iridium demand is tremendously increasing, especially since 1995 in Japan, with an increasing variety of applications. One application is the newly developed catalyst to reduce automotive engine emission. Another is in the electrochemical field where about 25% of total iridium demand is consumed for electrodes used in industrial electrolysis, chlor-alkali electrolysis, and electro-galvanizing among others. Demand for the iridium crucibles and parts comprises about 3% of total supply. Those include applications of the compound single crystal growth for advanced electronics devices, YAG laser tips, and SAW devices. Growth in iridium demand in the near future is expected. Due to its high refractory characteristics, iridium finds application in thermocouples, rocket parts, gas-turbine blades, glass manufacturing, and spark plugs. Use of ferroelectric memory so called F-RAM, having superior nonvolatility, speed, and volume and requiring less power consumption, is expected to grow requiring iridium as a best candidate per production of various materials such as PZT and SBZ. We have succeeded in developing various materials and overcoming various difficulties in processing through the special processing techniques including microstructure control, improved purification, and special additives. High efficiency in the recovery iridium is very important to minimize tremendous price changes caused by imbalance in demand and supply associated with increased applications of iridium. New recovering and recycling processes for iridium are discussed.

Kleppa Symposium on High Temperature Thermochemistry of Materials: Session III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Extraction & Processing Division, Thermodynamics & Phase Equilibria Committee, Process Fundamentals Committee

Program Organizers: Ray Y. Lin, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA; Y. Austin Chang, University of Wisconsin, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Dr. Susan Meschel, The University of Chicago, Chicago, IL 60637 USA; Ramana Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487 USA

Tuesday AM — Room: Lincoln E
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Ray Y. Lin, University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA; Bruce MacDonald, National Science Fundation, Div. of Matls. Rsch., Arlington, VA 22230 USA

8:30 AM
Solubility and Raman Spectra of Niobium (V) Fluoro and Oxo Fluoro Complexes Formed in LiF-NaF-KF-Melts: *Aasmund F. Vik*[1]; Terje Ostvold[1]; Vassilis Drakopoulos[2]; George N. Papatheodorou[2]; [1]Norwegian University of Science and Technology, Dept. of Chem., Trondheim N-7491 Norway; [2]Institute of Chemical Engineering and High Temperature Chemical Processes, Forth, P.O. Box 1414, GR-26500, Patras, Greece

The solubility of Nb (V) as a function of the Na_2O concentration in the ternary eutectic LiF-NaF-KF (FLiNaK) melt at 700°C has been determined. The Raman spectra at different oxide concentrations and temperatures were also recorded. Based on chemical analysis of melt samples and the observed Raman bands, suggestions related to the Nb-O-F complexes formed in the melt are given. In FLiNaK containing 0.22 mole kg^{-1} K_2NbF_7, the following observations were recorded at varying total molar ratios of oxygen to niobium in the melt + the solids formed, n^oO/n^o_{Nb}: 1. $n^oO/n^o_{Nb} < 2$; all Nb (V) and O^{2-} was dissolved. The Raman spectra indicated the presence of monomeric NbF_7^{2-} at low oxide concentrations, which reacted with oxide to form monomeric $NbOF_5^{2-}$ when n^oO/n^o_{Nb} was increasing. $NbO_2F_4^{3-}$ was formed upon further additions of oxide and was the dominating specie at $n^oO/n^o_{Nb} = 2$. 2. $2 < n^oO/n^o_{Nb} < 3$; a solid of the type $AlkNbO_3$ was formed; $n^oO/n^o_{Nb} = 3$; a minimum in the Nb (V) and O^{2-} solubilities were observed. The structure of the species in the melt was not easy to establish due to a very weak Raman signal. $3.3 < r < 4$; the $AlkNbO_3$(s) previously formed dissolved, possibly with the formation of both corner and edge sharing distorted NbO_6 octahedra. The solubility of Nb_2O_5 in FLiNaK was measured as a function of temperature (550°C to 800°C), and the dissolution mechanism seems to be given by the reaction: $Alk^+ + Nb_2O_5(s) + 4F^- = NbO_2F_4^{3-} + AlkNbO_3(s)$. H^o_{sol} for the reaction (based on concentrations in mole kg^{-1}) was found to be 89 $kJmol^{-1}K^{-1}$.

9:00 AM

High Temperature Electrochemical Study of the Na_2O-MoO_3 System: *Ray Y. Lin*[1]; John F. Elliott[2]; [1]M.L., Dept. of Matls. Sci. and Eng.; [2]University of Cincinnati, Cincinnati, OH 45221 USA

Physical chemistry of the Na_2O-MoO_3 system was studied using a high temperature electrochemical cell with Na, β-alumina as the solid electrolyte. The reference electrode is a solid mixture of tungsten, tungsten sulfide and sodium sulfide. The cell may be expressed as the following. W(s), WS2(s)| Na+| O_2(g), Pt(s)Na_2S(s) β-alumina Na_2O-MoO_3 melt. The activity of Na_2O in the Na_2O-MoO_3 melt in the composition range from 50.87 to 92.18 mol% MoO_3 and temperature from 890 to 1230 K was determined using this cell. Partial molar enthalpy of mixing for Na_2O in the melt was evaluated from the temperature dependence of the activity data using the Gibbs-Helmhotz equation. The value agrees well with those reported in the literature from calorimetric measurements. The activity of MoO_3 in the melt as a function of temperature and melt composition was also evaluated from the Gibbs-Duhem equation with the help from the Na_2O-MoO_3 phase diagram.

9:30 AM

Comments on the Formation Thermodynamics of the Rare Earth Compounds with the 15th and 16th Group Clements: *R. Ferro*[1]; G. Borzone[1]; [1]Universita degli Studi di Genova, Dept. di Chimica e Chimica Industriale, Via Dodecaneso, Genova 31-16146 Italy

In the framework of a description of the general alloying behaviour of the rare earth ® metals, their thermodynamics of reaction with the 15th and 16th group elements of the Periodic Table is reviewed. Also on the basis of our obtained laboratory results, special attention is given to the thermochemistry of formation of the arsenides, antimonides, bismuthides, selenides and tellurides of the R elements. The properties of these substances are also discussed and compared with those of compounds formed by other transition elements similar to the R metals (actinides, etc.). Problems met in the thermochemical characterization and in the experimental investigation of this family of substances are finally highlighted and discussed.

10:00 AM Break

10:10 AM

Thermodynamic Investigations of Pseudobinary Chalcogenide Systems: *M. Shamsuddin*[1]; [1]Banaras Hindu University, Instit. of Tech., Dept. of Metallu. Eng., Varanasi 221-005 India

In recent years thermodynamic measurements on several pseudobinary chalcogenide systems, viz. CdSe-CdTe, ZnTe-CdTe, HgTe-CdTe, ZnSe-ZnTe and PbSe-PbTe and PbSe-PbTe using fused salt galvanic cell technique have been conducted. Thermodynamic data show that these systems do not follow regular solution model and also do not satisfy Darken's formalism. Hence thermodynamic behaviour of these systems have been analyzed in the light of Darken's stability and excess stability functions. The stability and excess stability together with ideal stability, relative stability and relative excess stability parameters have been calculated at different temperatures. The continuous variation of Darken's stability and constancy of excess stability functions with composition confirm that ZnTe-CdTe, HgTe-CdTe, ZnSe-ZnTe and PbSe-PbTe systems consist of single phase field throughout the entire range of composition. On the other hand, in the CdSe-CdTe system, sharp changes in the variation of stability and excess stability functions near $x_{CdTe} = 0.4$ establish that the system consists of two terminal regions (i) $0 < x_{CdTe} \leq 0.35$ and (ii)$0.45 \leq 1.0$, in which the thermodynamic behaviour is relatively simple. The two terminal regions are connected by a narrow transient or central region ($0.35 \leq$ x CdTe ≤ 0.45), in which the behaviour appears to be complicated.

10:40 AM

Thermochemistry and Modelisation in Oxides: *G. Boureau*[1]; [1]Universite Pierre et Marie Curie, Lab. de Chimie Physique, Matiere et Rayonnement, 11 Rue Pierre et Marie Curie, Paris, Cedex 05 75231 France

In the present paper we show that the informations won from experimental thermodynamics are essential in the understanding of oxides: We shall investigate the following fields: energy of formation of defects in bulk oxides (1) and at the interfaces; (2) both, direct experimental investigation and ab initio studies of oxygen vacancies are difficult. The help of thermodynamics is invaluable in providing severe constraints. Ionic and electronic transport. (3, 4). Without any additional parameters, a purely thermodynamic analysis of experimental data provide a good understanding of electronic transport in some particular cases (polaronic models). Statistical thermodynamics (5). Such an approach needs reliable inputs which may have a number of origins. In some cases, thermodynamics allow to make guesses about the nature of defects and of interactions. Comparison with microscopic observation is particularly useful. Then we shall discuss links between thermodynamic data and charge transfer in silicium oxides and in germanium oxides.

11:10 AM

Thermodynamic Considerations on Solid Oxide Fuel Cell Materials: *Harumi Yokokawa*[1]; Katsuhiko Yamaji[1]; Teruhisa Horita[1]; Natsuko Sakai[1]; Hideyuki Negishi[1]; [1]National Institute of Materials and Chemical Research, Energy-Related Matls. Grp., 1-1 Higashi, Tsukuba, Ibaraki 305-8565 Japan

Thermochemical properties of ZrO_2-based ceramics and perovskite-type oxides were analyzed so as to obtain the good correlation with ionic properties and to reproduce the experimentally determined phase relations. For ZrO_2-based ceramics, the interaction parameters for cubic phases were derived from the phase diagram calculation in the absence of the experimental thermochemical properties. Those parameters which were derived for respective valences of dopants show the good correlation with ionic radii of dopants. This allowed us to calculate solubility of transition metal oxides over a wide range of oxygen patential, in which the valence of the transition metal ions changes drastically. The calculated solubility was compared with experimental values determined by Sasaki et al. Good agreement was obtained except for the zirconium vanadium oxygen system.

11:40 AM

Formation Energies of Molybdenum Borides Measured by Voltammetry: *G. Kaptay*[1]; J. Sytchev[1]; [1]University of Miskolc, Miskolc, Egyetemvaros 3515 Hungary

For measurements the molten NaCl-KCl-NaF (5 mol%)-KBF_4 system has been used at 973K, under argon gas. Experiments were performed in the glassy-carbon crucible, which served as counter electrode and reference electrode, as well. Cyclic voltammetric curves were taken at different scan rates from 0.001 V/s till 10 V/s. When silver was used as cathode, a one-step, 3-electron boron deposition was observed as a reversible electrochemical process. However, when molybdenum wire was used as cathode material, several pre-peaks appeared at more positive potentials compared to deposition of B on Ag cathode. The pre-peaks are due to the formation of molybdenum borides of different stoichiometry. The potential difference between the B-peak on the Ag-cathode and between the pre-peaks observed on the Mocathode are related to the formation energies of different mo-

lybdenum boride phases. Calculations proved, that our results are very close to the calorimetric results obtained by Meschel and Kleppa [3]. Thermodynamic properties of all molybdenum borides have been estimated by us using our voltammetry data coupled with information given on the Mo-B phase diagram, and taking into account the calorimetric result of [3].

12:10 PM

Equilibrium in the Liquid Fe-Al-N System: *G. Kaptay*[1]; M. S. Yaghmaee[1]; G. Janosfy[1]; [1]University of Miskolc, Miskolc, Egyetemvaros 3515 Hungary

Abstract text not available

Magnesium Technology 2000: Automotive Issues and Recycling

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

Tuesday AM — Room: Bayou C
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Gerald S. Cole, Ford Motor Company, Ford Rsch. Labs., Dearborn, MI 48121-2053 USA

8:30 AM

Conductivity Measurements on Ingots of Magnesium Die-Casting Alloys: *Carlton D. Fuerst*[1]; Cameron J. Dasch[1]; [1]General Motors R&D Center, Matls. & Process. Lab., MC 480-106-224, 30500 Mound Rd., Warren, MI 48090-9055 USA

Magnesium metal suppliers use a variety of methods to establish metal cleanliness, all of which seek to take advantage of differences between the properties of embedded oxide particles and those of the matrix (magnesium metal). Electrical conductivity measurements are a fast and sensitive means of nondestructive evaluation that offers the potential of detecting the electrically insulating oxide particles embedded in the conductive matrix. However, the results presented in this report reveal that other microstructural features also have a large impact on the conductivity of magnesium die-casting alloys. Aluminum concentration and microstructural porosity are two important variables governing conductivity. A 1 wt% increase in the aluminum level produced a 6-8% decrease in the conductivity of samples studied. This dependence on chemistry is large enough to make it easy to distinguish between the low-aluminum AM-type alloys (AM50A and AM60B) and the high-aluminum AZ91D alloy, using conductivity measurements. Increases in porosity also cause a decrease in conductivity; specifically, conductivities dropped about 3% for each 1% increase in porosity. Given its sensitivity to both chemistry and porosity, it is unlikely that conductivity could be used to directly measure oxide inclusions in ingots. Further work would be necessary to determine its usefulness for specially cast samples where chemistry and porosity can be tightly controlled.

8:55 AM

Observations of Intermetallic Particle and Inclusion Distributions in Magnesium Alloys: *John M. Tartaglia*[1]; John C. Grebetz[2]; [1]Climax Research Services, 39205 Country Club Dr., C-40, Farmington Hills, MI 48335-5718 USA; [2]Daimler Chrysler Corporation, Auburn Hills, MI USA

As part of an international program to qualify magnesium alloy ingots as feed stock for die castings, numerous studies have been conducted on the microstructures of AM50, AM60, and AZ91 virgin and recycled ingots. The studies have focused on intermetallic particles and inclusions distributed throughout the ingot bulk and concentrated in surface defect regions such as folds. The studies have included optical macrography and micrography to document the particle distributions; automated optical image analysis for quantifying the particle volume fraction and size; and scanning electron microscopy and energy dispersive spectroscopy to characterize the elemental content of the particles and surrounding matrix. Several exemplary case studies will be presented in this talk.

9:20 AM

Utilization of Centrifugal Casting in Recycling of Magnesium Alloy Scraps: *A. Arslan Kaya*[1]; Serdar Sevik[1]; Havva Zeytin[1]; [1]Tubitak-Mam, Marmara Rsch. Ctr., Matls. and Chem. Tech. Instit.-Matls. Grp., P.O. Box 21, Gebze-Kocaeli 41470 Turkey

The total production and consumption figures of magnesium metal indicate that recycling will soon become an integral part of the magnesium industry. Recycling of magnesium alloys necessitates the remelting of scrap metal and removal of inclusions as well as adjusting the chemistry before it can be reused for casting. This requires the development of an effective recycling technology for magnesium scrap. This study has been undertaken to assess the feasibility of recycling magnesium alloy AZ91 scrap by utilizing centrifugal casting process as part of the recycling technology. The goal was to concentrate the inclusions in the outer skins of billets during centrifugal casting for subsequent removal. Centrifugal casting parameters were varied to understand their effects on the distribution of inclusions throughout the cross section of billets. Light and scanning electron microscopy aided with microanalysis were used to determine the size, distribution and the types of inclusions. A critical assessment has been made regarding the applicability of the method as a recycling process.

9:45 AM

Magnesium Melting/Casting and Remelting in Foundries: *Horst Dörsam*[1]; [1]StrikoWestofen GmbH, Lorenz-Schott-Strasse 5, D-55252 Mainz-Kastel, Postfach 5909, Weisbaden D-65049 Germany

New furnaces and casting plants are described, having a capacity of 100 to 5400 kg at melting capacities of 100 to 1500 kg magnesium per hour. Tiltable, gas-heated furnace designs are also available which are mostly used as melting furnaces for clean recycling material for subsequent casting in ingot moulds. The application of modern ingot heating and charging machines is shown which prevent that humid material is charged into a liquid melt. Units for charging of ingots with the most different dimensions are available. The capacity is, for example, 36 ingots which can be applied by hand in a ferry‘s wheel. A preheating takes place and an automatic charging into the crucible furnace in accordance to the casting capacity. Throughputs of up to 1000 kg magnesium per hour at an average ingot size of 8 kg are possible. A mixed gas control cabinet enables the mixture with one another of up to three different mediums in a previously exactly defined relation. Normally, dry air is mixed with SF6 or SO2. CO2 as third gas can also be added. Thus, it is possible to constantly mix the desired protective gas into the furnace to cover the magnesium melt. A survey of the magnesium pumps available on the market with experimental values from the practical casting is given, especially those from Norsk Hydro and Dynarad. Casting quantities of 0.5 to 30 kg per shot are possible. The desired casting quantities can be preset and are casted directly into the filling chamber of the diecasting machine by means of the casting tube or in another casting form. Existing remelting plants are described and a preview is given about the developments which are to be expected, as for example the “inhouse recycling” plant with ingot casting machines and for liquid charging towards the pouring furnaces which is getting more and more important. New developments of furnace series with optimized handling systems and geometric sizes to minimize the respective space required for the furnaces are shown as well as economical and environmental aspects will be described in brief, especially for the trendy large casting parts in high pressure diecasting.

10:10 AM Break

10:20 AM
Filling and Solidification Modeling of Noranda's Magnesium Wheel Casting Process: *Randy Sheng*[1]; Don Argo[1]; [1]Noranda Technology Centre, 240 Hymus Blvd., Pointe-Claire, Quebec H9R1G5 Canada

Since 1993, Noranda has made significant efforts in developing expertise in the area of aluminum wheel casting simulation and process improvement. Recently, a counter-pressure wheel casting machine at Noranda Inc. Technology Center was successfully modified to cast magnesium wheels. To supply necessary process data such as the cycle time and the mold-cooling intensity, model simulations of the casting process were carried out using different magnesium alloys. The model results revealed that compared to aluminum alloy A356, the casting of magnesium wheel requires more process optimization. The lower volumetric heat content of magnesium alloys is more likely to cause an over-cooled mold and premature solidification in the wheel-rim region. Furthermore, the long solidification path from the wheel rim to the hub requires an optimized mold-temperature distribution to maintain a directional wheel solidification pattern. A carefully determined cycle time and programmed mold cooling process is therefore needed to obtain a stable casting operation and to produce sound magnesium wheels.

10:45 AM
Materials Comparison and Potential Applications of Magnesium in Automobiles: *Alan A. Luo*[1]; [1]General Motors Research and Development Center, Matls. & Process. Lab., 30500 Mound Rd., P.O. Box 9055, Mail Code 480-106-212, Warren, MI 48090-9055 USA

In this paper, the material properties, structural performance, mass saving potentials, design and manufacturing characteristics of magnesium are compared against various competing materials such as cast iron, steel sheet, aluminum alloys and polymers. The current and potential automotive applications of magnesium are reviewed, and the technical challenges for these applications are also discussed. Recent alloy development for powertrain applications and the creep resistance of several experimental magnesium alloys are critically reviewed. Generic R&D needs for expanding the use of magnesium in automobiles are given at the end of the paper.

Materials Processing in the Computer Age III: Process Optimization and Control

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

Tuesday AM — Room: Lincoln A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Vaughan R. Voller, University of Minnesota, Civil Engineering, Minneapolis, MN 55455 USA; Hani Henein, University of Alberta, Adv. Matls. and Process. Lab., Edmonton, Alberta T6G2G6 Canada

8:30 AM Opening Remarks

8:40 AM Keynote
Optimization of Manufacturing Processes: *Daniel Anthony Tortorelli*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mech. and Indust. Eng., Mech. Eng. Bldg., 1206 W. Green St., Urbana, IL 61801 USA

Structural optimization programs are often used to design products for minimum weight subject to constraints on stress, displacement, etc. These optimization problems are solved iteratively; and in each iteration 1) the appropriate finite element analyses are performed, 2) the cost and constraint functions are evaluated, 3) the sensitivities of these measures are computed, and 4) a nonlinear programming algorithm is used to perform a convergence check and, if necessary, update the design and initiate another iteration. These algorithms generally converge in a few iterations. However, to attain such desirable convergence characteristics, accurate sensitivity information must be available. The aforementioned optimization is for structures whose response is governed by the theory of linear elasticity. Unfortunately, manufacturing processes cannot be modeled with linear theories. To design such nonlinear systems we may use the same iterative optimization algorithm discussed above. However, the nonlinear analysis and subsequent sensitivity analysis are more complex. To facilitate the treatment of such design problems, the analysis and sensitivity analysis are derived for a general nonlinear system. It is shown that the computational demand that is required to perform the sensitivity analysis is only a small fraction of that which is required to perform the actual analysis. Three specific examples are provided to illustrate these methods. 1) The geometry of a polymer extrusion die is designed to minimize velocity and residence time variations over the outlet. A Hele-Shaw analysis is used to model the flow of the melt through the thin cavity. 2) An element-by-element discontinuous Galerkin method is used to simulate precipitate nucleation and growth during the quench process of an aluminum alloy extrusion. In the subsequent optimization, the process parameters are determined to control distortion and the size distribution of precipitates. 3) The die shape in an aluminum drawing process is designed to minimize distortion. The steady-analysis is modeled using a novel mixed method in which the displacement and internal field variables appear as the primary unknowns.

9:20 AM Keynote
Process Control: From PID to CIM: *J. Fraser Forbes*[1]; [1]University of Alberta, Chem. and Mat. Eng., 536 Chem. Mineral Bldg., Edmonton, Alberta T6G2G6 Canada

The meaning of the term "process control" has evolved significantly since its widespread use began in the manufacturing industries in the 1940's. Further, the scope of process control applications has drastically increased both in terms of breadth of industries using process automation technologies and the vertical integration of these technologies within a single industry. This evolution and integration of process control technologies within the manufacturing industries has proceeded to such an extent that process automation is an integral part of modern business practices. This paper will examine the current status of process control technology from simple process regulation through process performance optimization to business planning. Each stage of the operations automation hierarchy will be discussed both in terms of the current state-of-the-art and the directions in which these technologies are evolving. The key tools and tasks necessary for success at every level of the automation hierarchy will be introduced. Finally, the critical challenges that must be faced in accomplishing the goal of complete Computer Integrated Manufacturing (CIM) will be described. Finally, although this paper addresses issues of practical importance to any manufacturing company, the discussions will be framed in terms of the particular challenges facing and the opportunities available within the materials manufacturing industries.

10:00 AM Break

10:10 AM
Optimizing Heat Treatment Techniques for Large Castings and Forgings: *Koushik Ray*[1]; [1]Concurrent Technologies Corporation, Matls. Eng. Grp., 100 CTC Dr., Johnstown, PA 15904 USA

Military Specification testing requirements dictate that mechanical properties of large castings and forgings are to be examined at a particular depth (for e.g. at a depth of 3" for a 12" thick casting). The experimental method for determining the heat-treatment parameters to produce optimum mechanical properties, by uniquely heat-treating several identical blocks, is impractical for economic and material handling reasons. This paper describes a method in which Finite Element Analysis (FEA) is used to determine the heat treatment required to

obtain the optimum mechanical properties at a given depth. First, thermal response for a heat treatment at a given depth in the block is generated using FEA. This thermal response is then experimentally matched for a thin slice of the block material, so that it receives the same thermal response as if it were within the material, at that depth. By generating thermal response profiles for different heat treatments on the large block using FEA, and experimentally matching each of the thermal responses for different slices, the consequences of a large number of possible heat treatments can be studied by evaluating the mechanical properties of each of the slices. Experimental validation for a predicted heat treatment is also included.

10:30 AM

Optimization and Modeling of Thermomechanical Deformation and Microstructure Development during Metal Forming: Liming Zhang[1]; Ben Q. Li[1]; Reza A. Mirshams[2]; [1]Washington State University, School of Mech. Eng., Pullman, WA 99164 USA; [2]Southern University, Mech. Eng., Baton Rouge, LA 83808 USA

This paper presents a finite element-based numerical optimization approach to the control of thermomechanical deformation and microstructure formation during metals forming processes. A finite element model is developed based on the modification of our in-house code for the solution of non-Newtonian fluid flow and heat transfer to solve the rigid plastic deformation problems with penalty method applied to treat the pressure field. The finite element model is strongly coupled with a microstructure materials model that is capable of representing the microstructure development during metals forging processes. This coupled finite element model then forms as a core module that is used by a Newton based optimization methodology. With this approach, the thermomechanical field variables such as temperature distribution, strain rate and deformation and microstructural properties such as grain size distribution and recrystallization volume fraction can selectively controlled so as to achieve an optimized condition for metals forming. Numerical results will be presented to illustrate the application of this optimized approach to control the forging processes for isothermal and nonisothermal compression of carbon steel and TiAl alloys.

10:50 AM

Optimization of Continuous Slag/Matte Separation in Mitsubishi Process by Using Numerical Heat and Fluid Flow Analysis: Nozomu Hasegawa[1]; *Akira Kaneda*[1]; [1]Mitsubishi Materials Corporation, Cent. Rsch. Instit., 1-297 Kitabukuro-Cho, Omiya, Saitama 330-8508 Japan

In the Mitsubishi Continuous Copper Smelting and Converting Process, slag/matte mixture overflowed from a smelting furnace is separated continuously in an electric furnace (slag cleaning furnace, CL-furnace). In order to improve the separation of slag and matte and to minimize power consumption, the design and the operating conditions of the CL-furnace should be optimized. In the previous study, we developed a numerical model for simulating phenomena in the CL-furnace. The model consisted of 4 modules, electric field analysis, heat and fluid flow analysis, tracer simulation and the particle tracking method. In this study, the model was partially modified, k-ε model was adopted, and the effect of various conditions, such as furnace shape (including accretion) and scale, electrodes configuration, immerse depth of electrodes, thickness of slag layer and so on, was evaluated. And the optimum design and operating conditions are discussed.

11:10 AM

Development of a Model for Online Simulation and Control of a Continuous Steel Slab Caster: Richard A. Hardin[1]; Kai Liu[1]; *Christoph Beckermann*[1]; [1]University of Iowa, Dept. Mech. Eng., 2412 SC, Iowa City, IA 52242 USA

A computational model for online simulation and control of a continuous steel slab caster is described. First, a highly accurate, though slower than real-time, two-dimensional transient heat transfer model of the caster was developed. The model computes slab thermal and solidification conditions as a function of time-varying casting speed, secondary spray cooling water flow rates and temperature, slab thickness, steel chemistry, and pouring and ambient temperatures. This model was then streamlined into a real-time model that can be used for online simulation and control. Issues involved with the sacrifice of detail in the real-time model are addressed. This model was implemented on a Windows NT-based computer with an online data interface through the caster's Level 1 system. An overview of the computer-caster hardware interface and the multi-threaded graphical user interface is provided. Good agreement between measured and predicted temperatures for actual caster operating conditions is achieved by using a calibration procedure for the spray cooling correlations. Results of parametric studies are presented that provide insight into the effects of transient changes in casting variables on the slab thermal profile and the solidification endpoint. Finally, the development of a real-time, simulation-based algorithm for secondary spray cooling control is described. The use and effectiveness of dynamic spray cooling control for preventing surface temperature excursions from the desired profile is demonstrated.

11:30 AM Invited

An Interdisciplinary Approach Towards Optimal Continuous Casting of Steel: *Bozidar Sarler*[1]; Bogdan Filipic[2]; Miroslav Raudensky[3]; [1]University of Ljubljana, Fluid Mech. and Thermo., Askerceva 6, Ljubljana SI-1000 Slovenia; [2]Jozef Stefan Institute, Dept. Intelligent Sys., Jamova 39, Ljubljana SI-1000 Slovenia; [3]Technical University of Brno, Heat Trans. Lab., Technicka 2, Brno CZ-61669 Czech Republic

This paper describes main elements and practical use of the computational system for automated setting of optimized casting conditions in the continuous casting of steel. The system consists of the heat transfer model of the process, empirical metallurgical criteria, and optimization algorithm. The physical model of the strand temperature distribution relies on the classical mixture continuum formulation for the convective-diffusive heat transport. Fluid flow effects are considered through enhanced thermal conductivity of the liquid phase. The model is solved by the operator-splitting Crank-Nicolson finite volume enthalpy scheme. The solution procedure is coupled with the IDS solidification analysis package. Boundary conditions for the strand cooling rely on combination of the generic and plant specific mold, spray, and roll heat transfer coefficients by coping the effects of the casting speed, casting temperature, casting powder, mold oscillation mode, steel grade, water or air-mist spray type, running water, trapped water on the rolls, and radiation. The quality of the slab is related to seven empirical metallurgical criteria. The criteria include the maximum allowed length of liquid pool, recommended length of liquid pool, recommended surface cooling/reheating rate, surface temperature deviation at given axial location, and minimum surface temperature at unbending region. The criteria ensure the caster safety and limit the internal, longitudinal and transversal surface cracking possibility. The problem of finding optimum casting parameter values is stated as a minimization problem based on the analytical form of the cooling criteria evaluated from the steady-state temperature distribution of the strand. To solve the optimization problem, a genetic algorithm is employed. The algorithm explores process parameters settings heuristically by applying the principles of biological evolution. It starts with a random set of solutions and iteratively improves them through selection and variation. Solutions are evaluated by running the heat transfer model of the process and extracting the values of the empirical metallurgical criteria. The case studies for the ACRONI, Slovenia (stainless steel) and Nova Hut, Czech Republic (microalloyed steel) slab casters are presented.

Packaging & Soldering Technologies for Electronic Interconnects: Kinetics of Interfacial Reaction in Solder Joints

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

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

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Session Chairs: Martin Wieser, AlliedSignal, Plated and Discrete Products, Spokane, WA USA; K. N. Subramanian, Michigan State University, Dept. of Mats. Sci. and Mech., East Lansing, MI 48824-1226 USA

8:30 AM Invited

Modeling 3-D Intermetallic Compound Layer Growth between Sn-Pb Solder and Porous Substrate Coatings: *Polly Hopkins*[2]; Paul Thomas Vianco[1]; Kenneth Erickson[1]; [1]Sandia National Laboratories, Matls. Joining Dept., P.O. Box 5800, MS1411, Albuquerque, NM 87185-1411 USA; [2]Sandia National Laboratories, P.O. Box 5800, MS 0834, Albuquerque, NM 87185-0834 USA

Solder joints in hybrid microelectronic circuit (HMC) electronics are formed between the solder alloy and the noble metal thick film conductor that has been printed and fired onto the ceramic. The noble metal conductors is susceptible to solid-state reactions with Sn or other constituents of the solder, forming one or more intermetallic compounds (IMC). Unfortunately, because of the inherent porosity of thick film conductors, IMC growth in conductors cannot be well predicted by simply applying growth kinetics to a quasi-one-dimensional layer geometry. A previous paper summarized initial 2-D modeling results from coupled experimental and computational work. In the present paper, 3-D intermetallic growth in a porous substrate-solder system has been modeled using a 3-D pore structure determined experimentally by digitizing successive 2-D metallographic cross sections. The effects of the two-phase solder field in the 63Sn-37Pb/76Au-21Pt-3Pd solder-substrate system were addressed. Calculations were based on the reaction couple formed between 63Sn-37Pb solder and 76Au-21Pt-3Pd substrates. Physical constants in the model were evaluated from experimental data. Consumption of the thick film was predicted as a function of time and compared with data from independent experiments. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

8:50 AM Invited

Long-Term Aging Study on the Solid-State Reactions between 37Pb63Sn, 14Bi43Pb43Sn, 58Bi42Sn Solders and Ni Substrate: B. L. Shiau[1]; C. Chen[1]; C. E. Ho[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Chem. Eng., Chungli, Taiwan

The nickel layer with a thin Au coating is a very common surface finish in electronic components. During reflow, the Au coating reacts with the solder very rapidly and then disappears into the solder, exposing the Ni layer below. The Ni layer will then react with the solder to form intermetallic compound at the interface. In this study, the solid-state reactions between the 37Pb63Sn, 14Bi43Pb43Sn, 58Bi42Sn solders and the Ni substrate were investigated at several temperatures. Aging time as long as five months was used. It was found that in all cases the reaction product was Ni_3Sn_4 with the layered structure. The growth kinetics for the reactions with the three different solders will be presented. Compared to similar reactions with the Cu substrate, the reactions with the Ni substrate are about an order of magnitude slower. The microstructural evolutions in these three systems will be compared and discussed. The activation energies will also be compared.

9:10 AM Invited

Interfacial Phenomena of the Sn-3.5wt%Ag Solder Alloy on the Cu and the Ni Substrates: *Hyuck Mo Lee*[1]; *Won Kyoung Choi*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. and Eng., Kusung-Dong Yusung-Gu 373-1, Taejon 305-701 Korea

The interfacial phenomena of the Sn-3.5wt%Ag solder alloy on the Cu and the Ni substrates were investigated during soldering and aging. The soldering was performed at 250°C from 30s up to 10hrs followed by aging at 130°C for 100, 400 and 800hrs. The initial formation of IMC after soldering has been found to exert an effect on the growth and morphology of IMC during aging. The morphology of IMC formed in the soldering reaction was closely related with the change of the enthalpy (ΔH_m) which was generated when the IMC formed in the liquid phase of solder.

9:30 AM

Kinetics of Interfacial Microstructure in Diffusion Couples between Solder and Cu/Ni/Pd Metallization: *G. Ghosh*[1]; [1]Northwestern University, Dept. of Mat. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

The intermetallic formation at the solder/substrate interface, both during processing and in service, play a key role in determining the mechanical property and reliability of solder joints. Even though Pb-Sn eutectic solder is widely used in electronic industry, due to various reasons there is a growing interest in the use of Pb-free solders. Furthermore, electronic packaging paradigm requires solders having a hierarchy of melting points. As the solder joint size decreases and the service temperature of the advanced products increases, the issue of interfacial reaction kinetics becomes very critical. Metallization schemes using Ni and Pd are becoming increasingly popular in electronic packaging. We will present a comparative study of interfacial reaction kinetics in diffusion couples between Pb-Sn, Ag-Sn, Bi-Sn eutectic solders and Cu/Ni/Pd metallization scheme. The evolution of microstructures due to both liquid- and solid-state reactions will be presented. The products of interfacial reaction are characterized by SEM, TEM, and AEM. The interfacial microstructures will be discussed in terms of diffusion paths using the calculated isothermal sections of the relevant ternary system.

9:50 AM

Solder-Metallization Interdiffusion in Microelectronic Interconnects: *Eric J. Cotts*[1]; A. Zribi[1]; R. R. Chromik[1]; R. Presthus[1]; J. Clum[1]; K. Teed[1]; L. Zavalij[1]; J. Tova[1]; [1]Binghamton University, Physics Dept., P.O. Box 6016, Vestal Parkway East, Binghamton, NY 13902-6016 USA

The reliability of solder joints is in large part determined by the nature of the intermetallic compounds which form at the solder/metal interface, their stoichiometry and geometry. While dissolution rates of eutectic Pb-Sn solder on Cu substrates are fairly well known, the solderability, and integrity, of different solder/substrate combinations has become an issue. For instance, the increased use of Pd lead finishes, or the use of electroless Ni metallizations, or the increased interest in Pb free solders, creates the need for an increase in the scope of characterization of solder/metal systems. We report on our study of the rates of intermetallic compound formation in solder-metallization systems, and the mechanical integrity of specific solder-substrate combinations such as (Pd; [Ni-P]; Pd/[Ni-P], Au/[Ni-P], Au/[Ni-P]/Cu) substrates in combination with Sn or [Sn-Pb]. We characterize the reaction mechanisms and constants for new phases (such as intermetallics) during short and long term aging, with a view to understanding failure mechanisms. We carried out tests of reactivity (dissolution rate) between various substrate materials and solder alloys using differential scanning calorimetry (DSC). Reaction products were characterized by x-ray diffraction analysis, optical and electron metallography, and cross-sectional transmission electron microscopy. We focused on the reactions which produce alloys shown in mechanical testing to compromise the integrity of the solder joints. We quantify reactivities

and joint integrities as a function of initial substrate metallurgy and microstructure.

10:10 AM Break

10:25 AM Invited

Copper Substrate Dissolution in Eutectic Sn-Ag Solder and Its Effect on Microstructure: S. Chada[2]; *R. A. Fournelle*[1]; W. Laub[3]; D. Shangguan[4]; [1]Marquette University, Matls. Sci. and Eng. Pgm., P.O. Box 1881, Milwaukee, WI 53201 USA; [2]Srini Chada, Motorola, Plantation, FL 33322 USA; [3]Solectron GmbH, Solectronstr. 2, Herrenberg 71083 Germany; [4]Ford Motor Company, Visteon Auto. Sys., 17000 Rotunda Dr., Dearborn, MI 48121 USA

The dissolution of Cu into molten Sn-3.5Ag solder and its effect on microstructure were studied by light microscopy, scanning microscopy and x-ray microanalysis. X-ray microanalysis of the average Cu content of samples soldered under various conditions showed that the amount of Cu dissolved during soldering increased with increasing soldering temperature and time and that the rate of dissolution could be described by a Nernst-Brunner equation. Microstructurally it was found that the volume fractions of primary Sn dencrites and eta phase dendrites increase with increasing soldering temperature and time. The microstructural changes can be explained using Sn-Ag-Cu phase equilibria data. A numerical method was developed for calculating the amount of Cu dissolved under non-isothermal conditions, which describes dissolution reasonably well.

10:45 AM

The Growth of Cu-Sn Inter-Metallics in Soldered Cu and the Influence of Ag Content: *Ker-Chang Hsieh*[1]; Hen-So Chang[1]; [1]National Sun Yat-sen University, Instit. of Matls. Sci. and Eng., Kaohsiung, Taiwan

Pure copper soldered with Pb-Sn alloys and Pb-Sn-Ag alloys stored at 150°C for long period of time. The growth of Cu_3Sn and Cu_6Sn_5 inter-metallic phases are examined under SEM and EPMA after holding at 4, 9, 16 and 25 days respectively. The solder alloys include six Pb-Sn binary alloys and nine Pb-Sn-Ag ternary alloys. The inter-metallic growth rate constant are expressed as function of alloy compositions, D1/2 = f(Pb wt.%). The influence of Ag are examined a similar way in comparison the inter-metallic growth rate. Ag_3Sn inter-metallic phase formed and growth rate decreased. The contents of this research include, 1. sample preparation method; 2. solder/Cu reaction layer microstructure; 3. growth rate evaluation.

11:05 AM

A Kinetic Study of the IMC Growth at the Interface of Ni-Containing UBM's and Eutectic Pb/Sn Solder: *Peng Su*[1]; Tia M. Korhonen[1]; Matt A. Korhonen[1]; Che-Yu Li[1]; [1]Cornell University, Dept. of Matls. Sci. and Eng., 356 Bard Hall, Ithaca, NY 14853 USA

Ni-containing underbump metallizations (UBM's) have been proved to be able to effectively reduce the growth rate of intermetallic compounds (IMC) growth rate at the solder/UBM interface. This effect correspondly reduces the consumption of the wetting metals in the UBM during the reflow. The introduction of Ni into the conventional Cu/Sn system, however, brings extra complexity to the kinetics of the original intermetallic formation reaction. In this research, the UBM/solder reaction was studied with alloy foils of various Ni/Cu ratio and molten solder bath. EDX results of the element distribution at the interface revealed that different Ni/Cu ratios demonstrated different diffusion behaviors, which in turn affected the morphology of the intermetallic as well as the growth rate. A kinetic model of the consumption of the Cu/Ni during the soldering reaction is proposed, and the theoretical calculation is compared with the experimental data.

11:25 AM Invited

Interfacial Reactions in In-Sn/Ni Couples and Phase Equilibria of In-Sn-Ni System: *Sinn-Wen Chen*[1]; Ching-Yu Huang[1]; [1]National Tsing-Hua University, Dept. Chem. Eng., Hsin-Chu 30043 Taiwan

In-Sn alloys are promising lead-free solders. Their reactions with Ni substrate and the phase equilibria of the In-Sn-Ni ternary systems are investigated in this study. In-Sn-Ni alloys of various compositions are prepared and annealed at 160° and 240°C. The phases formed in the samples are analyzed by using metallography and powder XRD. The stable binary compounds are β-In_3Sn, η, Ni_3In, Ni_2In, Ni_3In_9, NiIn, Ni_2In_3, $Ni_{28}In_{72}$, Ni_3Sn, Ni_3Sn_2 and Ni_3Sn_4. In-49.1 wt.%Sn/Ni, In-60 wt.%Sn/Ni, and In-80 wt.%Sn/Ni couples reacted at 160° and 240°C are examined. Although there are many intermetallic compounds in the Sn-In-Ni systems and the phase equilibria relationships are complicated, there is only one compound formed in all the (Sn-In) alloys/Ni couples reacted at both 160° and 240°C up to 144 hours.

11:45 AM

Interfacial Reactions in Zn/Ni and Bi/Ni Couples: *Chih-Ming Chen*[1]; Sinn-Wen Chen[1]; [1]National Tsing-Hua University, Dept. Chem. Eng., Hsin-Chu 30043 Taiwan

β_1-NiZn, η-$Ni_{15}Zn_{21}$ and δ-$NiZn_8$ phases, formed at the interfaces of Zn/Ni couples reacted at both 150° and 200°C. The thickness of β_1 and η phases increased with longer reaction time, while that of the δ phase reached a maximum of about 6 μm. Only one intermetallic, $NiBi_3$ phase, was formed in the Bi/Ni couples reacted at 150° and 200°C. The $NiBi_3$ phase grew with time as well. The NiBi phase which is also a stable binary Ni-Bi intermetallic at both 150° and 200°C showed no sign of existence in the Bi/Ni couples reacted up to 144 hours. In addition to these reaction experiments mentioned above, an electric current of 300 A/cm² density was passed through the reaction couples to investigate the electromigration effect upon interfacial reactions of the two systems. The results are identical to those without the passage of electric current, which indicate that electromigration effects of 300 A/cm² electric density upon interfacial reactions are insignificant in the Zn/Ni and Bi/Ni systems at 150° and 200°C.

Pressure Technology Applications in the Hydrometallurgy of Copper, Nickel, Cobalt and Precious Metals: Pressure Technology Applications in the Hydrometallurgy of Nickel

Sponsored by: Extraction & Processing Division, Copper, Nickel, Cobalt Committee

Program Organizers: James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

Tuesday AM — Room: Lincoln C
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Nic Hazen, Hazen Research Inc., Golden, CO 80403 USA; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

8:30 AM

Pressure Leaching of Nickel Sulphide Concentrates: *Roman M. Berezowsky*[1]; [1]Dynatec Corporation, Metallu. Tech. Div., 8301-113 St., Fort Saskatchewan, Alberta T8L4K7 Canada

Potential advantages usually cited for the direct pressure leaching of nickel sulphide concentrates, over the traditional pyrometallurgically based processes, include the ability to treat lower grade and/or higher impurity (arsenic, magnesium) feeds, higher cobalt recoveries, and production of elemental sulphur, rather than sulphur dioxide or acid. To date, however, the commercial application of direct hydrometallurgical treatment has been limited to two applications of Sherritt's ammoniacal oxidative pressure leach based process, and to two rather specific applications based on sulphuric acid oxidative pressure leaching. The past decade has witnessed a strong resurgence in interest in the direct pressure leaching of nickel concentrates spurred, in part, by advances and the wider acceptance of solvent extraction for metals separation and recovery, and, the use of ultrafine milling and/or the use of chloride in an acid sulphate based system to promote the leach kinetic of the metals and the yields of elemental sulphur. This paper

provides, a discussion on leach chemistry, options on metals recovery from solution, and a review of the existing commercial operations, as well as some of the most promising recently promoted processes.

9:00 AM

Nickel Pressure Leach Process at Outokumpu Harjavalta Metals Oy: *Esa Lindell*[1]; Kari Knuutila[1]; Stig-Erik Hultholm[2]; [1]Outokumpu Harjavalta Metals Oy, Harjavalta Finland; [2]Outokumpu Research Oy, Pori, Finland

This paper deals with the pressure leach of the nickel mattes at Outokumpu Harjavalta Metals Oy. The overall nickel process is also shortly discussed. The nickel plant was modernized and expanded in 1995 which led to a production increase from 17 000 t/a to 40 000 t/a in 1999. Before the expansion only cathode nickel was produced. The additional capacity is nickel powder and briquettes produced in the new hydrogen reduction plant. Ammonium-sulphate is obtained as a by-product. There is also an optional capacity to produce cobalt powder via hydrogen reduction. In the nickel smelter where the Outokumpu DON process is utilized two separate granulated products are produced: flash smelting furnace (FSF) matte and electric furnace (EF) matte. Consequently the nickel refinery has two separate leach circuits one for each type of matte. The FSF matte leach circuit consists of two counter current atmospheric leach stages and two pressure leach stages both in counter current arrangement. The EF matte leach circuit consists of an atmospheric and a pressure leaching stage. All reaction vessels and autoclaves are designed by Outokumpu. Copper in the mattes is separated as a sulphide precipitate rich PGMs which is directed to the copper smelter whereas iron is removed as a hematite residue from the EF pressure leach stage. From the first atmospheric leach stage of the FSF circuit neutral, iron and copper free nickel sulphate solution is obtained. The nickel sulphate solution is further purified in a solvent extraction process where cobalt and other minor impurities are removed. The purified nickel stream is then fed to the electrowinning and to the hydrogen reduction plant. The pressure leach process has been in stable operation since the start-up. Furthermore, the nickel process has reached its design production capacity according to the schedule.

9:30 AM

The Viscous Behavior of Aqueous Slurries of Goethite and Limonitic Laterite: Characterization for Design and Analysis of Tubular Transport and Heating Systems: *Brian C. Blakey*[1]; D. F. James[2]; M. Kawaji[3]; E. Krause[1]; [1]Inco-ITSL, 2060 Flavelle Blvd., Mississauga, Ontario L5K1Z9 Canada; [2]University of Toronto, Dept. of Mech. and Indust. Eng., Toronto Canada; [3]University of Toronto, Dept. of Chem. Eng. and Appl. Chem., Toronto, Canada

Unlike most hydrometallurgical slurries, those containing significant concentrations of goethite are typically highly viscous and shear-thinning, even at solid volume fractions below 5%. The viscosity may also depend upon time. These characteristics must be taken into consideration in slurry transport and heating. The technical literature is replete with apparent contradictions regarding the viscous behavior of these slurries, which may be due, in part, to variations in viscosity-measuring techniques. We present techniques which yield consistent results suitable for use in process design. A striking degree of time-dependent behavior is demonstrated for a laterite slurry. Reliable methods to design and analyze tubular transport and heating systems for time-independent slurries are summarized. Rules of thumb are then offered to account for the effects of time-dependency.

10:00 AM Break

10:15 AM

Application of Hpal at the Syerston Laterite Project: *Geoff Motteram*[1]; [1]Black Range Minerals NL, Syerston Nickel Cobalt Project, Level 10, 190 St. Georges Tce, Perth, Western Australia 6000

The Syerston Nickel Cobalt Project in Central New South Wales is unique amongst the laterite projects being developed in Australia and internationally. The unique nature ensues from the laterite enrichment occurring over an Alaskan style ultramafic intrusion with dunite core. Characteristics that result include a goethite host mineralisation with low magnesium content and rapid extraction rates in High Pressure Acid Leach, along with low clay content and minimal viscosity problems. The base dunite mineralisation exhibits high cobalt to nickel ratios with associated high manganese content. It also results in significant metallic platinum content with associated chrome, plus relatively high scandium content within the goethite ore. The desire to maximise cobalt recovery in the presence of high manganese and chrome influence selection the metal recovery circuit. Whereas the potential to recover refractory platinum from the leach residues and scandium from circuit water introduces further circuit considerations.

10:45 AM

Nickel and Cobalt Recovery from Madagascar Laterite: *Eddie Chou*[1]; Phil Rooke[1]; Alan Williams[1]; Jerry Hanks[2]; G. Houlachi[3]; [1]Hazen Research Inc., 4601 Indiana St., Golden, CO 80403 USA; [2]Phelps Dodge Exploration; [3]Noranda Technology Center, Pointe Claire, Canada

Phelps Dodge, Inc. (PD) is currently developing a nickel/cobalt deposit in Madagascar. Hazen Research, Inc. provides technical assistance to PD to investigate recovery processes fro nickel and cobalt. Various process options were evaluated in laboratory experiments and pilot plant operations. The process selected for the feasibility study consists of feed preparation, high-pressure acid leach, countercurrent decantation wash, sulfide precipitation, sulfide dissolution, solvent extraction, ion exchange, and electrowinning. Among these unit processes, high-pressure acid leach, sulfide precipitation, and sulfide dissolution are applications of pressure technologies. Process development work and design criteria for commercial operations of these unit processes are presented in this paper. Concerns related to scaling in the high-pressure acid leach and the sulfide precipitation processes are discussed. Corrosion rates of various metal coupons under these environments were analyzed.

11:15 AM

Bulong Nickel Operations: A Review of the Commissioning and Early Operations Phases in the High Pressure Acid Leach Plant: *G. L. Frampton*[1]; G. J. McCunn[1]; [1]Bulong Nickel Operation, Tech. Svcs., P.O. Box 10391, Kalgoorlie, WA 6430

The Bulong Nickel Operation is a mining and metallurgical complex located approximately 30km east of Kalgoorlie in Western Australia. The process uses high-pressure acid leaching, solvent extraction and electrowinning to produce nickel and cobalt cathode. The projected ore-processing rate is up to 600,000 tonnes per year, with target production levels of approximately 9000 tonnes per year of nickel metal and 640 tonnes per year of cobalt metal in Phase 1. As expected from a complex, integrated facility consisting of mining, leaching and refining operations, the commissioning and early operating period during the first ten months of 1999 has provided numerous challenges to the process and engineering disciplines. The majority of these challenges have either been successfully addressed already, or are close to being resolved. This paper has been written with the intention of reviewing the performance of the High Pressure Acid Leach Plant during this period. Major issues influencing plant throughput rates, plant availabilities and plant recoveries have been highlighted and discussed. An attempt has been made to discuss these factors using a multidisciplinary approach that examines a number of significant chemistry, process and engineering aspects.

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

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

Tuesday AM — Room: Knoxville B
March 14, 2000 — Location: Opryland Convention Center

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

8:30 AM

Modeling of Open Die Forging for Ti-6Al-4V: *D. J. Li*[1]; K. O. Yu[1]; P. A. Russo[1]; J. M. Hjelm[1]; G. W. Kuhlman[2]; [1]RMI Titanium Company, 1000 Warren Ave., Niles, OH 44446 USA; [2]Consultant

Forging is used to refine coarse cast structure, and to develop fine equiaxed alpha for Ti-6Al-4V. It is more difficult to break alpha platelets in the center of large section forgings compared to the edge. This was hypothesized to be the result of lower strain at the center. However, 2.5D deformation modeling clearly shows the center already has the highest effective strain. Based on this fact, it is speculated that the above-mentioned problem is a result of the thermal history at the center rather than strain history. To solve this problem, one approach is to impart more strain into center. A few effective ways have been demonstrated by the deformation modeling. Another approach is to lower the critical strain required for recrystallization of alpha platelets by reducing alpha platelet thickness during beta quenching. The effect of billet section size as well as forging temperature on alpha platelet thickness after water quenching has been studied by thermal modeling. This information has been used to predict production processes that will yield fine grain alpha through the section thickness.

8:55 AM

Application of Finite Element Analysis to the Primary Conversion of Titanium Alloys: *Vasisht Venkatesh*[1]; S. P. Fox[1]; [1]TIMET Technical Laboratories, 8000 W. Lake Mead Dr., Henderson, NV 89015 USA

In order to develop accurate finite element analysis (FEA) techniques to model the primary processing of titanium alloys such as TIMETALâ-17 (Ti-5Al-2Sn-2Zr-4Cr-4Mo), a forging alloy for gas turbine engines, appropriate experiments to generate material properties and validation trials need to be conducted. Utilization of material flow behavior corresponding to specific microstructures that are relevant to the conversion process being modeled is discussed. Appropriate small scale tests for the purposes of FEA model validation and mechanical property database generation are also discussed. Forge validation results of TIMETALâ-17 (Ti-17) using DEFORM, a finite-element analysis software code, showed good agreement with the experimentally observed behavior.

9:20 AM

Redesign of Furnace Heating Practice for 34" Diameter Ti-6-4 Ingots: *Ramesh S. Minisandram*[1]; Robert Mark Davis[1]; Robin M. Forbes Jones[1]; Robert J. McHugh[1]; John W. Pridgeon[1]; [1]Allvac, Allegheny Teledyne Co., 2020 Ashcraft Ave., P.O. Box 5030, Monroe, NC 28110 USA

In response to a need for increased furnace capacity and reduced process cycle times, a new heating practice for 34" diameter Ti-6-4 ingots is now being implemented. The new practice was designed based on a wide array of numerical simulations of the ingot heating practice coupled with production-scale experimental trials using nominal 17,000 lb. ingots instrumented with sub-surface thermocouples. The new practice is expected to reduce furnace heating time for ingot product by up to 20%. The practical hurdles that were overcome in the design process will be discussed along with the experimental details.

9:45 AM Break

10:00 AM

Steady State Model for the Radial Forging Process: *Ramesh S. Minisandram*[1]; Robin M. Forbes Jones[1]; Erik G. Thompson[2]; [1]Allvac, Rsch. & Dev., 2020 Ashcraft Ave., P.O. Box 5030, Monroe, NC 28110 USA; [2]Colorado State University, Civil Eng., Fort Collins, CO 80523 USA

The radial forging process is a complex process that involves a number of repetitive steps. Radial forging machines, depending upon the type, can operate at a few hundred strokes per minute. The large number of strokes involved make it prohibitive to simulate the process as a transient (stroke-by-stroke) analysis. Allvac's radial forging model makes use of a unique steady state approach that allows for rapid turnaround of simulation results, making it a handy tool that is used on a routine basis in addressing production issues. Details of the model are presented along with a few examples that illustrate model verification and its application in addressing manufacturing problems.

10:25 AM

Modeling Texture Evolution in Titanium Rolling: *Paul Richard Dawson*[1]; Nathan Barton[1]; [1]Cornell University, 196 Rhodes Hall, Ithaca, NY 14853 USA

Mechanical properties of titanium and its alloys are strongly influenced by the crystallographic texture imparted by processing. Anisotropy is readily apparent in the strength of rolled products as well in the formability. This presentation will focus on the simulation of texture evolution during rolling, both for commercial purity titanium and for a two-phase alloy (Ti-6Al-4V). The modeling of plastic flow of titanium is made difficult by the strong yield anisotropy of the hexagonal close packed phase, which arises from the relative difficulty in activating slip modes associated with c-axis extension or compression. We will compare results obtained using several modeling assumptions (upper bound, lower bound, and constrained hybrid) within a finite element formulation for flat rolling which accounts for through-thickness variations in the straining and for texture evolution. We will discuss additional modeling results performed on aggregates of grains in which every grain is individually resolved with finite elements and subjected to an idealized rolling history (plane strain compression). These results quantify the degree to which the strong single crystal anisotropy influences the heterogeneity of deformation over the volume of the aggregate, and provide a means to evaluate simpler assumptions that link the micro and macro scales (again, upper bound, lower bound and constrained hybrid models). We will present results showing the influence of texture on the formability as determined by r-values, and conclude with presentation of detailed analyses of the strain partitioning between the hexagonal close packed and body centered cubic phases.

10:50 AM

Processing of Titanium Cylinders by Roll Forming Techniques: *David U. Furrer*[1]; Robert Niemi[1]; [1]Ladish Company Inc., P.O. Box 8902, Cudahy, WI 53110-8902 USA

Titanium is an important material utilized in numerous aerospace and non-aerospace applications. Components produced from titanium are often in cylindrical geometries for casings, tubes and other uses. Seamless processing of cylindrical structures have shown many advantages, and as such, methods to produce these types of components have been developed and optimized for titanium material. Roll forming is a unique, incremental extrusion process by which net or near-net components can be produced. Often conducted at room temperature, the dimensional tolerance capability can approach machining processes. Commercially pure titanium can be readily processed by roll forming techniques, while alloyed titanium materials require special processing consideration. This process is capable of producing a wide range of cylinder diameters, heights and wall thicknesses. A combina-

tion of manufacturing process design and material application can result in high performance and economical titanium components.

Surface Engineering in Materials Science I: Coating/Films Characterization (C)-I

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

Tuesday AM — Room: Canal B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Sudipta Seal, University of Central Florida, AMPAC & MMAE, Orlando, FL 32816 USA; Yip Wah Chung, Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL 60208 USA

8:30 AM
Microstructure of TiO2-x Thin Films Reactively Sputtered by Dc Magnetron: *K. Zakrzewska*[1]; *A. Brudnik*[1]; A. Kusior[1]; M. Radecka[1]; A. Kowal[2]; [1]University of Mining and Metallurgy, Al. Mickiewicza 30, Kraków 30-059 Poland; [2]Polish Academy of Sciences, Instit. of Catalysis and Surf. Chem., Ul. Niezapominajek 1, Kraków 30-239 Poland

GID (Grazing Incidence Diffraction) and AFM (Atomic Force Microscopy) were used to study the progressive structure evolution in thin films of TiO2-x. Thin films were deposited by the reactive magnetron sputtering with the controlled intensity of Ti emission line (l=500 nm). Variation in this parameter reflected a change in the oxidation state of the sputtered metallic target and as a consequence affected the growth of polymorphic forms of titanium dioxide: anatase and rutile. The anatase was found to dominate the structure of films obtained from the oxidized target. The ratio of rutile/anatase phases was found to increase with the increasing intensity of Ti emission line, i.e. decreasing target coverage by oxides. AFM images gave evidence of two types of grains: those of smaller size constituting the base of the sample and large ones formed at the film surface. Good correlation was established between the relative distribution of these species and the structure evolution observed in X-ray diffraction experiments performed at different angles of grazing incidence.

8:50 AM
Determination of the Structure and Chemistry of the Thermally Grown Oxide Scale in Thermal Barrier Coatings Containing a Platinum-Nickel-Aluminide Bondcoat: *Michael R. Brickey*[1]; J. L. Lee[1]; [1]Purdue University, Sch. of Matls. Eng., 1289 MSEE Bldg., Rm. 150, West Lafayette, IN 47907-1289 USA

A chief influence governing the reliability of modern gas turbine engine thermal barrier coatings (TBC's) is the adhesion of a thermally grown oxide (TGO) scale to both the bondcoat and the insulative ceramic topcoat layers. Factors affecting scale adhesion include TGO and bondcoat chemistry in addition to growth and thermal stresses developed during engine service. Several advanced TBCs incorporate an electron beam-physical vapor deposited (EB-PVD) yttria-partially-stabilized zirconia (YPSZ) ceramic topcoat and a platinum-nickel-aluminide (Pt-Ni-Al) bondcoat on nickel based superalloy engine components. It is known that Pt-Ni-Al bondcoats display excellent TGO scale adhesion relative to traditional bondcoat chemistries, but limited microstructural and chemical data exist in the literature for scales formed by this TBC system. Since the TGO is typically 1 to 2 microns thick, analysis of the scale is well suited to transmission electron microscopy (TEM). Conventional TEM, scanning TEM, windowless energy dispersive X-ray spectroscopy, and electron energy loss spectroscopy were among the techniques used to reveal the microstructural and chemical characteristics of the scale. It was found that the scale consists of hexagonal aluminum oxide (-Al2O3) in both as-deposited and in thermally cycled specimens; no metastable Al2O3 allotropes were observed. In the as-deposited specimen, the scale's grains were predominantly columnar. Zr-rich particles were observed to occur both intra- and intergranularly throughout the scale thickness, and their concentration increased upon approaching the YPSZ. Thermally cycled specimens had a morphologically banded scale structure: one band of grains adjacent to the YPSZ had an equiaxed morphology and contained Zr-rich particles; a second band of grains adjacent to the bondcoat had a columnar morphology with axes oriented normal to the TGO/bondcoat interface. It is believed that this is the first experimental observation of a morphologically banded scale containing Zr-rich dispersoids in a TBC system having a Pt-Ni-Al bondcoat. For specimens undergoing up to 10 thermal cycles, both the equiaxed band and the columnar band increased in thickness with increased thermal exposure. However, the proportion of the scale comprised by the equiaxed band tended to decrease with greater cycling. The columnar grains of the as-coated and thermally cycled specimens differed in that the latter contained no Zr-rich particles and were comparatively coarse in size. Porosity and cracks were associated with the irregular interface between the bands, but no porosity was observed at the YPSZ/TGO or TGO/bondcoat interfaces. Since these defects can act as a weak link promoting YPSZ spallation, it is expected that elimination of the banded structure is key to improving TBC reliability. It is proposed that formation of the equiaxed grains and the banded morphology is associated with the Zr-rich dispersoids.

9:10 AM Invited
Synthesis and Characterization of Ultrathin Overcoats: *Yip-Wah Chung*[1]; [1]Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL 60208 USA

The magnetic spacing budget at 100 Gb/in^2 storage density provides only 2nm for the protective overcoat. Such an ultrathin overcoat must have the necessary wear resistance, be smooth enough to allow low flying heights and be sufficiently defect-free to provide corrosion protection for the underlying magnetic media layer. One also requires the overcoat to be compatible with the lubricant and materials in contact. It is desirable that the technique to synthesize such a coating be compatible with existing manufacturing practice. In this paper, we present preliminary results on the synthesis and characterization of three promising candidate coatings: (i) amorphous CNx; (ii) crystalline TiB2 and (iii) hydrogen-free amorphous carbon. Advantages and disadvantages of each coating material and the associated synthesis technique will be discussed.

9:35 AM
Characterization of Oxide-Metal and Grain Boundary Interfaces in Aluminide Bond Coats: *Stacie LeSure*[1]; J. Allen Haynes[1]; Ian G. Wright[1]; Bruce A. Pint[1]; [1]Oak Ridge National Laboratory, Met. & Cer. Div./Cer. Surf. Sys. Grp., P.O. Box 2008, Oak Ridge, TN 37831-6063 USA

In an effort to increase gas turbine efficiency, thermal barrier coating systems (TBC's) are used to protect superalloy components in gas turbine engines. Most TBC systems consist of a superalloy substrate, an oxidation-resistant bond coat and a ceramic top coat. This study will focus on the formation and adhesion of a protective alumina scale at the bond coat- top coat interface. Adhesion of the scale, which is the weak link in TBC durability, is influenced by a number of factors, including bond coat platinum content. Thus, the purpose of this investigation is to examine the role of platinum in improving scale adherence. Analytical Transmission Electron Microscopy (ATEM) will be used to examine the alumina/bond coat interface in bond coats with and without platinum additions. Grain boundary segregation in the

bond coat will also be investigated by ATEM. Field emission gun scanning electron microscopy (FEG-SEM) will be used to characterize the microstructures of the bond coats and scales. Additionally, segregation phenomena occurring at the bond coat surface will be studied using hot-stage Auger Electron Spectroscopy (AES).

9:55 AM

Microstructures of TiSi2 Coatings Obtained by Plasma Spray in Both Air and Low Pressure Conditions: *G. Li*[1]; S. Uematsu[1]; [1]Ship Research Institute, Matls. and Process. Div., Shinkawa 6-38-1, Mitaka, Tokyo 181-0004 Japan

Effect of plasma spray processing parameter on the microstructure of coatings was reported in this article. TiSi2 particles (<44Êm) were sprayed, in both air and low pressure conditions, respectively. All experiments were carried out with a conventional DC arc plasma spray device. The phases present in coatings were analyzed by X-ray diffraction (XRD). Microstructures were observed by scanning electron microscopy (SEM). Chemical compositions were examined by electron probe microanalysis (EPMA). The XRD patterns showed that, sprayed in air condition, some starting TiSi2 particles were decomposed into Ti5Si3, which was predominant in coating. In addition, oxide products of TiSi2 were also found. In contrast, TiSi2 phase was a major constitute in coating obtained in low pressure chamber. SEM observation revealed that the features of coatings were characterized by splashing in both cases. It implied that most of the TiSi2 particles were very well melted. The phenomena were discussed. This investigation provided a step further toward the understanding and optimizing of the TiSi2 particles spraying process.

10:15 AM Break

10:30 AM

Grain Structure Development in Plasma Sprayed Coatings: Experiment and Modeling: *Guo-Xiang Wang*[1]; Sanjay Sampath[2]; [1]The University of Akron, Dept. of Mech. Eng., Auburn Sci. & Eng. Ctr. 106B, Akron, OH 44325-3903 USA; [2]State University of New York, Dept. of Matls. Sci. & Eng., Ctr. for the Thermal Spray Rsch., Stony Brook, NY 11794-2275 USA

Plasma-sprayed coatings are made of small splats formed when molten particles impact at a high speed on a substrate. The quality and the properties of a coating therefore strongly depend on the phases and microstructures of each splat. A variety of grain structures have been found in plasma-sprayed coatings, depending on the coating material, substrate material, and process condition. This paper presents some recent experimental observations of grain structures in single splats plasma-sprayed on different substrates. The coating materials used include molybdenum, alumina, and partially yttria-stabilized zirconia, and the substrate materials are steel and glass. It is found that, in most of cases, the ceramic coatings show a columnar grain structure with very high grain density, although occasionally some equiaxed grains were observed. Fine columnar grains were formed in the molybdenum splats sprayed on a steel substrate, but grains with significant large sizes were seen in the Mo splats sprayed on a smooth glass surface. A heat transfer model is developed to analyze the thermal characteristics of splat solidification. The classical nucleation theory is incorporated in the heat transfer model to predict the formation of crystalline nuclei and the melt undercooling at nucleation. Detailed heat transfer analyses are performed using the model to study the effect of coating material, substrate material, and the substrate surface condition on the formation of columnar or equiaxed grains. Solidification mechanisms in a single splat under different conditions are also investigated.

10:50 AM

Relation between Deposition and Recrystallization Textures of Copper and Chromium Electrodeposits: *Dong Nyung Lee*[1]; Joon Hwan Choi[1]; Soo Young Kang[1]; [1]Seoul National University, Matls. Sci. and Eng., Shinrim-dong Kwanak-gu, Seoul 151-742 Korea

The texture of electrodeposits varies with electrolysis conditions and may change after recrystallization. The <100>, <111>, and <110> textures of copper electrodeposits obtained from copper sulfate baths changed to the <100>, <100>, and <SQR(3)10> textures, respectively, after recrystallization. The textures of chromium electrodeposits obtained from the standard Sargent bath remained unchanged on recrystallization. The results are in agreement with the prediction of the strain energy release maximization model. In the model the recrystallized grains orient themselves so that their minimum elastic modulus direction can be parallel to the absolute maximum internal stress direction due to dislocations in the non-recrystallized grains.

11:10 AM

Effects of Pre-Treatments and Interlayers on the Nucleation and Growth of Diamond Coatings on Titanium Substrate: *Yongqing Fu*[1]; Nee Lam Loh[1]; Bibo Yan[1]; Chang Q. Sun[2]; Peter Hing[2]; [1]Nanyang Technological University, Matls. Lab., Schl. of Mech. & Prod. Eng., 639798 Singapore; [2]Nanyang Technological University, Schl. of Appl. Sci., 639798 Singapore

During diamond deposition on titanium substrate, there exist two processes: (1) Diffusion of hydrogen into titanium substrate and the formation of hydride thus degrading the mechanical properties of substrate. (2) Competition among the rapid diffusion of carbon atoms into substrate, the formation of carbide and the nucleation of diamond crystals (thus decreasing the nucleation and growth rate of diamond coating). To increase the diamond nucleation rate and prevent the rapid diffusion of hydrogen and carbon into the substrate, different surface treatments and interlayers were studied in this paper. Results showed that after polishing with diamond pastes and ultrasonically pre-treatment in diamond suspensions, the nuclei density of diamond crystals increased significantly. However, it could not prevent the diffusion of hydrogen into the substrate. Pre-etching of titanium substrate using hydrogen plasma for a short time significantly increased the nuclei density of diamond crystals. Results showed that on TiN interlayer, there was no significant improvement in diamond nucleation and growth, and the deposited diamond coatings showed a poor adherence. There were two mechanisms for diamond nucleation on diamond-like-carbon (DLC) interlayer. DLC film was etched by hydrogen plasma and changed to diamond crystals, and at the same time, new diamond crystals were formed on DLC interlayer in which DLC acted as the precursor for diamond nucleation. However, the so-formed diamond coating had poor adhesion strength. Plasma nitrided layer could prevent the rapid diffusion of hydrogen and carbon into titanium substrate, but results showed a relatively low nucleation density of diamond crystals and poor adhesion. A graded interlayer combining plasma nitriding followed by plasma carbonitriding was effective in preventing the rapid diffusion of hydrogen and carbon into the substrate and improving the nucleation rate and adhesion of diamond coating.

Ultrafine Grained Materials: Severe Plastic Deformation Processing: I

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

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

Tuesday AM — Room: Polk A/B
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Naresh N. Thadhani, Georgia Institute of Technology, Atlanta, GA 30332-0245 USA

8:30 AM Invited

Factors Influencing the Development of Ultrafine Grained Materials through Severe Plastic Deformation: Minoru Furukawa[2]; Zenji Horita[3]; Minoru Nemoto[3]; *Terence G. Langdon*[1]; [1]University of Southern California, Depts. of Matls. Sci. & Mech. Eng., Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Munakata, Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Eng., Faculty of Eng., Fukuoka 812-8581 Japan

Equal-Channel Angular (ECA) pressing is a processing method which may be used to achieve substantial grain refinement. Typically, metals subjected to ECA pressing have grain sizes in the submicrometer range. This paper considers the factors influencing the development of a homogeneous microstructure of equiaxed grains separated by high angle grain boundaries, including the pressing speed, the strain introduced on each passage through the die and the effect of specimen rotation between consecutive pressings.

8:55 AM Invited

Formation of Equilibrium Ultrafine Grain Structure in a 6061 Aluminum Alloy: R. Kaibyshev[2]; F. Musin[2]; D. Gromov[2]; *D. R. Lesuer*[1]; [1]Lawrence Livermore National Laboratory, L-342 P.O. Box 808, Livermore, CA 94551 USA; [2]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia

Ultrafine grain structures have been produced in a 6061 aluminum alloy using ECAE. The average size was 0.4-0.5 μm. A highly non-equilibrium structure was produced with a high density of extrinsic dislocations at grain boundaries which are sources of long-range stress fields. As a result, the alloy demonstrates poor workability and insufficient service properties. Two processing approaches for reducing the internal stress were evaluated. The first approach involved recrystallization annealing which was effective in relaxing internal stresses. However, the relaxation of internal stresses was accompanied by dramatic grain coarsening. Using a recrystallization anneal, microstructures with average grain size less than 1.5 μm could not be produced which were free of internal stresses. In addition, this structure is non-uniform. Secondary recrystallization was observed to occur with the formation of grains which were greater than 10 μm. An attractive alternative for the conversation of a non-equilibrium ultrafine grain structure into an equilibrium one is via plastic deformation. The microstructural evolution during plastic deformation of the 6061 aluminum alloy with an initial submicron structure will be discussed in detail. It was shown by X-ray analysis, TEM examinations and microhardness tests that plastic deformation led to a decrease of internal elastic stresses due to recovery of grain boundary dislocations. Adsorption of grain boundary defects and the formation of a recrystallized structure occurs after high strains and the microhardness decreases. This process was not accompanied by significant grain growth and the crystallite size was essentially stable during plastic deformation. As a result, the formation of an equilibrium submicrocrystalline structure took place. A mechanism of transformation of the non-equilibrium submicrocrystalline structure into a recrystallized one will be considered.

9:20 AM

Microstructural Evolution in an Al-1.7at.%Cu Alloy Deformed by Equal-Channel Angular Pressing: *M. Murayama*[1]; K. Hono[1]; Z. Horita[2]; [1]National Research Institute for Metals, Tsukuba 305-0047 Japan; [2]Kyushu University, Dept. of Matls. Sci. and Eng., Fukuoka 812-8581 Japan

The microstructural evolution during the equal channel angular (ECA) pressing process of an α+θ' two-phase Al-1.7at.%Cu alloy has been studied by transmission electron microscopy (TEM) and energy-filtered TEM. The θ' precipitates are severely deformed after the first pass of ECA pressing that with a strain of e~1. After 8 passes of ECA pressing, most of the θ' precipitates are dissolved and a fine grained (~500nm) supersaturated solid solution is obtained. When the ultrafine grain single phase alloy prepared by ECA pressing is aged for 24 h at 100°C, precipitation of the equilibrium θ phase is observed along the grain boundaries, whereas only GP zones are formed in the undeformed specimen. This suggests that the grain boundaries in the ultrafine grained microstructure provide heterogeneous nucleation sites for the θ precipitates. The supersaturation required for precipitation of the GP zones is lost quickly by diffusion of Cu to the grain boundaries.

9:40 AM

Ultrafine Grain Structure in the Friction-Stir Welding of Aluminum Alloy 2024 at Low Temperatures: *Samuel Benavides*[1]; Ying Li[1]; Lawrence E. Murr[1]; [1]The University of Texas at El Paso, Metallu. and Matls. Eng., 500 W. University Ave., El Paso, TX 79968-0520 USA

Friction-stir welding involves a solid-state intercalation process facilitated by dynamic recrystallization which accommodates superplastic flow. Adiabatic and frictional heating, a result of high strain and high strain-rate deformation, drive the recrystallization process. In this study we examined the residual grain structures for aluminum alloy 2024 friction-stir welded at starting temperatures which ranged from roughly 30°C to -100°C. The ability to weld any metals or alloys at -100°C is in itself rather extraordinary, especially since the steady-state (recrystallized) grain size appears to be somewhere between 0.5 μm and 0.8 μm. At higher welding temperatures the residual grain size in the weld zone increases to near 10 μm as a result of grain growth. At -100°C the welding produced a very narrow temperature spike of around 240°C, but the residual grain sizes remained in the sub-micron range. In this case the welding was actually performed with the workpiece submerged in liquid nitrogen. Microstructures have been examined in detail using transmission electron microscopy. The weld zone properties were monitored by measuring and comparing residual microhardness profiles through the stir-welded zone and extending into the base metal (workpieces). Research supported in part by a NASA Cooperative Agreement NCC8-137 and a Mr. and Mrs. MacIntosh Murchison Endowment.

10:00 AM

Amorphization of Al-Sm-Based Alloys by Cold-Rolling: *Rainer Johannes Hebert*[1]; [1]University of Wisconsin-Madison, 1500 Engineering Dr. 1217 ERB, Madison, WI 53706 USA

Melt-spinning and splat-quenching as traditional production techniques for amorphous materials often result in the formation of quenched in nuclei and subsequent nanocrystal formation during processing. Intense straining and cold-rolling offer not only the opportunity for the formation of bulk amorphous material but also for a new approach to study the nanostructural evolution during processing. Cold-rolling of Al92Sm8 yielded a partially amorphous sample that revealed a clear glass transition during linear heating in DSC. Modulated DSC reflected a similar Tg for melt-quenched Al92Sm8 thus indicating that amorphous Al92Sm8 is a true glass in contrast to amorphous, short-range ordered structures. A systematic study of Al-Sm based alloys following cold-rolling has been conducted in terms of the dependence of the glass-transition behavior as well as the grain size and micro-

structure on both the number of rolling cycles and solute concentration. The effect of multicomponent-alloying on the amorphization is accounted for by the influence of selected Ni-additions on the cold-rolling response of Al-Sm alloys. This experience has demonstrated that intense cold-rolling can yield glass/nanocrystal-structures and has the potential for synthesis of bulk nanocrystal or amorphous structures. The support of the ARO (DAAG 55-97-1-0261) is gratefully acknowledged.

10:20 AM Break

10:30 AM Invited

Severe Deformation Based Process for Grain Subdivision and Ultrafine Microstructures: *A. K. Ghosh*[1]; W. Huang[1]; [1]The University of Michigan, Dept. of Matls. Sci. and Eng., Ann Arbor, MI 48109 USA

Significant interest exists in the materials science community in creating nanocrystalline microstructures in metallic alloys in an effort to obtain improved strength and processability in these alloys. Vapor deposition processes have been used for producing small scale nanostructured samples, but repeated deformation methods, such as 3-axis forging, ECAE (Equal Channel Angular Extrusion), pressurized torsion etc. are found more convenient to create bulk materials with 50 ñ 500 nm size microstructures. Stability of these microstructures to further deformation and to thermal exposure is however a major problem which needs to be addressed in the future years. If the desired product is a sheet, further rolling is required after the severe deformation steps, and this has been found to alter their microstructure. Another issue for process scale-up is die-wall sticking with the moving workpiece, which make scale-up of billet size a nontrivial problem. To minimize these structure-altering aspects of rolling deformation, a surface-shear based deformation technique has been developed and applied to plates and sheets of aluminum alloys to create desired fine-scale microstructures. Initial studies on several Al alloys containing dispersoid particles have been analyzed by optical, and electron microscopy. The role of dispersoids in the structural stability has been studied as a function of annealing temperature and time. This talk will summarize our results to date on this investigation.

10:55 AM Invited

The Influence of Processing Route on the Evolution of Microtexture and Grain Boundary Character during ECA Pressing of Pure Aluminum: *Terry R. McNelley*[1]; Douglas L. Swisher[1]; Zenji Horita[2]; Terry G. Langdon[3]; [1]Naval Postgraduate School, Dept. of Mech. Eng., 700 Dyer Rd., Monterey, CA 93943-5146 USA; [2]Kyushu University, Dept. of Matls. Sci. and Eng., Fukuoka 812-8581 Japan; [3]University of Southern California, Dept. of Matls. Sci. and Mech. Eng., Los Angles, CA 90081-1453 USA

Severe plastic deformation by equal-channel angular (ECA) pressing is a promising approach to achieving extreme grain refinement in aluminum and its alloys. Grain sizes of about 1.0 µm have been reported for pure aluminum processed by repetitive ECA pressing and submicron grain sizes have been attained in aluminum-magnesium alloys. From investigations by transmission electron microscopy the evolution of the deformation microstructure and the roles of recovery and recrystallization during repetitive pressing operations are significantly dependent on the details of the process route. Direct measurement of grain-to-grain misorientation by use of recently developed computer-aided electron backscatter diffraction (EBSD) analysis methods enables the evaluation of microtexture and grain boundary character. These methods have been employed here to examine the evolution of the grain-to-grain misorientation as well as microtexture during repetitive ECA pressing of pure aluminum. Implications to selection of processing conditions will be discussed.

11:20 AM

Characterization of Sub-Micrometer Structures in Heavily Deformed Metals Over the Entire Misorientation Angle Range: *Darcy A. Hughes*[1]; Niels Hansen[2]; [1]Sandia National Laboratories, Ctr. for Matls. and Eng. Sci., P.O. Box 969, M.S. 9403, Livermore, CA 94551-0969 USA; [2]Risø National Laboratory, Matls. Rsch. Dept., P.O.Box 49, Roskilde DK-4000 Denmark

Grain subdivision during deformation results in a distribution of both low and high angle dislocation boundaries surrounding nanometer to micrometer scaled volumes. Key characteristics of these boundaries, including morphology, as well as spacing and misorientation angle distributions, are quantified using high resolution transmission electron microscopy and TEM Kikuchi diffraction as a function of the deformation. It is observed that these boundaries refine the microstructure and that the fraction of high angle boundaries increases with increasing deformation. Different deformation modes and material type can modify the boundary spacing and angle distributions as well as the boundary morphology. A comparison is made between monotonic deformation by rolling or simple shear and more complex paths such as cyclic-extrusion-compression and equal-channel-angle-extrusion. The question is considered of whether these deformation induced high angle boundaries constitute a kind of grain refinement. Part of this work was supported by the Office of Basic Energy Sciences, U.S. DOE under contract No. DE-AC04-94AL85000.

11:40 AM

Ultra-Fine Grained Ferrous and Aluminum Alloys Produced by Accumulative Roll-Bonding: *Nobuhiro Tsuji*[1]; Yoshihiro Saito[1]; Yoshinori Ito[2]; Hiroshi Utsunomiya[1]; Tetsuo Sakai[1]; [1]Osaka University, Dept. of Matls. Sci. & Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan; [2]Graduate Student of Osaka University, Dept. of Matls. Sci. & Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

Ultra-grain refining of metallic materials by intense straining has been significantly studied. We have recently developed a novel intense straining process applicable to large structural materials, the Accumulative Roll-Bonding (ARB). In ARB, the rolled material was cut, stacked to be the initial thickness, and rolled again. In order to obtain solid material, the rolling in the ARB is not only a deformation process but also a bonding process (roll-bonding). The ARB was applied to various aluminum alloys, interstitial free steel and low-carbon steel. At various temperatures in the range from room temperature to hot-rolling temperatures, the ARB was successfully carried out. Especially at warm temperatures below recrystallization temperature, the ARBed sheets were filled with ultra-fine grains whose diameter was several hundred nano-meters. Above recrystallization temperature, it was impossible to accumulate strain to obtain ultra-fine grains because of enhanced restoration during reheating. The materials ARBed at ambient temperature did not show ultra-fine grains but complicated deformation structure, however, ultra-fine grains formed in them by low temperature annealing. The formation process of the ultra-fine grains will be discussed. Effect of lubrication in roll-bonding on the properties and the microstructure of the material was also clarified. The ultra-fine grained materials showed very large strength two to four times larger than that of the starting materials at ambient temperature. Interestingly discontinuous yielding appeared even in pure aluminum and IF steel if they had ultra-fine grains. Though the ultra-fine grained materials showed limited ductility (especially uniform elongation) at room temperature, some of them showed low temperature superplasticity at elevated temperature.

TUESDAY PM

12th International Symposium on Experimental Methods for Microgravity Materials Science: Session 2

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

Tuesday PM Room: Memphis A
March 14, 2000 Location: Opryland Convention Center

Session Chair: C. Patuelli, Universita di Bologna, Dept. di Fisica ed Istituto Nazionale di Fisica della Materia, Bologna I-40127 Italy

2:00 PM

Electrostatic Levitation Processing of Zr-Nb-Ni-Cu-Al Bulk Metallic Glass Forming Alloys: *C. C. Hays*[1]; J. Schroers[1]; U. Geyer[1]; S. Bossuyt[1]; N. Stein[1]; W. L. Johnson[1]; [1]California Institute of Technology, Dept. of Matls. Sci., 138-78, Pasadena, CA 91125 USA

The bulk glass forming ability in the Zr-Nb-Cu-Ni-Al alloy system is well established. One of these bulk metallic glasses, $Zr_{57}Nb_5Cu_{15.4}Ni_{12.6}Al_10$ (Vit 106), developed at Caltech exhibits an exceptional glass forming ability (GFA) and has an excellent thermal stability with respect to crystallization. For example, the width of the supercooled liquid region ΔT=(Tx-Tg), as determined from calorimetric measurements is ΔT 70K. This alloy has been investigated by our group in recent electrostatic levitation (ESL) experiments conducted at the NASA Marshall Space Flight Center Electrostatic Levitation facility. Independent ESL experiments on Vit 106 specimens show conclusively that the Time-Temperature-Transformation (TTT) diagram for this alloy is a superposition of high and low temperature branches. The nose of the TTT diagram is located at tnose 1-2 seconds, which prevents vitrification of Vit 106 specimens on free cooling in the ESL. As part of our continuing investigations of alloys in the Zr-Nb-Cu-Ni-Al system, we have identified a neighboring composition, Zr58.5Nb2.8-Cu15.6Ni12.8Al1 0.3, that exhibits a substantially improved GFA compared to Vit 106. This alloys is easily vitrified by standard techniques; e.g., arc melting and melting on a water cooled silver boat apparatus. Using these methods, the critical casting thickness for this composition is near 1cm. The calorimetrically determined ΔT values are as large as ΔT 100K. Differential thermal analysis (DTA) measurements show that the onset of melting for this new alloys is 10 K less than that of Vit 106. When examined in the ESL the new alloys are vitrified by purely radiative cooling. This is the first non-Be containing alloy to be vitrified on free cooling in the ESL. This places the microstructural properties of the ESL processed materials were examined by x-ray diffraction, electron- and optical-microscopy, and thermal analysis methods. These results are used to interpret the complex evolution of the glass forming properties observed in this alloy system.

2:20 PM

Solution Growth of Semiconductor in an Axial Static Magnetic Field: *Yuko Inatomi*[1]; Ayako Kato[1]; Kengo Horiuchi[1]; Takao Maki[2]; Kazuhiko Kuribayashi[1]; [1]The Institute of Space and Astronautical Science, 3-1-1 Yoshinodai, Sagamihara, Kanagawa 229-8510 Japan; [2]Olympus Optical Company Limited, 2951 Ishikawa-chou, Hachiouji, Tokyo 192 Japan

In most of crystal growth experiments of semiconductor under microgravity, the materials were analyzed by static research methods on the ground after the completion of the mission. An in situ observation is considered as a promising methodology during microgravity experiment in order to make full used of the limited experimental opportunities and their resources, because simultaneous observation of the growing interface and the environment can more precisely reveal the influence of supersaturation, temperature and concentration gradients, convection, and surface kinetics on the growth behavior. An in situ observation setup for solution growth process of semiconductor was developed for a high static magnetic field experiment based on near-infrared microscopy. Subsequently the axial magnetic field experiments from 0 to 6 were performed to reveal the influence of strongly-damped fluid flow on the growth surface. As a result, the dissolution and growth rates were successfully obtained using the setup with a high accuracy. The morphological instability of the surface will be discussed in detail.

2:40 PM

The Microgravity Diffusion Experiment for Very Reactive Liquid Metals: *Toshio Itami*[1]; Akitoshi Mizuno[1]; Hirokatsu Aoki[1]; Minoru Kaneko[1]; Tomoharu Fukazawa[1]; Akira Tanji[1]; Yoshito Arai[1]; Kazumasa Goto[1]; Yukiko Yamaura[1]; Natsumi Tateiwa[1]; Sinnichi Yoda[1]; Tadahiko Masaki[1]; Tomihisa Nakamura[1]; Naokiyo Kosikawa[1]; Yukihiro Nakamura[1]; [1]National Space Development Agency of Japan, Tsukuba Space Ctr., 2-1-1 Sengen, Tsukuba, Ibaragi, Japan

The space experiment still has many different features from the laboratory experiments, for example, there is only one chance without the repetition, strictly a requirement for safety, the proceed of long time since sample is delivered, etc. Therefore it is not always easy to perform the microgravity experiments for very reactive materials. In this report description is given for the experimental method for diffusion experiments of liquid Li under microgravity and its application to rocket experiment due to the TR-IA-6-rocket.

3:00 PM

Wetting Phenomena in Immiscible Alloys: *L. J. Little*[1]; J. B. Andrews[1]; [1]University of Alabama, Dept. of Matls. and Mech. Eng., Birmingham, AL 35294 USA

Ground-based processing of binary immiscible alloys leads to sedimentation. In an attempt to obtain dispersed microstructures, early experiments were performed in which immiscible alloys were processed in microgravity. However upon evaluation of the samples from these experiments, it was discovered that a separated structure was obtained, where one phase completely surround the other. One possible cause for this segregation in microgravity-processed immiscible alloys was believed to be perfect wetting. The purpose of this research is to investigate the wetting behavior in immiscible alloys and determine the mechanisms behind the segregation process. A transparent cell assembly will be used in order to permit direct observation of the solidification process. The succinonitrile-glycerol system and the succinonitrile-water system will be utilized in the investigation to evaluate the wetting phenomenon. It is anticipated that this research will give insight into perfect wetting behavior and information concerning its influence on the microstructures obtained from microgravity processing.

3:20 PM Break

3:40 PM

Combustion Synthesis of Porous Glass Ceramic Materials: *A. R. Manerbino*[1]; H. C. Yi[2]; J. Y. Guigne[2]; J. J. Moore[1]; F. D. Schowengerdt[1]; [1]Colorado School of Mines, Dept. of Metallu. and Matls. Eng., Golden, CO 80401 USA; [2]Guigne International Limited, Paradise, Newfoundland A1L 1C1 Canada

Combustion synthesis also know as, Self-Propagating High Temperature Synthesis (SHS), is a processing method which utilizes exothermic reactions to produce advanced materials which could prove to have many applications. This technique is used to produce glasses in

the present work. Thermodynamic analysis was carried out to determine the adiabatic temperature (T_{ad}), and actual combustion temperature (T_c), and wave velocity of several chemistries. Microstructural analysis was done using X-ray Diffraction (XRD), light microscopy, and Scanning Electron Microscopy (SEM) to determine the crystalline and glass-forming regions within the samples. Samples produced were porous, typically containing 50-70% porosity. Pores are generated by a number of mechanisms including green density and reactant powders before combustion reaction. This advanced material could be used for a wide variety of applications including filters, bone replacement material, or lightweight high-temperature applications.

4:00 PM

Surface Tension Measurements of Molten Silicon with the Techniques of Free-Fall and Electromagnetic Levitation: *Hideki Minagawa*[1]; Yusuke Goto[1]; Masataka Sasamori[1]; Hideaki Nagai[1]; Masaki Orihashi[1]; Yoshiho Ito[1]; Takashi Tsurue[1]; Yoshinori Nakata[1]; Keiji Kamada[2]; Takeshi Okutani[1]; [1]Hokkaido National Industrial Research Institute, Matl. Div., Agcy. of Industry and Sci. Tech., 2-17 Tsukisamu-Higashi Toyohiraku, Sapporo, Hokkaido prefecture Japan; [2]Japan Space Utilization Promotion Center, 3-30-16, Nishiwaseda, Shinjuku-ku, Tokyo, Japan

The results of surface tension of molten silicon measured by electromagnetic levitator have been compared with these by free-fall experiment. The comparable results of surface tension of molten silicon have been measured, which were demonstrated by electromagnetic (E-M) levitation and free-fall. The measurements by E-M levitator have been performed under normal gravity condition, while measurements by free-fall system have been carried out at the 1.2 s HNIRI (Hokkaido National Industrial Research Institute) drop tower. The values of surface tension derived by the frequency of surface oscillation, which had been measured by E-M levitation and free-fall, were different from each other because of the effect of the perturbation of E-M field for the measurement of surface tension.

4:20 PM

Undercooling Comparison of Pure Cobalt Processed in Crucibles and the MSFC Drop Tube: *Mike B. Robinson*[1]; Delin Li[1]; Tom J. Rathz[2]; [1]MSFC/NASA, Science Directorate 47, Huntsville, AL 35812 USA; [2]UAH, SD47, MSFC/NASA, Huntsville, AL 35812 USA

A large number of undercooling cycles was performed on a single Co drop and many separate samples using crucibles and the 105-meter drop tube. The effects of sample size, material purity, crucible, and flux agent on undercooling were investigated. For fluxing experiments, average and maximum undercooling was observed to be insensitive to sample diameter in the 1 to 10 mm range and material purity between 99.5 and 99.999%. Among the total of 1,155 nucleation events recorded for the same 4-millimeter sample, 90% undercooling values are clustered from 300 to 330 K. Comparably large undercoolings have been achieved in the low gravity, containerless environment of the long drop tube. Undercooling data are analyzed within the framework of classical nucleation theory coupled with statistical approach. The average grain size was decreased from 500 to 30 mm when the undercooling was increased from 20 to 320 K, demonstrating the grain refinement in undercooled pure cobalt.

4:40 PM

Solidification Processing of Immiscible Liquids in the Presence of Applied Ultrasonic Energy: *Shinwoo Kim*[1]; *Richard N. Grugel*[2]; [1]Hoseo University, Dept. of Matls. Sci. and Eng., Chung-Nam Korea; [2]Universities Space Research Association, Marshall Space Flight Ctr., MS-MSD47, Huntsville, AL 35812 USA

Uniform microstructural development during solidification of immiscible liquids on Earth is hampered by inherent density differences between the phases. Microgravity processing minimizes settling but segregation still occurs due to gravity independent wetting and coalescence phenomena. Experiments with the transparent organic, metal analogue, succinonitrile-glycerol system were conducted in conjunction with applied ultrasonic energy. The processing parameters associated with this technique have been evaluated in view of optimizing dispersion uniformity. Experimental results to evaluate microstructural phase distributions, based on other liquid-liquid immiscibility systems, will also be presented. Support from NASA grants NAG8-1231 and NCC8-66 is gratefully acknowledged.

Advanced Technologies for Superalloy Affordability: Affordability Technology for Wrought Alloys

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: K. M. Chang, West Virginia University, Mechanical & Aerospace Engineering, Morgantown, WV 26506 USA; K. R. Bain, GE Aircraft Engines, Cincinnati, OH 45215 USA; D. Furrer, Ladish Company, Cudahy, WI 53110 USA; S. K. Srivastava, Haynes International, Kokomo, IN 46904 USA

Tuesday PM — Room: Canal C
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Kenneth R. Bain, GE Aircraft Engines, Cincinnati, OH 45215 USA; David Mourer, GE Aircraft Engines, Lynn, MA 01910 USA

2:00 PM Invited

New Materials and Processing to Enhance Affordability: *D. L. Klarstrom*[1]; [1]Haynes International Inc., Eng. & Tech. Depts., P.O. Box 9013, Kokomo, IN 46904-9013 USA

Over the last several years, new alloys and processing have been introduced which offer the superalloy user enhanced affordability. This has occurred in the form of a lower cost material such as HR-120® alloy which has properties equivalent to currently used HASTELLO® X alloy. This alloy has also been found to serve as a significant upgrade to stainless steels for use in landbased gas turbine applications in order to increase engine capability. In another case, gas turbine engine manufacturers have been able to increase use temperatures for seal rings without the need for protective coatings through the use of HAYNES® 242™ alloy, a relatively new, high strength alloy with a low coefficient of thermal expansion. Recent studies have also pointed the way for a two-step heat treatment for the alloy that will substantially reduce the aging time required. The strength of this material can also be substantially increased by coldworking and aging to provide an exceptional alloy for bolting applications. Its high nickel content also provides excellent resistance to stress corrosion cracking. Enhanced fatigue resistance for HAYNES 230® alloy obtained via low temperature processing is also presented and discussed. HAYNES®, HASTELLOY®, HR-120 and 230 are registered trademarks of Haynes International, Inc. 242™ is a trademark of Haynes International, Inc.

2:25 PM Invited

Microstructure Modeling of Forged Components of Ingot Metallurgy Nickel Based Superalloys: *Gangshu Shen*[1]; [1]Ladish Company Inc., Adv. Matls. & Process Tech., P.O. Box 8902, 5481 S. Packard Ave., Cudahy, WI 53110-8902 USA

The application of FEM to superalloy forging made it possible to obtain the detailed thermomechanical histories of each individual location of the forged components. Microstructure modeling procedures were developed to make use of this information for grain size prediction. The same microstructure modeling approach was used for two ingot metallurgy superalloys: Waspaloy and 718. The microstructure evolution evolved in forging process is considered in the following way: static structural changes in preheating, dynamic structural changes in deformation, and static structural changes in dwell resting between operations and final cool down. The microstructure is updated for the entire thermal and deformation processes during a forging. This meth-

odology has been successfully used in various processes such as hydraulic press forging, mechanical press forging and hammer forging on numerous Waspaloy and 718 parts.

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Weight Reduction of Pneumatic Ducting Though Alloy Strength Enhancement: *Gaylord Smith*[1]; Dan Yates[1]; Dan Wallem[2]; [1]Special Metals Corporation, 3200 Riverside Dr., Huntington, WV 25705 USA; [2]Boeing Commercial Airplane Group, Propulsion Sys. Grp., P.O. Box 3707, MS 4T-80, Seattle, WA 98124 USA

Weight reduction of the engines for wide-body aircraft is a critical issue. One area where weight saving is possible is through the use of mill processing to dramatically increase the strength and fatigue resistance of the material employed in the pneumatic ducting, thereby making a reduction in gauge and cost from current alloys possible. Since one of the preferred alloys for this ducting is INCONEL alloy 625, a mill practice was developed to produce a 75 kis yield strength sheet product. This product is described in some detail including certain steps pertaining to the method of manufacture aimed at enhancing fatigue resistance.

3:10 PM

Development of Direct-Aged HAYNES® 242™ Alloy for Fastener Applications: *L. M. Pike*[1]; S. K. Srivastava[1]; M. C. Losch[1]; Carl E. Kelly[2]; [1]Haynes International, P.O. Box 9013, Kokomo, IN 46904 USA; [2]Pratt and Whitney, Aircraft Rd., Middletown, CT 06457 USA

One cost-effective solution for the development of materials is the adaptation of an established alloy for new applications. Improvements in certain application-critical properties may be obtained through modifications in chemistry, processing, etc. and can often be explored at a considerably lesser expense than a full-scale alloy development program. An example of this is the adaptation of HAYNES® 242™ alloy for high-temperature fastener applications. This low-expansion, high-strength alloy is used extensively in gas-turbine engines as a seal/containment ring material. However, in the standard annealed and aged condition the strength properties are less than requisite for certain fastener applications. The development of a direct-aging process (cold-work + aging) has resulted in a material with significantly improved strength while retaining respectable ductility. Room-temperature (RT) yield strengths of more than 1750 MPa (250 ksi) are possible; this represents a 100% improvement over the standard annealed and aged condition. This dramatic increase in strength allows 242™ alloy to be considered for applications where it offers a unique combination of properties not found in competing materials such as alloy 718 or Custom 455®. These properties include low-thermal expansion as well as resistance to stress-corrosion cracking in both chloride and hydrogen environments. Potential applications for direct-aged 242™ alloy include fasteners and high-strength components in both aerospace and land-based gas turbines as well as other demanding industrial environments. HAYNES® is a registered trademark of Haynes International, Inc. 242™ is a trademark of Haynes International, Inc. Custom 455®. is a registered trademark of Carpenter Technology Corporation.

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Development and Utilization of Press Converted Powder Metal Superalloy Billet: *Anthony Banik*[1]; Xavier Pierron[1]; David Furrer[2]; Joe Lemsky[2]; Sushil Jain[3]; [1]Special Metals Corporation, 100 Industry Ln., Princeton, KY 42445 USA; [2]Ladish Company Inc., P.O. Box 8902, 5481 S. Packard Ave., Cudahy, WI 53110 USA; [3]Rolls-Royce Allison, P.O. Box 420, Indianapolis, IN 46206 USA

Powder metal superalloys for turbine disk applications have historically been produced by a two step hot compaction and extrusion sequence to consolidate and refine canned loose powder into consolidated billet material. Powder metal superalloys exhibit relatively low elevated temperature tensile ductility, and as such, extrusion processing has provided a means of bulk deformation under predominately compressive stresses. Recent advances in the consolidation of superalloy powders, which incorporate a sub-solidus hot isostatic press (SSHIP) consolidation practice, improves the consolidated billet ductility such that conventional conversion operations can be utilized to produce fine grain billet for subsequent disk forging. This alternate conversion route results in significant reductions in cost and lot size requirements. Results from disk forging evaluations indicate that through a controlled thermomechanical processing sequence, the microstructural response in the final components can be controlled to provide very uniform controlled grain sizes and ultrasonic inspectability.

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Modeling Grain Size Evolution of P/M Rene88DT Forgings: *Canan U. Hardwicke*[1]; Gangshu Shen[2]; David Furrer[2]; [1]General Electric Company, Corp. Rsrch. and Dev., One Research Circle, Niskayuna, NY 12309 USA; [2]Ladish Company, Adv. Matls. and Process Tech. Dept., 5481 S. Packard Ave., Cudahy, WI 53110-8902 USA

In a typical processing route for powder metal produced engine disks, forging is a key processing step influencing the component's final microstructure and hence, mechanical properties. The definition of a forge and heat-treat processing window to achieve the desired microstructures has been an iterative process. In this work, a microstructural model was developed for Rene88 to describe the change in grain size of a billet as it is isothermally forged and then heat treated. The first step was to develop a grain size database using isothermal compression tests with varied temperature and strain rate. Then empirical equations were developed to describe the deformation and annealing behavior. These equations were then integrated with an FEM code to predict the local microstructure variations for a given part geometry. Application of this model should reduce the trial-and-error steps in processing R88 and other commercial superalloys into useful component configurations. This work was supported by the AFRL under the contract number F33615-95-C-5229.

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HIP Bonding of Multiple Alloys for Advanced Disk Applications: *Tammy Marie Simpson*[1]; Allen R. Price[1]; Paul F. Browning[2]; Michael Fitzpatrick[2]; [1]Howmet Research Corporation, Adv. Tech., 1500 S. Warner St., Whitehall, MI 49461 USA; [2]Solar Turbines Inc., Adv. Tech. Matls. and Process., 2200 Pacific Hwy., San Diego, CA 92186 USA

Dual property gas turbine disks were investigated to optimize properties in both the rim and hub sections of the disk. For the rim material, it is necessary to have good high temperature creep rupture life. However, for the hub material, both high burst strength and good low cycle fatigue are important. These properties would increase turbine fuel efficiency by allowing an increase in the turbine inlet temperature and a reduction in the disk cooling air requirements. In the past, rims and hubs of different materials have been consolidated into dual alloy wheels. However, deleterious particles, namely hafnium oxide, may have contributed to planar failure and reduced overall ductility. Howmet has demonstrated the ability to HIP bond disks with tailored material properties in specific areas without the formation of a planar oxide layer. The alloys investigated were Mar-M 246, Mar-M 247 LC, IN 792 Mod5A, and U720 in cast-wrought, Spray-Cast®, and powder metallurgy form.

4:50 PM

A New Method to Improve the Hot-Workability of High-Strengthened Superalloys: *Zhengdong Long*[1]; Jingyun Zhuang[1]; Bo Dong[1]; *Di Feng*[1]; Zengyong Zhong[1]; [1]Central Iron and Steel Research Institute, Dept. of Superalloys, No.76, Xue Yuan Nan Rd., Hai dian District, Beijing 100081 PRC

A new method based on the optimization of microstructure to improve the hot-workability of high-strengthened superalloys is proposed. By this method, the coarsened gamma prime phase is obtained by lower cooling rate at specified temperature range. This paper discusses the detailed processing, microstructure change, hot deformation flow stress, ductility and recrystallization microstructure. The results show that the new method is very effective to improve the hot-workability of this kind of superalloys. Conducting this process, the deformation uniformity is enhanced greatly, the flow stress is lowered and the hot deformation ductility is increased obviously. The mechanism to improve the hot-workability can be explained that the coarsened gamma prime phase optimizes the distribution of alloying elements and lessens its solution strengthening effect at high temperature.

Aluminum Reduction Technology: MHD/Modeling

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

Tuesday PM — Room: Sewanee
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Jeff Keniry, Alumination Consulting, Director, Mount Macedon, Victoria 3441 Australia

2:00 PM Invited
Metal Pad Instabilities in Aluminium Reduction Cells: D. Shin[1]; *Alfred David Sneyd*[1]; [1]University of Waikato, Dept. of Math., Private Bag 3105, Hamilton 2001 New Zealand

The electric current flowing through an aluminium reduction cell generates an associated magnetic field, and hence a Lorentz force-field which can destabilise the cryolite-aluminium interface. The cell interior can be regarded approximately as two-fluid system (cryolite/aluminium) in a rectangular container. Determining the waves which can be excited in the system is a classical problem in hydrodynamics, but the Lorentz force adds a new twist. Stability can be analysed by expressing the (small amplitude) wave system as a combination of normal modes, which in the absence of a force field or energy dissipation are non-interacting and of constant amplitude. The Lorentz force however couples the normal modes and may lead to amplitude growth, disrupting the efficient operation of the cell, particularly if two normal modes have similar frequencies (resonance). The cell magnetic field and anode-cathode separation are crucial in determining stability, and a simple physical explanation can be give for the driving mechanism.

2:25 PM Invited
Multi-Physics Modeling of Aluminium Reduction Cells: *Mark Cross*[1]; K. Pericleous[1]; L. Leboucher[1]; T. N. Croft[1]; V. Bojarevics[1]; A. Williams[1]; [1]University of Greenwich, Ctr. for Numerical Modeling & Process Analysis, Wellington St., Woolwich, London SE18 6PF UK

Aluminium cells involve a range of complex physical processes which act simultaneously to provide a narrow satisfactory operating range. These processes involve electromagnetic fields, coupled with heat transfer and phase change, two phase fluid flow with a range of complexities plus the development of stress in the cell structure. All of these phenomena are coupled in some significant sense and so to provide a comprehensive model of these processes involves their representation simultaneously. Conventionally, aspects of the process have been modeled separately using uncoupled estimates of the effects of the other phenomena; this has enabled the use of standard commercial CFD and FEA tools. In this paper we will describe an approach to the modeling of aluminium cells which describes all the physics simultaneously. This approach uses a finite volume approximation for each of the phenomena and facilitates their interactions directly in the modeling-the complex geometries involved are addressed by using unstructured meshes. The very challenging issues to be overcome in this venture will be outlined and some preliminary results will be shown.

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Towards a More Stable Aluminium Cell Via Busbar Configuration Optimization: M. F. El-Demerdash[1]; *A. A. Adly*[1]; S. E. Abu-Shady[1]; W. Ismail[1]; F. M. El-Dawi[2]; [1]Cairo University, Faculty of Eng., Cairo, Giza Egypt; [2]Egyptalum-Aluminium Company of Egypt, Cairo, Egypt

A modification is suggested on the present busbar design used in the prebaked end to end 203 kA Aluminum production cells at EGYPTALUM, Nage-Hammady, Egypt. Both the base configuration currently working and the modified configuration are computed using PACEM3 package for magnetic fields, forces, and stability figures. Results showed more symmetric magnetic fields especially for vertical field component, and more stable stability figures for the modified design as compared to the base design. To reach the modified design, several busbar configurations were considered. Details of those configurations, computed fields results, and corresponding stability levels are presented in detail in the paper.

3:15 PM
Thermo-Electric Design of a 400 kA Cell Using Mathematical Models: A Tutorial: *Marc Dupuis*[1]; [1]GeniSim Inc., 3111 Alger St., Jonquiere, Quebec G7S2M9 Canada

This paper presents a typical application of thermo-electric mathematical models to produce a thermally balance aluminum reduction cell lining design. The paper is structured as a tutorial, the selected example is a modern prebaked PBF cell running at 400 kA. The type of models used are the now standard steady state thermo-electric 3D full cell slice model and the "lump parameters" dynamic process model as well as the newly developed dynamic thermo-electric 2D+ full cell slice process model.

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Electrolysis Cells with Symmetric Magnetic Field: *A. Panaitescu*[1]; *G. Dobra*[2]; [1]University Politehnica of Bucharest, Electrical Eng., Spl. Independentei 313, Bucharest 77206 Romania; [2]S.C. ALRO S.A., General Manager, Str. Pitesti 116, Slatina, Romania

In the cells with symmetrical magnetic field the vertical oscillations of the surface of the molten aluminium have been strongly decreased. This fact has been showed experimentally by the oscillography of the currents of the anodic rods. A standard cell in Hall no 9 loses its stability when the reference voltage decreases below 4V. In the case of the cells that work in symmetrical field, the instabilities appear at lower reference voltages. The reference voltage has been decreased to 3.65 V, and oscillations of the currents with long periods have not appeared. This is a proof that on the surface of the molten metal there are no waves. There are only oscillations with a frequency of about a second, due to the accumulations of gases under the anodes, and which determines only the decrease of the currents through rods. Long periods of time have been necessary to demonstrate the advantages of the technology in symmetrical field. Thus, at present, the data base contains the main technical indicators of cells with symmetrization for the last 910 days (two years and a half). At an industrial cell it can not be reduced only the reference voltage, because the thermal balance of the cell would be modified. The decreases of the voltages have been done with special precaution. The functioning characteristics have been averaged on 30-day periods (monthly), on 6-month periods and one year, because they have relatively important variations in time. The variation of the specific consumption of electric energy, of the efficiency of the electrolysis process (Faraday efficiency), and of the voltage drop on cell have been recorded, and the values of these quantities have been graphically represented. (Figures 1 and 2). One can notice that these cells can function at relatively low reference voltages (3.65 V has been the lowest reference voltage that has been tested and at which the cells have functioned in stability). On the x axes of the graphics there is the month the averaged value refers to. The first month is October 1996. The media have been done on groups of 6 symmetrical cells and 122 cells in the electrolysis Hall no 9 of the Aluminium Works. A specific consumption of electric energy below 13 MWh/t is a remarkable parameter, if one takes into account the type of industrial cells working in symmetrical field. (The experimental cells are open cells). In the second half of 1999 the cells with symmetrization will have a central, punctual supply, and each of them will be controlled by a microprocessor. Under these new circumstances the overvoltages due to anodic effects will be lower and it is expected that the Faraday efficiency should increase. A specific consumption of 12,5 MWh/t is achievable, which will probably be a world novelty for industrial electrolysis cells.

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Visualization of the Metal Pad Waves in the Aluminum Reduction Cell with Pre-Baked Anodes: *A. Panaitescu*[1]; A. Moraru[1]; *I. Panaitescu*[1]; [1]University Politehnica of Bucharest, Electrical Eng., Spl. Independentei 313, Bucharest 77206 Romania

In Hall-Heroult aluminum reduction cells the vertical oscillations of the metal pad have a great influence on the current and energy efficiency of the reduction process. In order to establish the least average anode to cathode distance which ensures a quiet functioning of the cell, a on-line method is necessary to survey the metal pad waves in different operation stages. In the cell with pre-backed anodes the electrolysis current is lead through several-usually four-stubs embedded in each anode block, as in Fig. 1. By measuring the beam to aluminum pad voltage drop and the stub currents, and by using an adequate model of the anode block quart resistance, including the electrolyte layer, the average thickness of the electrolyte bath below each anode quart can be established. On this basis a monitoring system was developed, using an 64 channel acquisition system, which is measuring the stub currents, with a sampling interval of 10 ms. After conditioning the acquired signals and by interoplating in space, for each set of data can be shown the profile of the pad surface. When are not known the channel scale factors stub current/voltage drop, a self-calibration of the data is used, accomplished on a "quiet" operation section. Several post-processing and plotting facilities are provided: plotting of the acquired or filtered signals, versus the time moment of acquisition, separated or grouped together on anode, plotting of the self-calibrated signals on anode rows, plotting of animated 3D aluminium pad's surface, determined with 64 stub signals, and plotting of animated 3D aluminium pad's surface, determined with 16 anode signals. The last two processing are based on the assumption that the signals (currents) vary inversely with the electrolyte layer thickness. For the 3D plot an weighted, direct and inverse, interpolation is used between the centre points which correspond to the acquired signals, using the function griddata from MATLAB. Below two 3D plots are shown (Fig.2 and Fig. 3), which illustrate the results obtained with the functions: cellfq.m for 64 stub signals and cellgq.m for 16 anode signals. The last plot is more poor in details as the first one, because also the primary information is more poor. The 3D plots may be animated, according to the duration of the acquisition cycle (0.64 s), what allows to follow the moving aluminium waves in the cell.

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Model of Process of Electrolysis: A. G. Barantsev[1]; *Vladimir V. Yurkov*[1]; V. C. Mann[1]; T. V. Piskazhova[1]; K. F. Nikandrov[1]; [1]PSC Krasnoyarsk Aluminum Plant, Krasnoyarsk 660111 Russia

A model of the process of reduction of aluminum metal has been developed allowing to describe in the first approximation the dynamic process variables such as bath temperature and bath chemistry, ACD, frozen cryolite ledge thickens, operating voltage and others. A mathematical model comprises several sub-models dedicated to different aspects of reduction process. Based on the presented mathematical model a virtual ("soft') cell was developed. A virtual ("soft") cell can be used to run analyses of various situation in a real ("hard") operating cell, for prior testing the process control algorithms and establishing optimum operating conditions for the newly designed cells and personnel training. Some fragments of the model can be incorporated into the process control system for doubly connected temperature/ratio control.

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3D Thermo-Electric Field Modeling Tool and Its Application for Energy Regime Simulations in Aluminum Reduction Cells: S. A. Sherbinin[1]; V. V. Pingin[1]; A. G. Barantsev[1]; *P. V. Polyakov*[1]; [1]PSC Krasnoyarsk Aluminum Plant, Krasnoyarsk 660111 Russia

The experience in software developing and application for various Russian aluminium plants is confirmed that parallel with universal software complexes, relatively small mobile programs for personal computers have the right to exist. The interface of these programs makes it possible for technologists and designer to work with them without any thorough training in the area of computer technique. Effective algorithms and relatively high speed of calculations allow to check a lot of versions. The developed complex was applied for calculation and analysis of 2d and 3d distributions of thermal and electrical fields in various industrial apparatus for aluminum manufacturing. The models were implemented for both cell operation optimization and new designs developing. Computer investigations of the transfer processes in aluminium reduction cells with a gas evolving anode carried in the following direction. A gas bubble does not practically conduct electrical current and it therefore insulates a certain part of the electrode surface. The 3d mathematical model for the analysis of these phenomena comprises a differential equation, describing the growth and changes of the bubble shape versus time and the Laplace equation for the electrical potential. On the basis of this model the relationship between voltage drop in electrolyte and shape, size and number of bubbles is obtained.

Carbon Technology: Green Mill and Anode Baking

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

Tuesday PM — Room: Knoxville A
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Nigel R. Turner, Tarconord Group, Bitmac Limited, Scunthorpe, North Lincs DN15 6UR UK

2:00 PM

Coke Characteristics from the Refiners to the Smelters: *Raymond C. Perruchoud*[1]; Markus W. Meier[1]; Werner K. Fischer[1]; [1]R&D Carbon Limited, Rsrch. and Dev., P.O. Box 362, Le Châblé, Sierre, VS 3960 Switzerland

Modern smelter technologies require consistent anode behaviour ending with long anode cycles for low gross and net carbon consumptions. Therefore high density but thermal shock resistant anodes as well as high performance forming machines have been developed in the last decade to match this goal. From the raw material side the coke properties like: pulverizing factor and grain stability, and bulk density and resiliency are becoming critical for the production of crack free anodes as they influence not only the fines preparation and pitching aspects but also the extent of lamination of the green anodes after forming and the crack propagation resistance. These mechanical and physical properties are influenced by the type of resid oil but also by the coking and calcining conditions. The impacts of the coke macrostructure (isotropy) from the coking to the calcining steps are reviewed. The blending aspects of green or calcined cokes with different macrostructures and sulfur content are also reviewed taking into account the anode burning behaviour. This knowledge allows optimizations of the coke production from the refineries to their usage in the carbon plant.

2:30 PM

Process Adaptations for Finer Dust Formulations: Mixing and Forming: *Kirstine L. Hulse*[1]; Raymond C. Perruchoud[1]; Werner K. Fischer[1]; Barry J. Welch[2]; [1]R & D Carbon Limited, P.O. Box 362, Sierre CH3960 Switzerland; [2]University of Auckland, Chem. and Matls. Eng., Private Bag 92019, Auckland, New Zealand

Traditionally recipes have used 3000 Blaine dust in the carbon paste due to limitations that existed previously in the processing equipment, particularly in the classifying, weighing, preheating and mixing stages. The optimum processing conditions will change for different recipe conditions and different equipment capabilities. For a given paste plant design, the recipe should be chosen requiring optimum processing conditions as dictated by the plant equipment limitations. This investigation demonstrates how the paste consistency of different recipes can be altered through adjusting the parameters in the preheating, mixing, forming and cooling stages to produce differing levels of anode quality.

This has been executed on pilot and production scale, vibrated and pressed anodes.

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A Method to Determine the Optimal Baking Level of Carbon Anodes: *Marilou McClung*[1]; J. Anthony Ross[1]; Gerald Chovanec[2]; [1]Century Aluminum, Primary Products, P.O. Box 98, Ravenswood, WV 26164 USA

The carbon's performance in the reduction cell is influenced by many factors including, but not limited to, raw materials, aggregate preparation, mixing, forming, and anode baking. To improve the anode's behavior in the electrolysis process each of these steps must be optimized to reduce net carbon consumption. Century's Primary Products Division has developed a systematic method to determine the maximum anode efficiency that can be achieved by the Baking Furnace operation. The method includes comparing baked anode core properties to a bake level indicator. Then using the R & D Carbon Net Carbon equation, the best possible combination of baked anode core properties can then be determined and bake levels adjusted to maximize baked anode performance. This paper will detail the procedures used to maximize the efficiency of the baked carbon, the method to review overall performance of a typical pit distribution and the method of determining bake level.

3:20 PM

A Method to Correlate Raw Material Properties to Baked Anode Core Performance: *Marilou McClung*[1]; J. Anthony Ross[1]; [1]Century Aluminum, Primary Products, P.O. Box 98, Ravenswood, WV 26264 USA

With new environmental regulations effecting cokers, changes in the steel industry's production, changing product demands, and changing business climates, the sources of feed stocks to the coke calcining and pitch processing facilities are frequently changing in quality and quantity. These changes affect net carbon, anode density, return butt weight, pitch demand, or optimal aggregate sizing among other factors in a smelter. The challenge for Carbon Plants around the world is to produce a consistently high quality product while experiencing changes in the incoming coke and pitch. Smelters have always had difficulty tracking raw materials changes through the Green Mill, Baking, and Rodding to the anode performance in the cell. Century Aluminum has devised a method of tracking the raw materials from delivery to baked anode core. This paper will outline the methods used and give examples of the effects of raw material changes to the baked carbon.

3:45 PM Break

3:55 PM

Improvement of Existing Anode Baking Furnaces by Use of an Advanced Firing and Control System-Benefits and Results: *Detlef Maiwald*[1]; [1]LVE Verfahrenselektronik GmbH, Essen D45138 Germany

In the primary smelters, a lot of open pit baking furnaces are already a long time in operation for the production of anodes. Many of these furnaces are still manually controlled or just have a very basic automation. Due to this situation the fuel efficiency can be poor and the pollution control according to CO, NOx and unburned carbon emissions may not satisfy the environmental authorities. With the introduction of an advanced firing and control system the fuel efficiency, the quality consistency and the productivity can be highly improved. Advanced control algorithms using the Firing Index Module and the Flooding Monitor Module ensure the complete combustion of the volatile components and increase directly the fuel efficiency. A non parametric neural network model reproduces the basic structure of the specific furnace. On-line test sequences adapts this structure to the actual flue situation and baking condition. The on-line prediction of the anode temperature by a dynamical model is used for the supervision of critical temperature gradients in the anode during pitch burn and for fine tuning of the Firing Index Module to the final baking temperature. Special instrumentation like IR-sensors and opacity meters increase the reliability of the system and can cut the running costs for consumables tremendously.

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Improvements in Albras Bake Furnaces Packing and Unpacking System: *Paulo Douglas Vasconcelos*[1]; [1]Albras Aluminio Brasileiro, Carbon Eng. Grp., Rod. PA483, Km 21, Barcarena, Para 68447-000 Brasil

ALBRAS operates four ring-type bake furnaces. Each furnace is composed by sections made up of six cells separated by partitions flue walls through which the furnace is fired. The cell is about four meters deep and accommodates four layers of three anode blocks, around which petroleum coke is packed to avoid air oxidation and facilitate the heat transfer. Each furnace is serviced by three multipurpose overhead cranes of 15 t/h of coke suction capacity. This paper shows how the Carbon Plant Engineering Group has projected and developed an internal solution using a mathematical model to analyze and optimize the pneumatic conveyor phenomenon, breaking paradigms and increasing more than four times the packing coke suction capacity.

Cast Shop Technology: Melt Shop Operations

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

Tuesday PM — Room: Mississippi
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Julian V. Copenhaver, NSA Division of Southwire Company, Hawesville, KY 42348 USA

2:00 PM Introductory Remarks

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The Development of the Modern Dross Press: *Ophneil Henry Perry*[1]; [1]J. McIntyre Machinery, Sales, Unit G, Acorn Park Ind. Estate, Harrimans Ln., Dunkirk, Nottingham, Nottinghamshire NG36JR England

Many different technologies for recovering aluminium from dross have evolved over the years. This paper describes how the best aspects of the dross cooler, the dross press and the dross stirrer have been combined to maximize metal recovery. First Generation Machines Dross presses have been used since 1895. Dross is raked from the furnace into cast iron or steel pots and squeezed by a hydraulic ram. Early systems failed due to the problems associated with high temperature of dross and associated sticking. Steel castings tended to crack and break, and meltdowns within the system often occurred. Operating Experience with 1st Generation Machine J.McIntyre Aluminium Ltd is the UKs largest recycler of aluminium, producing 60,000 tons (132mbls) ingot per year. During 1995 we at J.McIntyre Aluminium Ltd started to use a dross press. Although we had some success with medium temperature dross, we experienced serious difficulties with hot or thermitting dross. Our conclusion was that the machine we were operating in 1995 was not much different to the original machine of 1895. From our initial trials, therefore, we had identified the following problems with the dross press: 1) Dross sticking to press head; 2) Short life of press head; 3) Press could not process hot (over 900°C) or thermitting dross; 4) Press could not process low temperature dross (below 675°C); 5) Inconsistent on-site metal recovery from press; 6) No top drain of skull; 7) Environmental problems; 8) Safety. Each of these problems will be addressed in the paper.

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Multi-Functional, Articulated Vehicle which can be Easily Adapted to the Specific Needs of a Cast Shop: *Serge Desgagne*[1]; Charles R. Emond[1]; Luc Boivin[2]; [1]Multi-Functional Vallee Inc., 310

Principale, St-Alban, Québec G0A 3B0 Canada; [2]Alcan Primary Metals, Alma Project, P.O. Box 1500, Jonquiere, Quebec G7S4LS Canada

The use of efficient, specialized mobile equipment, adapted for furnace maintenance and charging purposes, can have a major, positive impact on cast shop's operations. Forklift trucks cannot perform all the tasks required in a cast shop. Customized tools can economically ensure the safety of the furnace operators, the quality of the cast metal, improve productivity and refractory life, while reducing the number of vehicles required. This paper describes the advantages of a multi-functional articulated vehicle, adapted to the cast shops. A quick-action attaching mechanism connects semi-automatically onto a variety of modular tools which perform various tasks such as scrap charging, alloy material charging, stirring, skimming and cleaning the furnace. The tools are rigidly clamped to the vehicle and hydraulically activated for tilting, rotating and positioning. The operator can complete his work cycle without ever having to leave the vehicle. He has an unobstructed view at all times while enjoying the comfort of an air-conditioned cabin. Safety and ergonomics have played a major role in the design of the equipment.

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Protecting Refractories Against Corundum Growth in Aluminum Treatment Furnaces: *Claude Allaire*[1]; Mohamed Guermazi[1]; [1]Ecole Polytechnique of Montreal, CRIQ Campus, 8475 Christophe Colomb Rd., Montreal, Quebec H2M2N9 Canada

In aluminum treatment furnaces, corundum growth may take place at the metal line which promote the deterioration of their refractory sidewall. While conventional non-wetting additives may increase the resistance to corrosion of refractories below the metal line in such furnaces, their efficiency is significantly reduced at the high operating temperature conditions prevailing at their metal line. The purpose of this paper is to present a new additive which permit to protect refractories against the action of corundum growth in the above application.

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An Electrical Method to Monitor the State of an Inductor of a Channel Induction Furnace: *Wim Boender*[1]; John van Heeswijk[1]; Dirk Van Nieuwerburgh[2]; Jan Quisthoudt[2]; [1]Hoogovens Research and Development, P.O. Box 10000, 1970 CA IJmuiden The Netherlands; [2]Hoogovens Aluminum NV, A. Stocletlaan 87, Duffel B-2570 Belgium

In the cast house of Hoogovens Aluminium NV in Duffel, Belgium, nearly all melting furnaces are channel induction furnaces. A smooth operation of these furnaces is important. The inductors that are the heating elements of these furnaces must be extremely reliable and predictable. Hence inductors are monitored systematically. An inductor is an a.c. device for heating liquid metals. Electrically, it is similar to a short-circuited transformer. Its resistance and reactance depend on the resistances and reactances of its primary and secondary circuits. The secondary circuit's resistance and reactance are dependent on the shape and the size of the inductor channels. The changes of these channels due to wear or clogging can be assessed measuring the resistance and reactance of an operating inductor. A comparison with the regular mechanical measurements has shown that the electrical method determines an inductor's state with sufficient accuracy to monitor geometry changes. Therefore it has been adopted in daily operations.

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Remelting by Continuous Feeding of Rolled Scrap into a Melt: *Snorre Farner*[1]; Frede Frisvold[2]; Thorvald Abel Engh[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. and Electrochem., Trondheim N-7491 Norway; [2]Ålesund College, Ålesund N-6025 Norway

Metal losses during remelting is common when recycling aluminium. Reduction of these losses could give a substantial economic gain. Experiments with continuous feeding of aluminium plates into molten aluminium have been performed. A simple steady-state mathematical model has been developed that gives the temperature profile and the penetration depth into the melt as a function of the feeding velocity, superheat, and the heat-transfer coefficients from melt to solid and from a solidified shell to the plate. A criterion for shell formation is also formulated.The results can be applied to understand more complex systems whereshredded scrap is fed into molten aluminium. The model presented could be of direct interest when feeding rolled scrap into molten aluminium.

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A Kinetic Study on the Antimony Removal of Molten Aluminum by CaSi Powder Injection: *Alfredo Flores*[1]; Juan de Dios Castrejón[1]; [1]CINVESTAV, Unidad Saltillo, Carretera Saltillo Monterrey, Km. 13, Ramos Arizpe, Coahuila 25900 Mexico

The deleterious effect of antimony over the behaviour of sodium or strontium as modifiers of the AlSi eutectic phase has been addressed in different technical papers through the last few years. Nevertheless, there are not enough publications related to the development of processess to remove antimony from molten aluminum alloys. In this sense, this paper describes the experimental results obtained after the application of a technique based on the submerged powder injection of CaSi blends, to remove antimony from molten aluminum. Kinetic measurements have been performed as a function of powder flow rate-to-carrier gas flow rate ratio, temperature of the bath, and powder size. Efficiencies in the order of 65% were reached for the best combinbation of operating parameters, so it was possible to sustain that the method studied is adequated for reaching the objective proposed.

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Dissolution Mechanism for High Melting Point Transition Elements in Aluminum Melt: *Young E. Lee*[1]; Stephen L. Houser[1]; [1]Eramet Marietta Inc., State Route 7 South Riverview Dr., P.O. Box 299, Marietta, OH 45750 USA

Alloying transition elements such as Cr, Fe, and Mn in aluminum melt with a consistent performance is a challenge to cast shop operators because of their higher melting point and density. This study is to provide an understanding of the alloying process for Cr, Fe, Mn by examining their alloying performance and dissolution behaior. The alloying performance for Cr, Fe, and Mn in aluminum melt was determined by measuring the recovery of the alloying elements in aluminum melt and the dissolution behavior by examining the microstructures of the interrupted samples during the alloying pocess. It was observed that the time for a full recovery is shorter for Fe and Mn than for Cr in a similar aluminum melt stirring condition and that their recoveries are affected by the design of the alloying additives as well as by the hydrodynamic condition of aluminum melt. The variation of the microstructures with the processing time shows that the alloying additives for Cr, Fe, and Mn go through the dissolution sequence of i ncubation, exothermic reaction, and dispersion in aluminum melt but their dissolution kinetics are different from each other. The dissolution mechanisms are proposed for the alloying process of Cr, Fe, and Mn in aluminum melt.

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

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

Tuesday PM — Room: Canal A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: L. Kunz, Institute of Physics of Materials, Brno, Czech Republic; K. Sadananda, Naval Research Laboratory, Washington, DC 20375 USA

2:00 PM

Fatigue Crack Initiation in FCC Single Crystals: *Petr Lukas*[1]; Ludvik Kunz[1]; [1]Institute of Physics of Materials As CR, Brno, Czech Republic

Initiation of fatigue cracks both in model fcc single crystals and in fcc single crystals used in engineering practice will be discussed. The emphasis will be placed on the role of the cyclic slip localisation in cyclic plasticity and in crack initiation. It is shown that the formation of persistent slip bands (PSBs) is confined to single crystals of suitable crystallographic orientations and of sufficiently high stacking fault energy cycled at a relatively narrow range of stress and/or strain amplitudes with zero or near-zero mean stresses at not too a high temperature. For single crystals of not suitable orientations and/or low stacking fault energy cycled under stresses and/or strains outside the critical range and/or under high temperatures the cyclic plasticity manifests itself by other forms of slip activity leading to the formation of coarse surface hill-valley topography. Thus the cyclic slip localisation plays always the crucial role in the initiation of fatigue microcracks, but this slip localisation need not be due to the PSBs; the PSBs represents only one of the modes of cyclic slip localisation. The necessary prerequisites for the microcrack initiation are (i) expressive notch-peak topography, (ii) locally higher cyclic plastic strain at the intrusion root, and (iii) irreversible slip processes around the surface intrusions including hardening of material around the root of the surface intrusions. The existing models of crack initiation are accessed in the light of these prerequisites.

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The Influence of Heat Treatment and Solidification Rate on the Behavior of Small Fatigue Cracks in a Cast Aluminum Alloy: *Michael J. Caton*[1]; J. Wayne Jones[1]; John E. Allison[2]; [1]University of Michigan, Dept. of Matls. Sci. and Eng., 2300 Hayward, Ann Arbor, MI 48109-2136 USA; [2]Ford Motor Company, Matls. Sci. Dept., MD 3182/SRL, 20000 Rotunda Dr., Dearborn, MI 48121 USA

For a wide range of stress amplitudes, the fatigue life of cast aluminum specimens is dominated by propagation of small cracks which initiate predominantly from microshrinkage pores. Therefore, an understanding of the small crack behavior and knowledge of the pore size distribution enables the prediction of fatigue properties. A study of fatigue crack growth of small cracks (~20 μm to 2 mm) in cast W319-T7 Al, a commercial Al-Si-Cu alloy used in automotive castings, showed a significant small crack effect as well as an influence of solidification rate on propagation behavior. Faster crack growth occurs in more slowly solidified material, which possesses a lower yield strength. Interestingly, a previous study revealed that the S-N curves are very similar for the over-aged (T7) and peak-aged (T6) conditions of W319. These conditions exhibit considerably different yield strengths and nominally identical pore size distribution. This suggests similar crack growth rates despite the differences in strength. A comparison of small crack growth data for the T6 and T7 conditions will be presented. Crack growth correlating parameters and the role of yield strength will be discussed.

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On Striations and Fatigue Crack Growth in 1018 Steel: *H. Cai*[1]; Arthur McEvily[2]; [1]Motorola Corporation, Austin, TX USA; [2]University of Connecticut, Dept. of Metallu. and Matls. Eng., Storrs, CT USA

A study of the fractographic features developed during fatigue crack growth in a 1018 steel has been carried out. This study included the use of stereographic techniques in a detailed examination of the geometry of striations formed at various ΔK levels. The results are compared with a number of proposed models for striation formation. The relation between the rate of fatigue crack growth in 1018 steel and the spacing of striations is also discussed.

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Initiation and Propagation of Stage-I Cracks in Copper Single Crystals under Load Control: *Ahsan Jameel*[1]; Pedro Peralta[2]; Campbell Laird[1]; [1]University of Pennsylvania, Dept. of MSE, Philadelphia, PA 19104 USA; [2]Arizona State University, Dept. of Mech. and Aerospace Eng., Tempe, AZ 85287-6106 USA

The initiation and propagation of stage-I cracks determines the magnitude of the life in copper single crystals as the transition to stage-II behavior heralds the advent of fracture. The behavior of initiation and propagation of stage-I cracks under strain control is well understood. However, the PSB behavior and consequently the behavior of the stage-I cracks under load control is significantly different and requires an understanding before a unified methodology for predicting fatigue lives of copper single crystals based on the physics of the kinetics of stage-I crack growth can be developed. Stage-I cracks are found to nucleate in PSB's under load control. However, unlike in strain control, these cracks are almost exclusively found in micro PSB's. The cracks then grow along the PSB's with the occasional crack "skipping" on a secondary slip system to an adjacent PSB. These cracks also show a tendency, similar to that in strain control, to "rob" adjacent PSB's of strain and halt the growth of cracks in those PSB's. The extent of this "strain robbing" region under load control is not known. The growth kinetics of the population of the cracks can be modeled as a function of the cumulative strain and leads to a Coffin Manson equation for predicting the fatigue life. It is seen the this Coffin Manson equation provides a lower bound for the fatigue life of the copper single crystals.

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Mixed-Mode Thresholds in High-Cycle Fatigue: *J. P. Campbell*[1]; R. O. Ritchie[1]; [1]University of California, Dept. of Matls. Sci. and Mineral Eng., Berkeley, CA 94720 USA

High-cycle fatigue (HCF) is a prime cause of military aircraft turbine engine failures. It results from fatigue-crack growth in blades and disks, initiated at small defects often associated with fretting or foreign object damage. Due to the high frequencies (>1kHz) involved, design must be based on a HCF threshold, such that crack propagation cannot occur within ~10^9 cycles. In this work, we examine the nature of the fatigue threshold, under representative high frequency and high load-ratio conditions, in a Ti-6Al-4V blade alloy with bimodal and lamellar microstructures, with emphasis on behavior under mixed-mode (modes I + II) versus mode I conditions. It is shown that whereas the mode I threshold is decreased with increasing mode-mixity (at $\Delta K_{II}/\Delta K_I$ ratios from 0 to 2, at load ratios from 0.1 to 0.8), in the finer-scale bimodal microstructure, provided the driving force is computed in terms of ΔG by summing up the contributions in both mode I

and II, the ΔG threshold is actually increased with mode-mixity, such that the mode I value is the worst case. In contrast, preferred crack paths in the coarser lamellar microstructures do lead to reduced ΔG_{TH} thresholds under mixed-mode conditions.

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Unified Approach to Fatigue Crack Nucleation and Growth: *K. Sadananda*[1]; A. K. Vasudevan[2]; [1]Naval Research Laboratory, Code 6323, Washington, DC 20375 USA; [2]Office of Naval Research, Arlington, VA 22217 USA

Unified approach to fatigue crack growth has been developed by the authors using a two parameter approach. It is shown that crack growth involves two thresholds, one in terms of delta K and other in terms of Kmax. Both thresholds need to be satisfied simultaneously although one or the other is the controlling parameter for a given range of load ratio R. It is shown that crack closure is neither necessary or sufficient to account for fatigue crack growth. The anomalous behavior of short cracks and the acceleration and retardation effects under under-loads and overloads have been successfully accounted using this two parameter approach. Using the Kitagawa diagram, it is shown that the concepts of fatigue crack nucleation and growth can be combined to provide a unified frame work that is self-consistent and physically meaningful. Role of internal stressed in crack nucleation, short cracks and overloads are discussed in the frame work of the unified approach.

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Effects of an Oxide Layer on Fatigue Crack Initiation in Iron and a High Strength Low Alloy Steel: *Shrikant P. Bhat*[1]; Morris E. Fine[2]; [1]Ispat Inland Inc., R&D Dept., 3001 E. Columbus Dr., East Chicago, IN 46312 USA; [2]Northwestern University, Dept. of Matls. Sci., 2225 N. Campus Dr., Evanston, IL 60208 USA

While it is well recognized that fatigue cracks initiate at the free surface, most of the mechanistic studies are conducted on carefully prepared, highly polished specimens. In contrast, in most commercial applications that utilize hot rolled steels, the oxide layer formed during the manufacture of the steel remains intact through the in-service life of the component. Through a series of graduate research projects, the Inland-Northwestern University cooperative research program examined the effects of a thermally grown wustite layer of controlled thickness on fatigue crack initiation. In this paper, fatigue crack initiation with and without the wustite layer is compared and modeled. In both commercially pure iron and an 80 ksi HSLA steel, the surface oxide layer drastically changes the crack morphology. A polished surface results in multitude of intrusions and extrusions at low strain amplitudes. In contrast, in the presence of a wustite layer, fewer surface cracks were seen. They appeared earlier in life, mainly occurring along the grain boundaries. Modeling of the crack formation in this paper is based on the accumulation of dislocations at the interface leading to the formation of crack surfaces at a critical dislocation density. The influence of grain size and substrate yield strength on fatigue crack initiation in the presence of an oxide layer will also be discussed.

High Resolution Electron Microscopy in Materials Science: Defect Structures

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

Tuesday PM — Room: Canal D
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Diane E. Albert, Los Alamos National Laboratory, Matls. Sci. and Tech., Los Alamos, NM 87545 USA

2:00 PM
25 Years of HREM in Materials Research: From Phase Transformations to Interface Reactions: *Robert Sinclair*[1]; [1]Stanford University, Dept. of Matls. Sci. and Eng., Stanford, CA 94305-2205 USA

Twenty-five years or so ago, we were demonstrating the applicability of lattice fringe imaging, at 0.2nm line resolution, to revealing details of phase transformation processes. As microscope resolutions gradually improved and cross-section specimen preparation methods became routine, structure imaging was successfully applied to semiconductor interfaces and eventually to defects in close-packed metals and ceramics. Accordingly high-resolution electron microscopy (HREM) became one of the most important investigative techniques in materials research. Our own work followed the path of studying reactions at interfaces, including of course development of in situ HREM. Following a brief historical overview, our current understanding of interfacial reactions will be presented, drawing on recent examples of direct contemporary technological importance (e.g. graphitization of computer hard disk overcoats, amorphization at Ta-Cu metallization interfaces etc.).

2:30 PM Invited
HREM Characterization of Slip Transmission in Lamellar TiAl: *Hamish Fraser*[1]; Jörg M.K. Wiezorek[2]; Michael Mills[1]; [1]The Ohio State University, Matls. Sci. and Eng., Columbus, OH 43210 USA; [2]University of Pittsburgh, Dept. of Matls. Sci. and Eng., Pittsburgh, PA USA

Alloys based on the intermetallic compound TiAl can be heat-treated to yield various forms of microstructure, among which the lamellar version offers advantages in terms of optimizing strength. This optimization is associated with the scale of the microstructure that can be effected by heat-treatment. While strength may be optimized, room temperature ductility usually remains at quite low values (<2%). Most useful compositions of lamellar TiAl involve (at least) two phase microstructures of TiAl and Ti3Al. Characterization of deformed samples yields the result that in the main the TiAl lamellae deform readily by twinning and slip, there is little evidence for extensive deformation in lamellae of Ti3Al. This study is aimed at developing an understanding of the deformation behavior in lamellar Ti3Al, focusing on the transmission of slip from adjacent lamellae of TiAl and the factors affecting the activation of dislocations whose Burgers vectors with components perpendicular to the basal plane of this compound (i.e.,[c]-component dislocations). It has been found that transmission of slip as well as stress induced activation of dislocations occurs but that generally motion of [c]-component dislocations is a very difficult. HREM studies have shown that the cores of these dislo-

cations are spread fairly significantly out of their slip planes and this presumably accounts for the lack of mobility. This work has been supported by the National Science Foundation, with Dr. Bruce MacDonald as Program Manager.

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Fine Structure of Dislocations and Deformation Behavior of Intermetallic Compounds: *Michael J. Mills*[1]; [1]The Ohio State University, Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43221 USA

Ordered intermetallic compounds are of considerable technological and scientific interest due to their potential as structural materials for high temperature applications. In many cases, the attractive strength at high temperatures and unique flow properties of intermetallics are linked directly to their complex crystal structures (as compared with simple metals) which affect both the core structure and dissociation of dislocations. This presentation will focus on developments in our understanding of plastic flow in several ordered intermetallics, emphasizing the connection between the fine structure of dislocations and macroscopic mechanical properties. The results of experimental investigations using high resolution TEM techniques will be described, as will the direct comparison of these fine structures with atomistic and continuum modeling approaches. The insight that these comparisons provide with respect to the unique flow properties in these alloys will also be presented. Specific examples to be discussed include the anomalous flow strength behavior observed in Ni_3Al, the anisotropic flow properties of NiAl and the deformation of gamma-TiAl at higher temperatures.

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Comparisons of HREM Observations and Theoretical Analysis Based on Ab Initio Calculations of Dislocation Core Structures: *Kevin J. Hemker*[1]; T. John Balk[1]; Oleg N. Mryasov[2]; Yuri N. Gornostyrev[3]; Arthur J. Freeman[2]; [1]Johns Hopkins University, Dept. of Mech. Eng., 122 Latrobe Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Northwestern University, Dept. of Phys. and Astro., Rm. F275, 2145 No. Sheridan Rd., Evanston, IL 60208-3112 USA; [3]Institute of Metal Physics, 18 S. Kovalevskaya St., Ekaterinburg 620219 Russia

Dislocation core structures control the mobility of dislocations in a large number of metals and alloys and play an important role in determining their mechanical behavior. We have implemented a combined experimental and theoretical approach to compare and contrast the dislocation core structures in three different sets of metals and alloys: fcc Au-Ir, L12 Ni3Ge-Fe3Ge and L10 TiAl-CuAu. HREM has been utilized to experimentally observe the arrangement of atomic columns surrounding the dislocation cores, allowing for the characterization of planar and non-planar core dissociations. Theoretical analysis based on the 2D-Peierls-Nabarro model and ab initio calculations have been used to predict dislocation core spreading and splitting and to derive their effects on dislocation mobility. The theoretical predictions also provide atomic level models of the dislocation cores that can be used as inputs for HREM image simulations. Direct comparisons of simulated and experimentally obtained HREM images will be presented and used to characterize the dislocation core structures and to provide benchmarks for the ab initio calculations. This work was supported by the Air Force Office of Scientific Research, Grants No. F49620-98-1-0208 and F49620-98-1-0321.

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Structure and Dynamics of Grain Boundary Defects: *Douglas L. Medlin*[1]; [1]Sandia National Laboratories, Thin Film and Interface Sci. Dept., 7011 East Ave., Livermore, CA 94551 USA

Interfacial defects such as dislocations and steps play important roles in determining the structure and behavior of grain boundaries. Though a long standing issue in materials science, over the last decade the atomic scale information provided by high resolution transmission electron microscopy (HRTEM) has significantly improved our understanding of such defects. After reviewing some of these key advances, I will discuss our work on the structure and behavior of interfacial dislocations present at Σ=3 {112} and {111} interfaces in FCC metals. Two classes of interfacial dislocations play a role in this system: Dislocations with Burgers vector a/6<112> and with Burgers vector a/3<111>. Such dislocations originate as a means of accommodating deviations from ideal lattice coincidence or through lattice dislocation decomposition reactions. Even this relatively simple system yields a rich variety of phenomena that can be directly understood and predicted from the interfacial dislocation structure. In particular, I will concentrate on two examples: (1) glide and climb processes of a/3<111> dislocations leading to interfacial sliding and twin growth and (2) shear processes of a/6<112> dislocations and their impact on interfacial dissociation. Throughout, I emphasize the necessarily close coupling between computation and experiment, with HRTEM observations of defect configurations motivating atomistic and continuum calculations and the resulting calculations providing insight critical to interpreting the observations. This work is supported by the U.S. Department of Energy, in part by the Office of Basic Energy Sciences, Division of Materials Science, under contract DE-AC04-94AL85000.

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HREM of Crystals with Unusual Internal Lattice Bending Formed as a Result of Amorphous-Crystalline Transformations in Thin Films: *Vladimir Kolosov*[1]; A. R. Thölén[2]; [1]Ural State Economic University, Eng. Dept., 8-th March 62, GSP-985, Ekaterinburg 620219 Russia; [2]Chalmers University of Technology, Dept. of Exp. Phys., Göteborg 41296 Sweden

Unusual microstructures formed during amorphous-crystalline transformation were revealed earlier by TEM studies in some thin films [1]. This new phenomenon resides in strong (up to 120°per 1 mm of crystal length) regular, dislocation independent internal bending of the crystal lattice planes (IBCLP) in a growing crystal (realized around an axis lying in the film plane). In this paper we present HREM of some structures, demonstrating the influence of film thickness, composition and lattice orientation on the IBCLP phenomenon. Crystallization was usually initiated by electron beam inside TEM and traced in situ. HREM was performed in a plane view of the samples with a CM200FEG and a slowscan CCD camera and analyzed with the Digital Micrograph software. Bend contour technique was used to evaluate lattice orientation (in combination with selected area diffraction), internal lattice bending and crystal thickness. EDX, EELS and CBED were also used. The most general result: HREM displayed neither any lattice imperfections nor inclusions of amorphous material which could initiate and keep going the IBCLP phenomenon. For the crystallisation front moving to the thinner part of amorphous antimony selenide film evaporated with a strong thickness gradient (thickness range 100¸15 nm) lattice bending is strongly increasing. At the same time HREM reveals a steady mean interplanar spacing while lattice fringes lose the contrast and become less and less discernible as the film gets thinner. The magnitude of internal bending in crystals of hexagonal Se growing (0.5-1 mm/s) in Se-Te films with a gradient in concentration of elements (in the range 5-20% Te) is built up upon the increase of Te content in amorphous film, whereas mean interplanar spacings are the same for the samples with minimal and maximal Te content. It is rather surprising fact since the lattice constants along [001] for Se (0.496 nm) and for Te (0.593 nm) differ significantly. Crystallisation of amorphous iron oxide films is usually realised by formation of alternating circular zones of two kinds which differs in lattice orientation, imperfection and growth rate [2]: the crystal centre (initial nucleus) and the central areas of odd zones with [001] normal to the film plane are highly perfect single crystalline areas growing with the maximal rates, the central areas of even zones (where [001] tends to orient radially in the film plane) are most imperfect and are growing most slowly. At the grain boundaries which are most frequent at the latter areas no misfit or dislocation-like contrast has been observed. In some of the places examined in more detail lattice fringes are passing continuously from one grain to the other and with no very sharp offset at the boundary, corresponding to tilting boundaries. The observed regularities are explained by combination of IBCLP and crystal anisotropy. The interface between amorphous material and crystallized region (analysed in dynamics on the photos made by through-focus series) is rather sharp, with the transition region about 1 nm characterised by intermediate atom ordering and a change of diffraction contrast. HREM of CuSe whiskers with IBCLP grown in amorphous films gives direct evidence of the regular curvature of lattice

planes. References: 1. Kolosov V. Yu., Proc. XII ICEM, Seattle, San Francisco Press, v. 1, 574 (1990). 2. Kolosov V. Yu. And Thölén A.R., NanoStructured Materials, 9, 323 (1997). Partial support of RSAS (grant 1557) and RFBR (grant 97-02-17784) is gratefully acknowledged.

Honorary Symposium for Professor Oleg D. Sherby: Creep Mechanisms and Behavior 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

Tuesday PM — Room: Bayou E
March 14, 2000 — Location: Opryland Convention Center

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

2:00 PM Invited
Creep of Oxide-Dispersion-Strengthened Ferritic Steels: *Brian Wilshire*[1]; [1]University of Wales Swansea, Matls. Eng., Singleton Park, Swansea, Wales SA28PP UK

It has recently been shown that, during creep of oxide-dispersion-strengthened (ODS) alloys, the principal role of the dispersoid particles is to stabilize the retained dislocation substructures present after thermo-mechanical processing. Linking the creep resistance of ODS alloys to their retained dislocation densities then defines three categories of stress/creep rate behaviour, depending on grain size and shape. (i) Category A characteristics are found for alloys with equiaxed grain structures and low retained dislocation densities. (ii) Category B behaviour is observed with polycrystals having high grain-aspect-ratios (GAR), relatively fine grain sizes and high retained dislocation densities. (iii) Category C properties are displayed by ODS single crystals and by coarse grain high-GAR polycrystals, both having high retained dislocation densities. The validity of this classification system is illustrated by reference to the high-temperature creep properties recorded for three types of ODS ferritic steel.

2:20 PM Invited
Critical Assessment of Creep Extrapolation Using OSD and BAS Parameter Methods: *Yoshio Monma*[1]; [1]Kochi University of Technology, Dept. of Environ. Sys. Eng., Tosayamada-cho, Kami-gun, Kochi 782-8502 Japan

Among various TTP (Time-Temperature Parameter) methods proposed for the extrapolation of creep-rupture data, the merit of OSD (Orr-Sherby-Dorn) and BAS (Barrett-Ardell-Sherby) parametric procedures are not recognized widely as compared with the LM (Larson-Miller). Because of the availability of the long-term stress rupture data from the NRIM Creep Data Sheet program, which includes many data points beyond 100,000h for various heat-resisting steels and alloys, we can directly validate the ability of fitting and extrapolation by TTP methods. This paper is to examine the accuracy of extrapolation OSD and BAS methods using computerized data evaluation system. We consider the treatment of the heat-to-heat variation, how to incorporate the microstructural changes during long-time creep in TTP analysis.

2:40 PM
The Effect of Particle Reinforcement on the Creep Behavior of Aluminum Alloys: *Paul E. Krajewski*[1]; [1]General Motors, Rsrch. and Dev. Ctr., MC-480-106-212, 30500 Mound, Warren, MI 48090 USA

The effect of particle reinforcement on the creep behavior of aluminum alloys will be reviewed. The direct and indirect strengthening effects of particle reinforcement are examined in pure, solid solution strengthened, and precipitation strengthened aluminum alloys. In pure aluminum, where strengthening is controlled by dislocation substructure, indirect composite strengthening controls creep behavior. In solid solution strengthened alloys, where a dislocation substructure does not develop, direct strengthening dominates creep behavior and strengthening can be predicted by continuum models. Finally, in precipitation strengthened alloys, indirect strengthening dominates creep behavior. The effect of the particle reinforcements on precipitate distribution and stability can actually lead to composite weakening of aluminum alloys during creep deformation. Ideas for designing a creep resistant aluminum composite will be suggested.

3:00 PM Invited
Tensile Behavior of Open Cellular 7075-T6 Al at Ambient and Intermediate Temperatures: *James C. Earthman*[1]; [1]University of California, Irvine, Dept. of Chem. Eng. and Matls. Sci., Irvine, CA 92697-2575 USA

Abstract text is not available

3:20 PM Break

3:40 PM Invited
Powder Densification Maps and Applications in Selective Laser Sintering Post Processing: *David L. Bourell*[1]; [1]University of Texas at Austin, Texas Matls. Instit., Mech. Eng., MC C22000, Austin, TX 78712 USA

Selective Laser Sintering (SLS) is a Solid Freeform Fabrication process in which a part is built quickly from powders without the use of part-specific tooling. Production of metallic, ceramic and composite parts often requires some form of post-process sintering to achieve full density. Powder Densification Maps represent a potent tool for optimizing the post-processing parameters to achieve full density parts. Such maps are computational representations of part density as affected by time, temperature, pressure and materials properties. Critical to the formulation of densification maps is an understanding of time-dependent plasticity. This presentation will summarize SLS developments at The University of Texas at Austin with emphasis on the utility of powder densification mapping of part post-processing.

4:00 PM Invited
Enhanced Densification of Titanium Powders by Cyclic Transformations under Stress: *David C. Dunand*[1]; Christopher Schuh[1]; Philippe A. Noel[2]; [1]Northwestern University, Dept. of Matls. Sci. and Eng., Evanston, IL 60208 USA; [2]Matra Defense, STTN, 20-22 Rue Grange Dame Rose, Velizy 78141 France

The densification of titanium powders is investigated in uniaxial die pressing experiments at 980°C (in the beta-field of titanium), and compared with densification during thermal cycling between 860 and 980°C (about the alpha-beta phase transformation of titanium). In agreement with the 1982 paper by Ruano, Wadsworth and Sherby on white cast iron powders, we observe that thermal cycling enhances densification kinetics of titanium powders through the emergence of transformation-mismatch plasticity (the mechanism responsible for transformation superplasticity) as a densification mechanism. The isothermal hot-pressing data compare favorably with existing models of powder densification, and these models are successfully adapted to the case of transformation-mismatch plasticity during thermal cycling.

4:20 PM Invited
Strain Rate Sensitivity and Creep Behavior of Some Nickel-Based Intermetallic: *Shu-En Hsu*[1]; [1]Hsu-Yang Technologies Company Limited, 80 Erh-Chia Rd., Ying-Keh, Taipei-Hsian, Taiwan

In response to the influence of Prof. Oleg D. Sherby's pioneering contributions to the field of high-temperature mechanical behavior, strain-rate effects and creep behavior of NiAl and TiNi intermetallics have been investigated. NiAl intermetallics were once recognized as

ideal aerospace materials for key components in turbo-engines at elevated temperature. However, after many years of study, the outcome was not so successful when strain-rate sensitivity was evaluated. This report reviews the results of the strain-rate study of polycrystalline and single-crystal NiAl-intermetallics. Experimental results show that both the stress exponent and the activation energy for creep increase significantly. However, a decrease of the coefficient of strain-rate sensitivity resulted in reduction of ductility and toughness. In fact, this was a common trend for many other structural intermetallics, such as TiAl or FeAl. As a result, except for some effective toughening mechanisms that can be applied to overcome strain-rate sensitivity, the hot-shortness of these intermetallics prevented their application to crucial components in aero-engines at elevated temperature. On the other hand, unlike most of the structural intermetallics, TiNi has exceptional inherent ductility. Besides the interesting phenomena of shape memory, high damping, and pseudo-elasticity due to martensitic transformation, the co-existence of superplasticity and super-elasticity in the same material at different temperatures will attract more attention among the modern advanced materials. This report will review the strain-rate sensitivity of Cu and V-Co modified TiNi-SMA. It was found that the coefficient of strain-rate sensitivity increased as elastic modulus decreased. Modulus change was due to the stress induced martensitic transformation at low temperature, below Ms. A pseudo-elastic (Super-elastic) material can be obtained by proper control of the martensitic transformation. Alternatively, Cu-modified TiNi-SMA exhibited superplasticity at high temperature, well above Af, where the damping capacity was high since the modulus was very low. The presence of this dual behavior of super-elasticity and superplasticity is technologically significant for manufacturing engineering. Several illustrations of applications for TiNi-based intermetallics used in sporting goods will be presented in this report.

4:40 PM Invited
Singularity of Creep Strength in Ni_3Al at Stoichiometry: Seiji Miura[1]; Yoshinao Mishima[2]; *Tomoo Suzuki*[1]; [1]Hokkaido University, Div. of Matls. Sci. and Eng., Kita-13 Nishi-8, Kita-ku, Sapporo 060-8628 Japan; [1]Kochi University of Technology, Faculty of Tech., Tosayamada-cho, Kochi 782-8520 Japan; [2]Tokyo Institute of Technology, Dept. of Matls. Sci. and Eng., 4259 Nagatsuta, Midori-ku, Yokohama 226-8502 Japan

Systematic investigations on both poly- and single-crystalline Ni_3Al have shown the existence of singularity at stoichiometry for creep strength. The singularity had previously been observed for the apparent activation energy for the stress anomaly of the octahedral slip at intermediate temperatures as well as for the athermal stress of the cube slip. We have revealed that the viscous motion of dislocations on (111) plane governs the creep deformation of Ni_3Al at temperatures higher than the peak temperature, and the mechanism controlling the stress anomaly persists in its effect on the dislocation motion even at such high temperatures. Large number of studies have been attempted to explain the stress anomaly with the formation of cross-slipped part of screw dislocation, well-known as the Kear and Wilsdorf (K-W) mechanism, as a basis. This cross-slipped parts which have a double-kink type configuration can be expected to move with a dragging mechanism controlled by diffusivity of vacancy in the high temperature range. The double-kink type locking parts are composed of a pair of jogs, one of which acts as a vacancy source and the other as a vacancy sink for the climb motion. The time for vacancy migration from ones to the others governs the velocity of dislocations. By taking the effect of Al concentration on inter-diffusion coefficient of Ni_3Al, both the singularity at stoichiometry and the effect of off-stoichiometry on creep behavior could be explained.

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

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

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

Tuesday PM — Room: Johnson A/B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Toru Miyazaki, Nagoya Institute of Technology, Matls. Sci. and Eng., Nagoya 466-8555 Japan; Long-Qing Chen, Penn State University, Matls. Sci. and Eng., University Park, PA 16802 USA

2:00 PM Invited
Multimillion Atom Simulations of Nanostructured Materials on Parallel Computers-Sintering and Consolidation, Fracture, and Oxidation: *Priya Vashishta*[1]; Martina E. Bachlechner[1]; Timothy Campbell[1]; Rajiv K. Kalia[1]; Hideaki Kikuchi[1]; Sanjay Kodiyalam[1]; Aiichiro Nakano[1]; Phillip Walsh[1]; [1]Louisiana State University, Concurrent Comp. Lab. for Matls. Sims., Depts. of Phys. and Comp. Sci., Baton Rouge, LA 70803-4001 USA

Multiresolution molecular-dynamics (MRMD) approach for multimillion atom simulations has been used to investigate mechanical failure in crystalline ceramics, nanophase ceramics, and at interfaces. Structural correlations determined by neutron scattering experiments are used to validate the interatomic potentials used in the simulations. Crack propagation and fracture are studied and the morphology of fracture surfaces is examined. Mechanical failure at semiconductor/ceramic interface is studied by applying tensile strain parallel to the interface. Multimillion atom simulations of oxidation of aluminum nanoclusters and nanoindentation simulation at room temperature and at elevated temperature will be reported along with local stress and temperature distributions. Research supported by the US DOE, NSF, AFOSR, ARO, USC-LSU MURI (DARPA & AFOSR), Austrian FWF, and PRF.

2:30 PM Invited
A Defect Model for Twinning in Ferroelectrics: *David J. Srolovitz*[1]; N. Sridhar[2]; Jeffrey Rickman[3]; [1]Princeton University, Princeton Matls. Instit., Bowen Hall, 70 Prospect Ave., Princeton, NJ 08540 USA; [2]Rockwell Science Center, Thousand Oaks, CA 91360 USA; [3]Lehigh University, Matls. Sci. & Eng., Bethlehem, PA 18015 USA

We examine electrostatic and elastic contributions to the twinning of a thin ferroelectric film. The analysis employed focuses on the multipole character of the imperfections associated with a twin, treating electrostatic and elastic defects within a unified framework. Our analytical results are complemented by simplified descriptions of the twinned system in terms of idealized defects, leading to an intuitive understanding of defect energetics. For completeness, a number of different twin geometries are examined.

3:00 PM Invited
Phase Transitions in Metallic Multilayers: *Hamish L. Fraser*[1]; Rajarshi Banerjee[1]; Suliman A. Dregia[1]; [1]The Ohio State University, Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Dimensionally induced phase transitions in thin films and multilayers is of considerable interest because it lends the ability to tailor material

properties by engineering their structure at the nanoscale. Recently, a series of interesting phase transitions have been reported in Ti/Al multilayers wherein Ti transforms from its bulk stable hcp structure to an fcc structure at small layer thicknesses (~ 5nm) and Al transforms from fcc to hcp at ~ 2.5 nm. These reported structural transitions were based on transmission electron microscopy (TEM) studies on sputter deposited Ti/Al multilayered specimens thinned in the cross-section geometry. Subsequently, a classical thermodynamic model has been proposed for explaining phase stability in multilayered materials as a function of the composition and bilayer thickness. Using this model it has been possible to rationalize the effect of cross-section thinning on the phase stability in terms of the anisotropic ingress of hydrogen into the Ti/Al multilayers. The model presents an elegant framework for rationalizing the effect of alloying/impurity additions on the phase stability. Furthermore, the scope of model has been expanded by considering its applicability to other systems including non close-packed ones such as Co/Cr multilayers. Recent experiments have explored in detail the formation of fcc Ti and the coherent to incoherent transition in Ti/Al multilayers as a function of composition and bilayer thickness. In addition, the initial thermodynamic model has been modified to account for the effect of coherency strains on the phase stability. These results will be discussed in detail and a new phase stability diagram will be proposed for Ti/Al multilayers.

3:30 PM Break

3:45 PM Invited

Constrained Phase Transformations: Thermodynamics and Microstructure: *Alexander L. Roytburd*[1]; [1]University of Maryland, Matls. and Nuc. Eng., College Park, MD 20742 USA

Thermodynamics of constrained phase transformations is discussed. The concept of elastic domains is used for description of polydomain microstructures formed under 3D-, 2D- and 1D- constraint. As examples phase transformations in composites and epitaxial films are discussed, as well as self-constrained coherent phase transformations. Dependence of phase diagrams on constraint conditions is demonstrated.

4:15 PM Invited

Late-Stage Phase Separation in Elastically Stressed Solids: K. Thornton[1]; N. Akaiwa[2]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [2]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba 305-0047 Japan

A major challenge in predicting the late-stage evolution of microstructures in two-phase solids is that the difference in lattice parameters between the particle and matrix phases engenders a long-ranged elastic stress field. Unlike stress-free systems, where the evolution of the microstructure is driven by a decrease in the total interfacial energy of the two-phase mixture, the ripening process in these materials is driven by a decrease in the sum of the elastic and interfacial energies. It is thus not clear if the classical results of interfacial energy driven ripening hold true in elastically stressed solids. Due to the strong shape dependence of the interparticle elastic interactions this issue can only be addressed using large-scale numerical calculations. Through the use of boundary integrals and the fast multipole method, we have examined the morphological evolution of many thousands of elastically and diffusionally interacting particles. We shall discuss the temporal evolution of the statistically averaged properties of these coarsening ensembles, such as the exponent of the temporal power law for the average particle size, the dependence of the rate constant on volume fraction, and the evolution of the spatial correlations between particles.

4:45 PM Invited

Elastic Misfit Interactions at Phase Transitions in Solids: *Akira Onuki*[1]; [1]Kyoto University, Dept. of Phys., Kyoto 606-01 Japan

I will discuss effects of elastic misfit interactions in phase separation and structural phase transitions and present some new computational results in 2D and 3D. I am particularly interested in (i) slowing down of phase separation at high volume fractions with elastic misfit, (ii) critical behavior of binary solids influenced by elastic misfit (where even thermal fluctuations can be elastically affected), and (iii) structural intermediate states realized by a dilation-adjustment mechanism recently proposed by the present author.

International Symposium on Global Innovations in Materials Processing and Manufacturing: Direct Fabrication and Metal Powders

Sponsored by: Materials Processing and Manufacturing Division,

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

Tuesday PM — Room: Canal E
March 14, 2000 — Location: Opryland Convention Center

Session Chair: Iver E. Anderson, Iowa State University, Ames National Lab., Ames, IA 50011 USA

2:00 PM

Fabrication of Designed Materials: *Debasisch Dutta*[1]; Amit K. Ghosh[2]; Noboru Kikuchi[1]; Jyoti Mazumder[1]; [1]University of Michigan, Dept. of Mech. Eng. and Appl. Mech., Ann Arbor, MI 48109 USA; [2]University of Michigan, Dept. of Matl. Sci. and Eng., Ann Arbor, MI 48109 USA

We describe a new technology for design, representation, and fabrication of graded materials; and components. It involves the computer integration of three core technologies, that of homogenization design method, heterogeneous solid modeling, and direct material deposition. We show the overall system framework and describe each core technique in detail. Examples of design and fabrication of a materials system with desired properties using proposed methodology are presented in the paper.

2:30 PM

The Development of Residual Stress in Laser Engineered Net Shaping (LENS) Fabrication: *Michelle Griffith*[1]; Drew Nelson[2]; Mark Ensz[1]; Daryl Reckaway[1]; Donald Greene[1]; Michael Oliver[1]; [1]Sandia National Laboratory, Albuquerque, NM USA; [2]Stanford University, Stanford, CA USA

In direct laser metal deposition technologies, such as the LENS process, it is important to understand and control the emergence of residual stresses during fabrication. In essence, the thermal behavior and its transients during fabrication must be monitored and, hopefully, controlled to reduce residual stresses to usable levels. This talk will describe techniques to monitor the thermal signature and history during LENS processing. Methods to measure the residual stress in LENS components will be described, and correlation between thermal history, microstructural evolution, and resulting stresses will be discussed. Development of an understanding of residual stress manifestation and ways to reduce residual stress will be discussed.

2:50 PM

Engineering the Microstructure and Properties in 316 SS and Composite Materials: *John E. Smugeresky*[1]; D. M. Keicher[2]; J. A. Phillibert[1]; J. Anthony Romero[3]; W. H. Hofmeister[4]; [1]Sandia National Laboratory, Livermore, CA 94551-0969 USA; [2]Optomec Design Company, Albuquerque, NM USA; [3]Sandia National Laboratory, Albuquerque, NM USA; [4]Vanderbilt University, Nashville, TN USA

Using the Laser Engineered Net Shaping (LENS™) process, we have evaluated the process parameter space for making useful fully dense metal shapes with monolithic, composite, and functionally graded microstructures. Samples of 316SS with, and without fine tungsten carbide particles, have been engineered to obtain a range of properties dependent on process parameters and composition. By varying only the process conditions, it is possible to obtain a range of tensile yield strengths between two to three times that of conventionally processed annealed material with little change in ductility. At double the yield strength of annealed material there is no loss in ductility. The results of our microstructure analysis will be discussed to show how both grain refinement and dislocation density affect the mechanical properties. Work supported by the U. S. Department of Energy under contract DE-AC04-94AL85000.

3:10 PM

Variable Composition Laser Cladding and Its Application in Laser Direct Casting: *Ken Watkins*[1]; Matt Murphy[1]; [1]University of Liverpool, Dept. of Eng., Laser Eng. Grp., Brownlow St., Liverpool L693BX UK

Variable composition laser cladding has been demonstrated at Liverpool by employing a multiple powder hopper system under computer control. It has been found that compositional control and microstructural variation in tertiary alloy systems can be achieved by this means. At the same time, it has been shown that laser direct build of components from CAD data can be achieved by adapting the laser cladding process to the incremental addition building of components (Laser Direct Casting). This is done using a unique six-axis manipulator that maintains a null point beneath the stationary laser at all times during the build procedure. The objective of this paper is to review these developments and to suggest how the bringing together of these two approaches can be utilized in the production of near net shape components with novel features such as variable composition layers, composite (reinforced) microstructures and SMART component functionality.

3:30 PM Break

3:50 PM

Materials Research in the IRC Relevant to Tooling Produced by Rapid Prototyping and by Direct Laser Fabrication: T. Sercombe[1]; X. Wu[1]; I. T.H. Chang[1]; *M. H. Loretto*[1]; [1]University of Birmingham, Interdis. Rsch. Ctr., Edgbaston B152TT UK

The Interdisciplinary Research Centre (IRC) has a major activity in the area of Net Shape Manufacturing with the recent opening of a new laboratory fitted out with over £3.5M of processing equipment. Among the equipment is a sinterstation with a laser upgraded to 100W and a direct laser fabrication facility with a 1.4kW laser. Programmes are now underway aimed at producing tooling by both of these techniques which aims to produce long-life, fully dense tooling for a number of tool steels for different applications. Preliminary work has suggested that the approach which is being used has promise and progress in these fields and in other closely linked areas of research will be discussed.

4:20 PM

Advanced Mold Design and Construction: *Paul F. Jacobs*[1]; [1]Express Tool, Warwick, RI USA

This presentation will discuss some recent developments in advanced plastic injection mold design and construction. Specifically, information will be presented regarding each of the following important mold characteristics: low thermal inertia inserts, high thermal conductivity mold materials, nickel active surfaces, copper thermal management layers, conformal cooling channels, sequential heating and cooling, quasi-isothermal active mold surfaces, reduced part distortion, and dramatically increased mold productivity. Finite Element Analysis results and actual mold test data will be presented comparing the performance of conventional steel tools with the new, advanced nickel-copper electroformed/steel backed tools.

4:40 PM

Use of Elemental Powder Blends in the Formation of Complex Alloys Using Lens™: *Hamish L. Fraser*[1]; Katrin Schwendner[1]; Sundar Amancherla[1]; Rajarshi Banerjee[1]; [1]The Ohio State University, Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

There are some very exciting possibilities afforded by application of LENS™ technology including rapid solidification without the need to post-consolidate particulate and net-shape processing. In contrast, there are some potential barriers to its widespread application, one being the cost involved in procuring pre-alloyed powders, particularly when the alloying elements are reactive, for example Ti. An obvious solution is the use of elemental blends of powders. The present study is aimed at assessing the viability of the use of elemental powders during LENS™ processing. As part of the initial assessment, elemental blends of Ni and Mo powders corresponding to the stoichiometry of Ni_3Mo have been processed and it has been shown that rapidly cooled, single phase material results. The compositional variations over the as-processed samples have been determined and it has been shown that the processed material is rather homogeneous. Among the various factors influencing the use of elemental powders is the heat of mixing. Some control experiments, aimed at assessing the influence of this factor, have been undertaken involving the use of elemental blends of Ti-Cr and Ti-Nb. Finally, the results of the processing of complex alloys taken from the TiAl, Mo-Si-B and Nb-Nb silicide systems will be presented and discussed.

5:00 PM

Microstructural Evolution During LENS Fabrication of H13 Tool Steel: *C. V. Robino*[1]; R. C. Dykhuizen[1]; J. A. Brooks[2]; T. J. Headley[1]; M. L. Griffith[1]; [1]Sandia National Laboratory, Albuquerque, NM 87185 USA; [2]Sandia National Laboratory, Livermore, CA 87185 USA

In the Laser Engineered Net Shape (LENS) process, parts are fabricated by creating a laser melted pool into which particles are injected. Fabrication proceeds by building the structure line by line and layer by layer. The process is thereby similar to multi-pass welding, and complex thermal histories are experienced in different regions of the build. These histories include remelting as well as numerous lower peak temperature thermal cycles. In the current work, models for the evolution of microstructure in H13 tool steel during the various cycles are being developed. These include solidification models, phase transformation models to account for reheating to intercritical peak temperatures, and carbide coarsening models to account for thermal cycles with subcritical peak temperatures. The models are based on classical descriptions of the various processes coupled with experimental measurements for the H13 alloy. The relationships between processing parameters and the resultant microstructures will also be discussed. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

International Symposium on Iridium: Oxidation and Diffusion

Sponsored by: Structural Materials Division, Refractory Metals Committee

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

Tuesday PM Room: Jackson A/B
March 14, 2000 Location: Opryland Convention Center

Session Chairs: Peter Panfilov, Urals State University, Strength Lab., Ekaterinburg 62001 Russia; Easo P. George, Oak Ridge National Laboratory, Metls. and Ceram. Div., Oak Ridge, TN 37831-6093 USA

2:00 PM

Effect of a Low Partial Pressure of Oxygen on the Grain Growth Characteristics of Iridium Alloys: *C. G. McKamey*[1]; E. H. Lee[1]; J. W. Cohron[2]; A. N. Gubbi[3]; J. L. Wright[1]; L. Heatherly[1]; E. P. George[1]; [1]Oak Ridge National Laboratory, Mets. and Cer. Div., P.O. Box 2008, Oak Ridge, TN 37831-6115 USA; [2]Byron Products Inc., 3781 Port Union Rd., Fairfield, OH 45014 USA; [3]Dentsply International, Preventive Care Div., 1301 Smile Way, York, PA 17404 USA

The grain-growth behavior in a low-pressure oxygen-containing atmosphere of an Ir-0.3 wt.% W alloy was studied. This alloy, designated DOP-26, is used for cladding of 238-plutonium oxide fuel in radioisotope thermoelectric generators (RTGs). A small amount of added thorium (~60 wppm) improves the impact properties through two different mechanisms: (1) by segregating to and strengthening the grain boundaries and (2) by forming Ir_5Th precipitates in the matrix which refine grain size. However, earlier studies showed that, in an atmosphere containing a low partial pressure of oxygen (on the order of 1 mPa), the dissolution of the Ir_5Th precipitates and diffusion of thorium to the surface to form ThO_2 is thermodynamically favorable, leading to anomalous growth of near-surface grains and reduced impact strength. In this study, specimens were exposed to oxygen partial pressures of 1.3 and 13.3 mPa at temperatures of 1230, 1280, and 1330°C for times up to 3000 h. The results show the presence of anomalous growth of near-surface grains in this alloy. However, the data also show that there is no significant difference in grain size or grain growth rates for the 1.3 versus 13.3 mPa oxygen levels. This tends to indicate that, at least under these exposure conditions, grain growth in DOP-26 is controlled by the outward diffusion of thorium rather than the inward diffusion of oxygen. This is supported by Auger results which show a depletion of thorium as one moves from the interior towards the surface. Research sponsored by the Office of Space and Defense Power Systems of the U.S. Department of Energy. This work was performed for the Department of Energy at the Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corporation, under contract DE-AC05-96OR22464.

2:20 PM

Oxidation Behaviour of Iridium-Based Intermetallic Compounds Doped with Boron: *I. M. Wolff*[1]; P. J. Hill[1]; [1]Mintek, Phys. Metallu. Div., Private Bag X3015, Randburg 2125 South Africa

Intermetallic compounds based on Ir and Ru are candidate materials for high-temperature applications, notably as spark plug electrode (SPE) materials. The high thermodynamic stability and resistance to deformation at elevated temperature have also profiled these alloys for structural applications. Of particular interest are the systems based on IrAl (B2), Ir_3Nb (L12), and RuAl (B2), which have high melting points, and exceptional mechanical properties. The well-known 'boron ductlising effect' has been found to improve both strength and ductility in the above systems. However, additions of as little as 0.5 at % boron modify the oxidation resistance of the RuAl based compounds, leading to escalated internal oxidation rates at temperatures approaching 1300°C. Current work seeks to elucidate the effect of boron additions on the oxidation resistance of the Ir-Al and Ir-Nb systems. Thermogravimetric analyses and static isothermal oxidation techniques have been used to characterise the operative oxidation mechanisms, which leads to a comparison between the behaviour of the ruthenium- and iridium-based alloys. The companion paper explores the convergence between the singular room temperature toughness in the RuAl system and the superior strength and oxidation resistance in the IrAl system in the RuxIr50-xAl50 series.

2:40 PM

Oxidation Behavior of IrAl Alloys Containing Ni: *H. Hosoda*[1]; S. Watanabe[2]; S. Hanada[2]; [1]University of Tsukuba, Instit. for Matls. Rsch., Tsukuba 305-8577 Japan; [2]Tohoku University, Instit. for Matls. Rsch., Sendai 980-8577 Japan

Oxidation behavior of B2 IrAl alloys containing Ni was investigated. IrAl has large potential to be ultrahigh-temperature oxygen-diffusion-barrier (ODB) due to formation of smart structure composed of Ir and Al_2O_3. Ir plays a role of ODB and formation of iridium oxides (IrO_2 and IrO_3) is suppressed by Al_2O_3 formed above Ir layer. A problem of IrAl fabricated by ingot metallurgy is that oxidation easily occurs at boundaries between IrAl and Ir domains: Ir domains are usually introduced through solidification. This may be solved by a powder metallurgical method and improvement of B2 phase stability, and Ni addition was expected to improve phase stability of B2 IrAl. Alloys containing 51 mol % Al were fabricated by a reactive hot pressing method using high-purity elemental powders. Oxidation tests were carried out under the conditions of (1) continuous heating of 10 K/min up to 1863 K and (2) isotherms between 1273 K and 1863 K in an O_2 atmosphere. Oxidation behavior was studied by simultaneous thermogravimetry (TG)- differential thermal analysis (DTA) and SEM observation. It was found that Ni addition dramatically improves oxidation resistance of IrAl. Effect of Ni addition on mechanical property is also discussed.

3:00 PM

Oxidation of Alloys Based on B2 IrxRu50-xA150: *P. J. Hill*[1]; I. M. Wolff[1]; L. A. Cornish[2]; M. J. Witcomb[3]; [1]Mintek, Phys. Metallu. Div., Private Bag X3015, Randburg 2125 South Africa; [2]University of the Witwatersrand, Schl. of Proc. and Matls. Eng., Private Bag 3, Wits 2050 South Africa; [3]University of the Witwatersrand, Elect. Micro. Unit, Private Bag 3, Wits 2050 South Africa

Alloys for application as spark-plug electrodes, requiring good high-temperature strength and exceptional oxidation resistance at elevated temperatures, are being investigated. Previous work has shown that alloys based on IrAl have exceptionally high hardnesses, and oxidation properties comparable with those of RuAl-based alloys. Additionally, some Ir-based alloys are known for their superior high-temperature mechanical properties. An isostructural B2 phase has been found to form between IrAl and RuAl, allowing substitution between Ir and Ru. Convergence between the room temperature toughness of RuAl, and the superior high-temperature mechanical and oxidation properties of IrAl is being sought. Although a thorough assessment of the oxidation, cyclic oxidation and hot corrosion behaviour of the alloys is required, an initial indication of the alloys' properties can be obtained from isothermal oxidation testing. This paper is concerned with the isothermal oxidation behaviour of IrxRu50-xAl50 alloys in the temperature range 900 to 1400°C. Thermogravimetric testing under air and techniques such as X-ray diffraction and metallography have been used to assess the oxidation behaviour of these alloys, allowing optimal alloy compositions to be selected.

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Mutual Diffusion in Couple of Iridium-Rhenium: *A. Smirnov*[1]; [1]Engelhard-Clal L.P., 700 Blair Rd., Carteret, NJ 07008 USA

Bimetal composition of a rhenium and iridium has provided high-temperature capability for usage in different applications-rocket nozzles or crucibles for crystals growth. The use of different materials at high-

temperature requires that allowance be made for possible changes of structure, strength and corrosion properties of composite material as a result of interdiffusion of components. In this work, we have studied mutual diffusion of bimetal compositions of iridium-rhenium, which was obtained by successive electrodeposition to form Ir and Re layers from conforming molten salts. Representative samples of composite material were vacuum annealed at 1950°C for different time. Formation and expansion of diffusion zone will be discussed.

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On Brittle Fracture of Iridium and Iridium-Based Alloys Exposed to Melts of Chemical Elements: V. A. Dmitriev[1]; N. I. Timofeyev[1]; *A. V. Ermakov*[1]; [1]Ekaterinburg Non-Ferrous Metals Processing Plant, The Head of Rsch. Ctr., Lenin Ave. 8, Ekaterinburg 620014 Russia

Newest area for application of iridium is manufacture of reactors for pyrometallurgical extraction of gold and silver from industrial scraps, inasmuch as it is the sole metal that can be in contact with melted metals during long time. Study of iridium contacted with liquid Ag, Au, Pb, Sn, Zn, and Al in vacuum is the subjects for current work. It was shown that iridium does not chemically interact with melted noble metals and led, while thin films of Au and Ag appear on the samples contacted with melts. Despite this they do not embrittled iridium. On the contrary, liquid zinc, aluminum and tin dissolve iridium, what is the cause for failure of iridium containers.

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Mechanical Properties and Oxidation Resistance of Ir-Added FeAl Alloys: *H. Hosoda*[1]; K. Yoshimi[2]; S. Miyazaki[1]; S. Watanabe[2]; S. Hanada[2]; [1]University of Tsukuba, Instit. of Matls. Sci., Tsukuba 305-8577 Japan; [2]Tohoku University, Instit. of Matls. Sci., Sendai 980-8577 Japan

Effects of micro- and macro-alloying with Ir on mechanical properties and oxidation resistance were investigated for B2 FeAl alloys. Concentrations of Al and Ir were systematically changed to be 30, 35, and 40 mol % AL and 0 (binary FeAl), 1 and 5 mil % Ir, where additional Ir must substitute Fe sites. These Ir-added FeAL alloys were fabricated by a reactive hot pressing (RHP) method using elemental powders of 99.9% purity. RHP was carried out at 1523 K for 10.8 ks under 70 MPa in vacuum. Vacancy-eliminating and vacancy-introducing heating treatments were additionally carried out. ICP-OES chemical analysis, optical microscopy, differential scanning calorimetry up to 1773 K and X-ray diffractometry were carried out for alloy characterization. Mechanical properties were investigated by Vickers hardness tests at ambient temperature and compression tests at elevated temperature. Oxidation behavior was evaluated using simultaneous thermogravimetry-differential thermal analysis at 1273 K in an 0_2 atmosphere. Effect of vacancy on mechanical property and oxidation of Ir-added FeAl alloys will be also discussed.

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Grain-Boundary Diffusion of 57Co and 195Au Atomic Probes in Polycrystalline Iridium: *A. V. Ermakov*[1]; S. M. Klotsman[2]; V. N. Kaigorodov[2]; S. A. Matveev[2]; V. K. Rudenko[1]; G. N. Tatarinova[2]; A. N. Timofeev[2]; N. I. Timofeev[1]; [1]Ekaterinburg Non-Ferrous Metal Processing Plant, Lenin Ave. 8, Ekaterinburg 620014 Russia; [2]Institute of Metal Physics, Russian Acad. of Sci., S. Kovalevskaya 18, Ekaterinburg 620219 Russia

The pioneering study of intercrystallite diffusion of 57Co and 195Au atomic probes in polycrystalline iridium (poly-Ir) produced by deformation and subsequent recrystallization of a single crystal was performed. The sectioning analysis of the diffusion zone was used. It included traditional measurements of "layer activities" and measurements of intensity of single-energy components of the atomic probe radiation from the rest of the sample left after part of the intercrystallite diffusion zone was removed. Functions sensitive to the diffusion profile type showed that a one-dimensional diffusion flow was absent at homologous temperatures, which are extremely low for FCC metals. In poly-Ir two-dimensional diffusion flow was realized at these conditions. Existence of the two-dimensional diffusion flux provides the high occupancy of states which are localized outside the grain boundary core even at such low homologous temperatures as 0.2Tmelt. The intercrystallite diffusion parameters of 57Co in poly-W and poly-Ir at comparable homologous temperatures are nearly equal. This fact is due to the same type of interatomic interactions in d-transition metals.

Kleppa Symposium on High Temperature Thermochemistry of Materials: Session IV

Sponsored by: ASM International: Materials Science Critical Technology Sector, Extraction & Processing Division, Thermodynamics & Phase Equilibria Committee, Process Fundamentals Committee

Program Organizers: Ray Y. Lin, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA; Y. Austin Chang, University of Wisconsin, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Dr. Susan Meschel, The University of Chicago, Chicago, IL 60637 USA; Ramana Reddy, University of Alabama, Department of Metals and Materials Engineering, Tuscaloosa, AL 35487 USA

Tuesday PM Room: Lincoln E
March 14, 2000 Location: Opryland Convention Center

Session Chairs: Ramana Reddy, University of Alabama, Dept. of Met. & Matls. Eng., Tuscaloosa, AL 35487 USA; Alexandra Navrotsky, University of California, Dept. of Chem Eng. & Matls. Sci., Davis, CA 95616 USA

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A Thermodynamic Study of the Al-Cr-Nb Ternary System: *Kamal Mahdouk*[1]; Jean Claude Gachon[2]; [1]Universite Ibnou Zohr, Thermo. Metallu. et Rheo. des Mat., B.P. 28/S, Agadir Morroco; [2]Universite Henri Poincare, Lab. de Chimie du Solide Min., UMR 7555 Nancy 1, B.P. 239, Vandoeuvre, Cedex 54506 France

Al-Cr-Nb is one of the ternaries which is of interest in understanding the behavior of many industrial alloys. At the same time, the available results are confusing. We have undertaken an experimental study of both phase equilibria and enthalpies of formation in this system. Alloys were synthesized by reaction between powders of the pure metals and annealing at 1000°C followed by water quenching. Equilibria were characterized by X-ray diffraction and electron microprobe analysis. Enthalpies of formation were measured by direct reaction calorimetry for most of the binary intermetallics as well as for some solid and liquid solutions. The experimental results will be given and compared to existing data available in literature. The ultimate task will be a new assessment of the system.

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Thermodynamic Properties of Ti-Al Alloys: *R. G. Reddy*[1]; Leo Brewer[2]; [1]The University of Alabama, Metallu. and Matls. Eng., P. O. Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]University of California, Chem. Dept., M.C.1460, Berkeley, CA 94720 USA

Activities of Al in Ti-Al alloys were determined using the emf method. The emf of concentration cell was measured in the temperature range 820 to 900K. Experimental results, activities of Al in alloys showed a large negative deviations from the Raoult's law. The activity of Al 2.98x10-3 for the molefraction of Al 0.48 in Ti-Al alloys at 850K was determined.

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A Model for Predicting Thermodynamic Properties of Metallic Solutions from Fundamental Physical Quantities of Constituent Elements: *Peng Fan*[1]; [1]The University of Tokyo, Dept. of Metallu. and Matls. Sci., Hongo 7-3-1, Bunkyo-ku, Tokyo 113-8656 Japan

A new model for predicting thermodynamic properties of metallic solutions from the fundamental physical quantities of constituent elements has been developed, based on the new generation geometrical model proposed by Chou and Miedema model. The equations for interaction parameters in multi-component systems and for activity coefficients and excess Gibbs free energy in ternary systems have been derived. Theoretical discussions on these equations show that it is more reasonable using the new generation geometrical model than using other geometrical models such as Toop model and Kohler model. This model has been applied for predicting interaction parameters in a large number of metallic solutions, and the agreement between prediction and experimental data is reasonable.

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Thermodynamic Properties of Laves Phases: *J. H. Zhu*[1]; C. T. Liu[1]; [1]Oak Ridge National Laboratory, Mets. and Cer. Div., P.O. Box 2008, Bldg. 4500S, MS 6115, Oak Ridge, TN 35831-6115 USA

Thermodynamic properties of binary and ternary Laves phases have been critically surveyed and reviewed. The enthalpies of formation of many Laves phases have been determined recently by Dr. Kleppa and his coworkers using the high-temperature calorimetry. The vibrational entropies of formation of many Laves phases are now also available. Some regularities in the thermodynamic properties of Laves phases were deduced using the experimental data. A thermodynamic interpretation is offered to explain the size ratio limits for Laves phase formation. As the deviation from the ideal size ratio increases, the maximum negative enthalpy of formation decreases linearly, which is assumed to be due to the elastic strain energy expended in compressing the atoms. At RA/RB =1.03 and 1.65, the enthalpy of formation reaches zero. Further deviation in the RA/RB ratio will lead to the enthalpy of formation positive. Thus, the free energy of formation becomes positive, due to the negligible entropy of formation term. Therefore, Laves phases can only be stabilized in certain atomic size, RA/RB, ratios. The enthalpies of formation of many binary Laves phases were calculated by the semiempirical Miedema model, which showed good agreement with the available experimental data. Finally, the importance of understanding thermodynamic properties of Laves phases in predicting phase stability, point defects, and glass formability in these phases is discussed.

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In Situ Observation of Carburizing Reaction of Solid Iron with Graphite under Different Oxygen Potentials: *Taichi Murakami*[1]; Hiroyuki Fukuyama[1]; Miyuki Hayashi[1]; Masahiro Susa[2]; Kazuhiro Nagata[1]; [1]Tokyo Institute of Technology, Chem. and Matls. Sci., 2-12-1, Ookayama, Meguro-ku, Tokyo 152-8552 Japan; [2]Tokyo Institute of Technology, Metallu. and Ceramic Sci., 2-12-1, Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Exhaustion of fossil fuel and minimization of carbon dioxide emissions have been serious and urgent problems in the current steelmaking industry. From this point of view, further development, aiming at more efficient operations in the ironmaking process, is required for the coming 21st century. Temperature is one of the most effective operational factors in the ironmaking process, which is a key to reduce consumption of energy and emission of carbon dioxide. In order to make production of pig iron at temperatures as low as possible, better understandings of the carburizing reaction of solid iron are essentially required. In the present paper, reactions between solid iron and graphite have been observed in situ using a high-temperature microscope which enables a sample to be heated up to 1473K in one minute. The effect of oxygen potential on the carburizing reaction is also discussed.

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In-SnZn and Al-Sn-Zn New Solder Materials: *A. Mikula*[1]; [1]University of Vienna, Instit. of Inorg. Chem., Waehringerstrasse 42, Vienna A-1090 Austria

Lead-tin solders are commonly used in the electronic packaging due to their unique combination of electrical, chemical, physical, thermal and mechanical properties. Since lead alloys cause great environmental concern and health hazards it is more economical to replace lead in solder materials instead of cleaning up the electronic waste. To get the same or better properties it is therefore necessary to investigate some multicomponent systems. Two candidates are the In-Sn-Zn and Al-Sn-Zn systems. DTA measurements, lattice parameter investigations and micro-probe analyses were performed to determine the phase diagrams. The thermodynamic date was obtained by emf and calorimetric measurements. Based on this data the ternary phase diagrams were optimised by different models. A theoretical investigation of the bulk as well as the surface of liquid solder alloys has been made through the study of the concentration dependence of various properties such as concentration fluctuations in the long wavelength limit, diffusion, surface tension and surface composition. A statistical mechanical theory based on the layered structure has been used to develop expressions for the surface tension and surface composition in the frame-work of self-association and compound formation models.

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Hydrogen Storage Alloys and Their Application to Hydride Batteries: *Kuochih Hong*[1]; [1]Evergreen Energy Technology Corporation, Taipei, Taiwan

Hydrogen can be stored in a suitable metal as a solid metal hydride. There are many potential applications for the metal hydride technology. The most important one today is the development of commercial rechargeable hydride batteries. A metal hydride battery has many advantages over a nickel cadmium battery such as (1) much higher capacity, (2) no memory effect and, (3) no environmental pollution. Not every hydrogen storage material can be used as a hydrogen storage/hydride electrode. The criteria of useful hydrogen storage electrode alloys will be discussed. A semi-empirical formula, based on thermodynamic and electrochemical approach to predict and/or develop a useful hydride electrode alloy, Abx, is presented. There are two major alloy systems: Abx titanium-based and Abx rare earth metal-based alloy, currently used in the nickel hydride batteries. In general, a titanium-based alloy can have a higher electrochemical capacity than that of a rare earth metal-based alloy. However, a sealed nicked hydride battery using a rare earth metal-based alloy as the active material of the anode generally has a better electrochemical performance, including high working potential, high rate charging and discharging and lower internal pressure during overcharging. A brief discussion of making a nickel hydride battery will be described. Finally, several examples of hydrogen storage electrode alloys and their thermodynamic and electrochemical properties will be presented.

Liquid Metal Atomization: Fundamentals and Practice: Overview and Visualization

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

Tuesday PM Room: Bayou B
March 14, 2000 Location: Opryland Convention Center

Session Chairs: Fernand Marquis, South Dakota School of Mines and Technology, Dept. of Matls. & Metal Eng., Rapid City, SD 57701-3995 USA; Khershed P. Cooper, Naval Research Laboratory, Matls. Sci. & Tech., Washington, DC 20375-5343 USA

2:00 PM Opening Remarks and Introductions:
Khershed P. Cooper, Primary Organizer, Powder Materials Committee

2:10 PM Keynote

Metal Powder Production Via Melt Atomization: *Alan Lawley*[1]; [1]Drexel University, Dept. of Matls. Eng., LeBow Matls. Bldg., Philadelphia, PA 19104 USA

The fundamentals and industrial practice of liquid metal atomization are reviewed with particular reference to the interplay of melt properties and processing conditions on powder characteristics (size, size distribution, shape). Empirical relations, models and mechanisms are assessed for commercial and near-commercial water, gas, vacuum and centrifugal atomization and their implications considered with respect to the production of fine powders. The importance of powder yield and particle size distribution on the economics of atomization are illustrated. To conclude, trends and challenges in industrial-scale atomization are delineated.

2:45 PM Invited

Liquid Metal Atomization: Fundamentals and Practice: *Klaus Bauckhage*[1]; [1]Universitat Bremen, Institut fur Werkstofftechnik, Badgasteiner StraBe 3, Bremen D-28359 Deutschland

Atomization of liquid metals can be achieved in a variety of ways, most of them, in principle, show more or less similarity to the atomization techniques of “normal liquids”, i.e. of aqueous or oil-based pure liquids, suspensions or emulsions. But since most of the technically interesting metal melts need significantly higher temperatures for atomization compared with normal liquids this in consequence results in a limitation of the variety of those techniques and in modifications of the devices. It is not only the supply of high kinetic energy (with reference to the high surface tension of the majority of melts) but also of heat energy at, in most cases, high temperature levels in order to keep the melt liquid which often in combination with a high reactivity of the liquid metals requires special materials, purpose-built devices and tailor made processes. The fundamentals mainly deal with the heat and mass transfer balances starting at the melt flow in the tundish, including the melt break-up mechanism and ending with the cooling and solidification conditions of the particles in the spray cone. The paper confines the discussion of fundamentals and practice to the most commonly and commercially used atomization techniques as for instance two-fluid atomization (gas/melt and water/melt), centrifugal atomization (by pouring the melt onto a rotating disc), and ultrasonic or ultrasonic assisted atomization.

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Fundamental Fluid Dynamics in Liquid Metal Atomization: *Qingzhou Xu*[1]; Yizhang Zhou[1]; Gerardo Trapaga[2]; Enrique J. Lavernia[1]; [1]University of California, Dept. of Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697 USA; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. and Eng., Cambridge, MA 02139 USA

Computational fluid dynamic techniques are used to analyze velocity and pressure fields as well as the influences of processing parameters and geometric arrangements during the liquid metal atomization process. The calculated results are summarized as follows. At first, the atomization gas is accelerated to a certain velocity as it expands through the orifice of an atomizer from the high pressure gas reservoir into the low pressure chamber. Under high atomization pressures, the atomization gas at the exit of the atomizer reaches the speed of sound and is typically underexpanded. It is conically focused in front of the liquid metal delivery nozzle, and then, spreads and decelerates into the atmospheric environment. When the atomization gas enters into the chamber at a high speed, recirculating flow fields often form in the vicinity of the delivery nozzle; and meanwhile, negative (or aspiration) pressure zones with respect to the atmospheric pressure may occur there. The aspiration pressure is determined by atomization pressure, delivery nozzle geometry and its protrusion length. In addition, the protrusion length of the delivery nozzle also influences the velocity field since the incoming atomization gas bounces off on its surface in the initial stage.

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Gas Flow Imaging and High Speed Photography of Gas Atomization: *Iver E. Anderson*[1]; R. L. Terpstra[1]; Jason Ting[2]; [1]Iowa State University, Ames Lab., 126 Metals Dev., Ames, IA 50011-3020 USA; [2]Crucible Research, Pittsburgh, PA 15205 USA

High pressure gas atomization (HPGA), a close-coupled, discrete jet atomization method, has proved to be one of the most effective methods of producing rapidly solidified fine metal and alloy powders with high yields less than 20 microns using Ar, N_2, or He gas. Visualization of gas-only flows and high speed photography of the atomization process have provided valuable insight to guide the development of a succession of HPGA nozzles. This presentation will compare the gas flow characteristics of convergent and convergent-divergent single jets and full gas jet ensembles used for HPGA. High speed photography and cinematography characterization of the HPGA process will also be presented, including brief high speed movie selections. An enhanced understanding of the melt disintegration mechanisms that operate during HPGA processing can be derived from this data. Different aspects of this work were supported by USDOE-BES under contract no. W-7405-Eng-82 and the US Naval Research Laboratory.

4:30 PM Invited

Optimization of an Annular Jet Commercial Gas-Metal Atomizer: *Aaron Johnson*[1]; Frank S. Biancaniello[1]; Stephen D. Ridder[1]; P. I. Espina[1]; G. J. DelCorso[1]; [1]NIST, Fluid Flow Grp., 100 Bureau Dr., Gaithersburg, MD 20899 USA

The performance of a commercial gas-metal atomizer was studied using a number of previously published research techniques. Initially the flow was visualized via schlieren photography to determine the location of important flow features (e.g., shock waves, expansion fans, shear layers). With this information at hand, an experiment was designed to determine the aspiration performance of two prototype geometries. The aspiration results were confirmed with the help of computational fluid dynamic models, which although not as accurate, yielded more spatial resolution of the phenomena at hand. Using these results, a number of 316 SS production runs were performed, and it was concluded that the recommendations suggested by the fluid experiments produced improvements in the fine powder yield of the considered geometries.

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Analysis of Molten Metal Atomization Process Using Modified Phase-Doppler-Anemometry: *Jens Ziesenis*[1]; Jörg Tillwick[1]; Volker Uhlenwinkel[1]; [1]Universität Bremen, FB4/FG01, Badgasteinerstr. 3, Bremen 28359 Germany

Phase-Doppler-Anemometry (PDA) is an appropriate tool to investigate sprays. Since standard phase-Doppler systems can easily be applied to conventional materials, e.g. small water droplets or metal spheres with smooth surfaces, the optical behaviour of the solidifying particles in the liquid atomization process required the modification of the PDA technique. To demonstrate the sensibility of the modified Phase-Doppler-Anemometry results obtained by examining the spray of a moved atomizer are shown. With this modified non-intrusive, optical measurement method not only characterisation of local particle size and velocity distributions but also additional information as for instance the determination of local mass flux distributions of the spray cone can be obtained. For the particle properties the PDA results are compared to results received with different measurement techniques including sieve analysis of particles, that are collected using water filled tubes and with results obtained using a infrared pyrometer for inflight diagnostics on individual particles. Furthermore the comparison between measured PDA results and results of simulations of the metal atomization process is demonstrated.

Magnesium Technology 2000: Alloy Development and Corrosion

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

Tuesday PM Room: Bayou C
March 14, 2000 Location: Opryland Convention Center

Session Chair: Eli Aghion, Dead Sea Magnesium, Potash House, Beer-Sheva 84100 Israel

2:00 PM
Corrosion and Galvanic Corrosion of Die Cast Magnesium Alloys: *J. Senf*[1]; E. Broszeit[1]; M. Gugau[1]; C. Berger[1]; [1]Darmstadt University of Technology, Instit. of Matls. Tech., Grafenstr. 2, Darmstadt 64283 Germany

One main disadvantage of magnesium alloys is the chemical and electrochemical reactivity of this group of materials. Consequences of this reactivity are problems in functionality, reliability and lifetime of machines and constructions made of these alloys. High purity alloys have reduced corrosion sensitivity. But looking at corrosion behaviour, the corrosion of magnesium alloys for itself is only one part of the corrosion problem. Galvanic corrosion is the other and maybe the main part, especially for machines and vehicles. Typical for the galvanic corrosion of different metallic materials in contact with each other is the enhanced corrosion of the minor noble material. Magnesium alloys are the least noble metallic materials used for machine parts. In this paper corrosion and galvanic corrosion behaviour of four die cast magnesium alloys (AZ 91, AM 60, AS 41, AE 42) are presented and discussed. Looking at the galvanic corrosion the magnesium alloys were connected to different aluminium alloys typically used in the automotive industry.

2:25 PM
Laboratory Evaluation of Galvanic Corrosion Resistance of Anodizing Film on Magnesium: Vladimir Tchervyakov[1]; *Guilian Gao*[1]; John Bomback[1]; Gerry Cole[1]; [1]Ford Research Laboratory, Matls. Sci. Dept., MD 3182 SRL, 20000 Rotunda Dr., Dearborn, MI 48121 USA

Galvanic corrosion of magnesium alloys is the major issue in corrosion reliability of Mg alloys in automotive applications. Anodization is a very effective way of corrosion protection for magnesium alloys. In this paper, the corrosion resistance of AZ91 alloys with different surface treatment (bare, anodized, anodized and sealed) were compared. Electrochemical test results showed that anodization not only improves general corrosion resistance, but also significantly increases its resistance to galvanic corrosion. Sealing the anodized film is also shown to further improve the corrosion resistance.

2:50 PM
Characterisation of Manganese-Containing Intermetallic Particles and Corrosion Behaviour of Die Cast Mg-Al-Based Alloys: *Liu-Ying Wei*[1]; Håkon Westengen[2]; Terje Kr. Aune[2]; Darryl Albright[3]; [1]Luleå University of Technology, Div. of Eng. Matls., Luleå S97187 Sweden; [2]Norsk Hydro ASA, Rsch. Ctr. Porsgrunn, P.O. Box 2560, Porsgrunn N-3901 Norway; [3]Hydro Magnesium, Magnesium Market Dev., 39209 Six Mile Rd., Ste. 200, Livonia, MI 48152 USA

Manganese is an element often used to remove iron from the magnesium melts in order to produce high purity magnesium alloys. A study of manganese-containing particles in the magnesium alloys is therefore of technological significance with regard to the corrosion properties of the Mg-Al-based alloys. TEM/SEM investigations of the as-cast microstructure and the corrosion morphologies revealed that the corrosion resistance of the studied alloys is dependent on (1) the distribution and types of small Mn-containing particles where corrosion pits could form to initiate corrosion fissures, (2) the barrier effects for the fissures propagation in the alloys. Two types of manganese-containing phases were found in the alloys studied. Type I is a flower-shaped phase and type II is of equiaxed or short-bar morphologies. The type II particles have a lower Al/Mn ratio and a much higher cathodic reaction rate than the type I phase. The type II particles could provide sites to form pits and initiate corrosion fissures. The segregated regions of higher Al content adjacent to the grain boundaries and the β-$Mg_{17}Al_{12}$ particles could act as barriers to the propagation of corrosion fissures.

3:15 PM
Mechanochemical Characteristics and Perspectives of New Magnesium-Lithium-Alloys: *H. Haferkamp*[1]; P. Juchmann[1]; V. Kaese[1]; M. Niemeyer[1]; T. Phan-tan[1]; [1]University of Hanover, Instit. of Matls. Sci., Fachgruppe Nichteisen-Metallurgie, Applestrasse 11a, Hannover 30167 Germany

On the base of growing tendencies towards an ecological increase in efficiency of technical products and processes it is the light weight material magnesium that enjoys recurrent attention. However, its basic attraction is presently spoiled by the deficiency of alloys that doesn't allow more complex applications. Therefore the general aspects of the alloy development are focused on improving creep resistance and enhancement corrosion resistance as well as the increase of plasticity. The aim of current research activities at the Institute of Materials Science is the development of Magnesium-Lithium-alloys with further lowered density, high ductility and high corrosion resistance. The presented research work includes a discussion of phase constitutions and their impacts on mechanical and corrosive properties. A variety of Aluminium and Silicium alloyed cph Magnesium-Lithium-materials is tested. Investigations include tensile or impact bending tests, potentiostatic tests, atmospheric corrosion test and water vapour cabinet. The materials are compared to Mg standard materials. A selection of Magnesium-Lithium-materials shows superior strength and ductility properties in comparison to representative standard Magnesium materials. Principal mechanisms of the active corrosion protection of Magnesium alloys are presented and used to achieve a satisfying corrosion resistance.

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Microstructure Property Studies of In Situ Mechanically Worked PVD Mg-Ti Alloys: *Tim Mitchell*[1]; *Panayiotis Tsakiropoulos*[1]; [1]University of Surrey, Schl. of Mech. and Matls. Eng., Guildford, Surrey GU25XH England

Physical vapour deposition is suitable for the production of Mg-TM alloys. Typical microstructures of PVD alloys consist of columnar grains and exhibit inter-columnar porosity and chemical inhomogeneity, the latter being present in bands growing perpendicular to the direction of growth of the columnar grains during deposition. Porosity and chemical inhomogeneity are detrimental to the mechanical properties and the corrosion resistance of the alloys. In situ mechanical working by flailing has been used previously to reduce porosity and eliminate columnar crystals in the microstructures of PVD deposits. In this work flailing was used in the production of PVD Mg-xTi (x=8, 14, 26, 34, 40 and 48wt%) alloys. The microstructures and surfaces of alloys in the as deposited condition and after immersion in 3wt% NaCl were characterised by electron microscopy and surface analysis techniques. The solid solubility of Ti in Mg was extended to 48wt%Ti by PVD. Flailing eliminated the columnar structure and reduced porosity locally in the regions where mechanical working was applied. In these regions there was some evidence of recrystallisation of the microstructure. The corrosion resistance of the flailed alloys decreased with increasing alloying content and was inferior to the corrosion resistance of PVD Mg. This behaviour has been attributed to the localised effects of flailing, with preferential attack occurring along the flailing lines leading to exfoliation of the deposits.

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Studies of Mg-V and Mg-Zr Alloys: *Spyros Diplas*[1]; *Panayiotis Tsakiropoulos*[1]; [1]University of Surrey, Schl. of Mech. and Matls. Eng., Matls. Sci.and Eng., Guildford, Surrey GU25XH England

Physical vapour deposition is a non-equilibrium process which is suitable for the production of Mg-X alloys, where X is alloying element with significantly higher melting point than Mg. Thus, PVD has been used for the development of Mg alloys with improved mechanical properties and corrosion resistance. As part of this effort Mg-TM alloys have been considered, where the choice of TM has been influenced by the need for a beneficial contribution of the alloying addition to the formation of a stable and if possible self-healing surface film, which could improve corrosion resistance in saline environment. Alloy design has selected candidate solute additions which include V and Zr. In this paper we will report on PVD Mg-V and Mg-Zr alloys which have been studied by bulk and surface characterisation techniques in the as-deposited condition and after immersion in 3wt% NaCl. All deposits exhibited compositional inhomogeneity, columnar microstructures and a strong basal texture. The solid solubilities of V and Zr in Mg were extended approximately to 17.5 wt% V (8.9at%) and 10.5 wt% Zr (3.5at%) respectively. The solid solution break up temperature decreased as the V and Zr content in the alloys increased. Pure V precipitated when the extended solid solubility of V in Mg was exceeded. Both the c and a lattice parameters, as well as the c/a ratio decreased with increasing V content in the Mg-V alloys. The increase of the a- lattice parameter and the decrease of the c lattice parameter led to a decrease of the c/a ratio with increasing Zr additions in the Mg-Zr alloys. The two alloy systems behaved differently regarding corrosion resistance. Mg-V alloys exhibited lower corrosion resistant than pure Mg or other Mg alloys, which has been attributed to the absence of V from the surface film formed on the alloys. Mg-Zr exhibited extremely low corrosion rates, which were attributed to the participation of Zr in the surfaces of both the as-deposited and corroded alloys.

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Solubility of Nickel in Molten Magnesium-Aluminium Alloys Above 650°C: *Harsharn Singh Tathgar*[1]; Per Bakke[2]; Eivind Øvrelid[3]; Jo Fenstad[1]; Thorvald Abel Engh[1]; Frede Frisvold[3]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. and Electrochem., Alfred Getz Vei 2B, Trondheim N-7491 Norway; [2]Norsk Hydro Research Centre, Porsgrunn N-3901 Norway; [3]SINTEF, Matls. Tech., Trondheim N-7465 Norway

Solubility of nickel in liquid Mg-Al-Ni alloys has been measured in the temperature range 650-900°C for 1-10% aluminium content. Comparison of nickel solubility in pure magnesium with solubility in Mg-Al alloys shows a marked reduction of nickel solubility with a small addition of aluminium. SEM analysis shows that the precipitated phases in equilibrium with the melt are AlNi and $Al3Ni_2$ with the latter becoming more dominant as the aluminium concentration increases. The solubility of Ni in equilibrium with phase AlNi in the melt according to the dissolution reaction AlNi = Al + Ni is given. The thermodynamic parameters evaluated using the least square method are also presented.

5:05 PM

A Perspective View on the Design of Magnesium Alloys: How Computational Thermodynamics Can Help: *Zi-Kui Liu*[1]; [1]The Pennsylvania State University, Dept. of Matls. Sci. and Eng., University Park, PA 16802 USA

Alloy development is traditionally conducted by “make-and-see” empirical approach, which is usually costly and time consuming. Alloy theories tend to explain the behavior of materials after their development. The situation is now changing thanks to the accumulated knowledge on alloy theories in the past century and the significant progress in computational techniques in the past decade. One of the emerging fields is the systems materials design. Systems materials design integrates processing, structure and properties through computational thermodynamics, kinetic simulations, and experimental prototype evaluations. The essential feature is to express the design objectives in terms of thermodynamic and kinetic parameters. Thermodynamics has often been viewed applicable to states near equilibrium only. The CALPHAD technique for computational thermodynamics developed since early 1970’s has changed this view. This technique couples the phase diagram and thermochemical properties to explicitly characterize all phases in a system, including stable, metastable, and unstable phases over a wide range of temperature, pressure and composition. The modeling of Gibbs energy of individual phases enables the calculation of driving forces between any intermediate non-equilibrium states for simulating dynamic microstructure evolutions. As all commercial alloys, magnesium alloys are multi-component in nature with many intermetallic phases. To develop robust alloys that less sensitive to process variability, phase relations in multi-component alloys under both equilibrium and non-equilibrium conditions are extremely valuable for the design of alloy compositions and processing procedures. In this presentation, the concept of systems materials design applied to magnesium alloys will be introduced, and the CALPHAD technique will be discussed. Particular attention will be paid to the phase relations in the Mg-Al-Zn ternary alloys covering the composition range from AZ series to ZA series along with the future activities of the systems design of magnesium alloys.

Materials Processing in the Computer Age III: Physical and Mathematical Modeling of Materials Processes

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: Vaughan Voller, University of Minnesota, Saint Anthony Falls Laboratory, Minneapolis, MN 55414-2196 USA; Hani Henein, University of Alberta, Edmonton, AB T6G 2G6 Canada; Sulekh Jain, Ge Aircraft Engineering, MId M-89, Cincinatti, OH 45215 USA

Tuesday PM — Room: Lincoln A
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: James W. Evans, University of California, Dept. of Matls. Sci. and Min. Eng., Berkeley, CA 94720 USA; Daniel Paul Cook, Reynolds Metals Company, Corp. Rsch. and Dev., Chester, VA 23831 USA

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Physical Experimentation Coupled with Mathematical Modeling of Aluminum Automotive Wheel Casting Operations: *Daniel P. Cook*[1]; Melissa I. Bloch[2]; Juergen Biermann[3]; Jonathan A. Dantzig[2]; [1]Reynolds Metals Company, Corp. Rsch. and Dev., 13203 N. Enon Church Rd., Chester, VA 23831 USA; [3]SIMTEC Inc., 3663 Broadmoor SE, Grand Rapids, MI 49512 USA

The percentage of automotive parts manufactured from aluminum alloys has been increasing since the price of gasoline started to increase in the 1970’s. The car wheel is one of these components which has been most successfully produced in aluminum. Reynolds Metals currently produces roughly 6.5 million wheels per year with production slated to increase to almost 8 million per year in the next two years. In this paper, results will be presented from plant trials conducted in a low-pressure die casting facility. Temperature measurements were conducted at a number of positions in a wheel mold during normal operation. This data yielded information such as the thermal profile in the mold side cores and the mold filling time. This data was used to validate several mathematical models of filling and solidification in wheel casting. Changes in casting practice, e.g. mold coating application, inlet pressure map, die cooling modification, and mold design suggested by further model calculations will be presented.

2:50 PM

A Capacitance Probe for the Measurement of Bubbles in Molten Metals: Qian Fu[1]; *James W. Evans*[1]; [1]University of California, Dept. of Matls. Sci. and Min. Eng., Berkeley, CA 94720 USA

There are many metallurgical processes where bubbles are injected into high temperature melts. Obvious examples are copper converters and the chlorine fluxing units found in the aluminum industry. These melts are opaque and the bubbles within the melt are therefore not readily observed. Electroresistivity probes have been developed (notably by Brimacombe's and by Iguchi's groups) to measure the frequency, size and velocity of bubbles in liquid metals. The probes work by detecting the interruption of electrical contact between a conductor (usually metallic) immersed in the melt and the surrounding melt, as a bubble intercepts the conductor. The paper describes an alternative probe which relies on a measurement of the capacitance between a conductor within the probe and the melt; that capacitance changes as a bubble passes. Unlike the electroresistivity probe, this capacitance probe does not have its conductor in contact with the melt, rather it is shielded by a sheath of refractory material such as alumina. Consequently the probe can withstand hostile environments and one such probe has survived, while detecting bubbles for 30 minutes in molten aluminum. The paper describes the probe construction, illustrates the signals it gives in molten metals and treats the deconvolution algorithm and software necessary to convert the bubble signals to useful information.

3:10 PM

Argon Bubble Behavior in Tundish Nozzles during Continuous Casting of Steel Slabs: *Hua Bai*[1]; Brian G. Thomas[1]; [1]University of Illinois, Mech. Eng. Dept., 1206 W. Green St., Urbana, IL 61801 USA

Argon injection into tundish nozzle is an efficient and widely employed method to reduce nozzle clogging in continuous casting process. It also affects casting operation and product quality by changing the flow pattern in the nozzle and mold. In this paper, a 3-D finite difference model is developed to study the liquid steel-argon bubble multi-phase turbulent flow in continuous casting tundish nozzles. Experiments are performed on a 0.4-scale "water caster" to verify the model by comparing the model prediction with the measurements using PIV (Particle Image Velocimetry) technology. The developed model is then employed to investigate the effects of various variables on flow patterns and jet characteristics for the slide-gate nozzle. Because the input and output between the real-life continuous casting operation and numerical simulation are usually different, HPQF model, based on advanced curve fitting of the multiple variable numerical results, is then developed and applied to convert the numerical modeling results to present trends that correspond with real-life operation conditions.

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Physical Modeling of the Viscosities of Two-Phase Mixtures: *Wei Liu*[1]; Sichen Du[1]; Seshadri Seetharaman[1]; [1]Royal Institute of Technology, Metallu. Dept., Brinellv. 23, Stockholm 10044 Sweden

The viscosities of two-phase mixtures are of great importance in the modelling of the fluid flow in metallurgical processes. For example, in steelmaking and refining, the intimate mixing of metal droplets in the slag phase requires a consideration of the effective viscosities for the mixture. The present work was aimed at an understanding the phenomena underlying the viscous flow in a two-phase mixture with widely differing viscosities. Emulsions of silicone oils of known viscosities (3.45 dPa·s and 10.10 dPa·s at 293 K) with small amounts of water evenly distributed in the same were prepared by subjecting the mixture to uniform stirring using a magnetic stirrer. The uniformity of the emulsions was checked by photography. The viscosities of these emulsions were measured at constant temperature by the rotating cylinder method using Brookfield Digital Rheometer. The uniformity of the method of preparation of the emulsions was confirmed by the reproducibility of the results. The measured viscosities were generally found to be independent of the torque under the experimental conditions so that the two-phase mixture could be considered as a Newtonian liquid. The variations of viscosities with temperature and the effect of addition of a surface-active substance were also studied in this work. The measured viscosities were found to be higher than those of both pure water and silicone oil. The experimental viscosities showed a positive deviation from linearity and the deviation was found to increase with increasing concentration of water. The influence of surface tension on the viscosities was examined by the measurement of contact angle and evaluating the interfacial tension between the silicon oil and water. The results are discussed in the light of the various forces involved in the viscous flow in such systems as well as the chemical affinities.

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Fluid Flow and Particle Removal by Bubble Flotation in a Mechanically Stirred Vessel: *Lifeng Zhang*[1]; Shoji Taniguchi[1]; [1]Tohoku University, Taniguchi Lab., Dept. of Metallu., Aoba-ku, Sendai 980-8579 Japan

In this paper, firstly the 3D single-phase turbulent steady fluid flow in a vessel with four baffles stirred by a single impeller is mathematically simulated. Turbulence is modeled by using the standard model. Sliding-grid method is used to model the impeller. The flow domain is divided into two cylindrical, the inner one rotates at the same speed as the impeller, and the outer one is fixed with the baffles. Results indicate that the high-speed zone is near the paddles, and larger stirring speed generates larger fluid flow velocity. The calculated stirring intensity data are used to analyze the experimental results. Secondly the effects, such as filter pore size, gas flow rate and NaCl concentration, on bubble size are studied by experiment. For the particle removal rate by bubble flotation in this vessel, first order kinetics is adopted. The effects of initial number of the particles, gas flow rate, particle and bubble size, stirring speed and surface condition, on the removal rate constant are discussed, and an empirical equation is derived by experimental data. At last, a simple model is developed to study the particle attachment probability on bubble surface under turbulent flow conditions.

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Study on Mass Transfer Characteristic between Molten Steel and Particles in RH-PTB Refining: *Ji He Wei*[1]; Miao Wang[1]; [1]Shanghai University, Dept. of Metallic Matls., 149, Yan Chang Rd., Shanghai 200072 PRC

The mass transfer characteristic between the powder particles and the liquid steel in the RH-PTB (Powder Top Blowing) refining was investigated in a hydraulic model of 1/5 scale for a 90t RH degasser. The sodium chloride powder with chemical purity was used as the flux for blowing. The mass transfer coefficients of the liquid side were determined under the conditions of PTB. The influences of the main technological parameters on the mass transfer rate were examined. The results indicated that the mass transfer coefficient on the liquid side increases with raising the gas blowing rate, internal diameter of the up-snorkel, circulation rate of liquid and powder size, and decreases with an increase of the internal diameter of the down-snorkel. It was in the range of (1.36-7.30)x10(-4) m/s under the conditions of the present work. The appropriate dimensionless correlations were obtained.

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Mathematical Simulation of Fluid Flow in Gas-Stirred Liquid Systems: *Lifeng Zhang*[1]; Kaike Cai[2]; Shoji Taniguchi[1]; [1]Tohoku University, Taniguchi Lab., Dept. of Metallu., Aoba-ku, Sendai 980-8579 Japan; [2]University of Science and Technology Beijing, Steel. Instit., Schl. of Metallu., Beijing 100083 PRC

In the present paper, based on the two-phase (Eulerian-Eulerian) model, the 3D fluid flows in gas-stirred systems, i.e., air-stirred water vessel and argon-stirred liquid steel ladle, are simulated. In the Eulerian-Eulerian two-phase model, gas and liquid are considered to be two different continuous fields. The phases are assumed to share space in proportion to their volume fractions. The exchange between the phases is represented by source terms in conversation equations. Turbulence simulated by the model is assumed to be the property of the liquid phase. The following effects of mathematical treatments and operational factors on the fluid flow are discussed, such as interphase drag force, turbulent model, the size of bubble, and gas injection mode. Some interesting results are derived. Except the interphase drag force, the interphase lift force should be taken into accounted in order to exactly simulate the fluid flow in gas-stirred systems. Suitable turbulent model and drag coefficient have effect on the mathematical simulation. Injecting small bubbles can realize a well mixing flow condition. The distance between the two gas-injection nozzles has effect on the fluid flow.

5:10 PM
A Kinetic Model of Desulfurization by Powder Injection and Blowing in RH Refining of Molten Steel: *Ji-He Wei*[1]; Shou-Jun Zhu[1]; Neng-Wen Yu[1]; [1]Shanghai University, Dept. of Metallic Matls., 149, Yan Chang Rd., Shanghai 200072 PRC

The desulfurization process by injection and blowing in the RH refining of molten steel and its mechanism were considered and analyzed. Based on the two-resistance mass transfer theory and the mass balance of sulfur in the system, a kinetic model for the process was developed. The related parameters of the model, including the mass transfer coefficients, the effective powder amount in the molten steel being treated for desulfurization, were more reasonably determined. Predicting and modeling for the process by injecting and blowing the lime-based powder flux under the assumed operating modes with the different initial contents of sulfur and amounts of powder injected and blown in a RH degasser of 300t capacity were carried out using the model. The relevant circulation rate of the liquid steel and the powder injection and blowing rate were taken to be 100 t/min and 150 kg/min, respectively. The initial contents of sulfur in the liquid steel to be treated and the amounts of powder injection and blowing were respectively assumed to be 0.007, 0.006, 0.005, 0.004, 0.003, 0.002 mass-% and 10, 8, 6, 5, 4, 3 kg/t-steel. The total treatment time for desulfurization under each mode was set up to be 24 min, that is equivalent to eight circulation cycles of the liquid steel to be treated. The results indicated that the prediction made with this model is in good agreement with some data of the industrial experiments and production practice. Injecting and blowing the lime-based powder flux with the chemical composition of 85 mass-% CaO + 15 mass-% CaF_2 of 3-5 kg/t-steel, it is entirely possible to decrease the sulfur content in the molten steel to the ultra-low level below (5-10)x10-4 mass-% from (60-80)x10-4 mass-%. The total treatment time needed will appropriately be 12-20 min. Intensifying the powder injection and blowing operation and increasing the circulation rate of the liquid steel may effectively raise the rate of the process in the RH refining. The model may be expected to provide some useful information and a reliable basis for determining the reasonable technology parameters and optimizing the technology and process of desulfurization by powder injection and blowing in the RH refining of molten steel.

Packaging & Soldering Technologies for Electronic Interconnects: Interfacial Reaction and Reliability of Solder Joints

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

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

Tuesday PM — Room: Lincoln D
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: D. Frear, Intel; Srini Chada, Motorola, Plantation, FL 33322 USA

2:00 PM Invited
Microstructures and Fatigue Resistance of Electroless-Ni/Solder Interfaces: *Pilin Liu*[1]; Zhengkui Xu[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

Electroless-Ni is widely used as a diffusion barrier against the rapid metallurgical reaction between solder alloys and copper metallization in both flip-chip and BGA packages. The interfacial microstructures developed between electroless-Ni solder alloys are complicated not only by the reaction of Sn and Ni, but also by the phosphorous dissolved in the nickel. In this work, the changes in the interfacial microstructure following reflow and aging, and the resulting fatigue resistance of the Ni-solder interfaces were studied by transmission electron microscopy and interface-fracture mechanics techniques. The results showed that phosphorous had a strong influence on both the interfacial microstructure and interfacial fatigue resistance. The effects were highly dependent on phosphorous concentration in the nickel and on thermal processing conditions. The potential for improving interfacial fatigue resistance by reducing P-concentration will be discussed.

2:25 PM
Under Bump Metallizations for Lead Free Solders: *Tia M. Korhonen*[1]; Peng Su[1]; Matt A. Korhonen[1]; Che-Yu Li[1]; [1]Cornell University, MSE, 356 Bard Hall, Ithaca, NY 14853 USA

In order to use lead free solders in flip chip bonding, compatible underbump metallizations (UBMs) are needed. To obtain good adhesion, the wettable layer of the underbump metallization, which is usually Cu, must have sufficient resistance to reaction with the solder. Most commonly used lead-free solders, such as eutectic Ag-Sn and Bi-Sn, contain large amounts of tin, so that the Sn-Cu reaction during the reflow is very intense and can deplete the UBM of copper, which causes dewetting and failure of the joint. Since Sn-Ni reaction is slower, Ni is a viable alternative as the wettable layer. Another approach is to use a CuNi alloy. In this study, UBM's with different CuNi alloys were fabricated and reflowed with lead-free solders. The solder/UBM interfaces were analysed with SEM to find out how the Ni-concentration affects the reaction, and how much Ni is needed to obtain a sufficiently slow reaction rate. Shear tests were also performed to assess the reliability of the joints.

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Interfacial Reaction and Solder Joint Reliability of Sn-3.5Ag, Sn-3.3Ag-0.7Cu Solders in Lead Frame Chip Scale Package (LF-CSP): *Seung Wook Yoon*[1]; Sung Hak Hong[1]; Yoon Hwa Choi[1]; Chang Jun Park[1]; *Jong Tae Moon*[1]; [1]Hyundai Electronics Corporation, Memory Rsch. Div., Ichon, Kyoungki-do 467-701 Korea

To evaluate the Pb-free solders in application of CSP packages, Sn-3.5Ag and Sn-3.3Ag-0.7Cu solders were studied in the fields of interfacial reaction and solder joint reliability. Various substrates were prepared by electroplating as Ni, Sn/Ni, Ag/Ni on Cu alloy and with electroless Au/Ni/Cu substrate. Each jointed samples were characterized after environmental tests such as temperature cycles (-65°C~150°C) and high temperature storage test (150°C). Their fractured surface, microstructure of solder joint interface and of bulk solder ball were examined and analyzed by optical microscopy, XRD, SEM and EDX. The different types of reflow profile were investigated to find out the optimum reflow condition and the specimens prepared by higher reflow temperature showed the superior solder joint strength. The solder joint strength and the microstructural change were observed with number of reflow cycle in considering the real board mounting, too. To simulate the real surface mounting condition, Sn-3.5Ag and Sn-3.3Ag-0.7Cu balls were attached on LF-CSP packages and each components were placed on PCB board, then temperature cycle test was performed. To compare the solder joint reliability, Sn-36Pb-2Ag solder ball was also applied. After T/C test (-65°C~150°C) of PCB board level mounting, interfacial reaction and microstructure such as crack initiation site and crack propagation, were investigated. Also the Daisy electrical test PCB board was prepared and evaluated the solder joint reliability with T/C test.

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Effects of Solder Reflow on Wettability, Microstructure and Mechanical Properties: F. Guo[1]; S. Choi[1]; *J. P. Lucas*[1]; K. N. Subramanian[1]; [1]Michigan State University, Matls. Sci. & Mech., 3536 Eng. Bldg., East Lansing, MI 48824-1226 USA

Solder joints used in electronic applications undergo reflow operations. Such operations can affect the solderability, interface intermetallic layer formation and the resultant solder joint microstructure.

These in turn can affect the overall mechanical behavior of such joints. In this study the effects of reflow on solderability and microstructure were studied. These studies were carried out with Sn-Ag solders, with or without Cu or Ag reinforcements, using Cu and Cu-Ni-Au substrates. Mechanical properties were carried out on joints made with the same solders using copper substrates.

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BGA Underfill Reliability for Underhood Automotive Electronics: *Ping Kwong Seto*[1]; John Evans[1]; Wayne Johnson[2]; [1]Daimler Chrysler Corporation, Huntsville Elect. Div., 100 Electronics Blvd., Huntsville, AL 35824 USA; [2]Auburn University, 200 Broun Hall, Auburn, AL 36849 USA

As the use of Ball Grid Array (BGA) packages proliferate, the demand for packaging density and cost savings is driving automotive electronics towards smaller form factor BGA to be used in the underhood environment. In order to ensure the solder joint integrity of the BGA components are not compromised due to this miniaturization, DaimlerChrysler Huntsville Electronics (DCHE) and Auburn University have embarked on a study to use BGA underfill to achieve the reliability goal. Since the use of underfill for flip chip has been demonstrated to be very effective in enhancing solder joint life. In this multiple phases study, DCHE is evaluating the feasibility of using underfill to extend the BGA solder joints while still meeting the aggressive manufacturing cycle time. The reliability impact of various printed circuit board (PCB) finishes and pad geometries in thermal cycling performance are also assessed. BGA packages ranging from 27mm to 15mm body sizes are included in the study.

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4:00 PM Invited

Interfacial Microstructure and Mechanical Reliability of Sn-Ag Solder Joint on Electroless Ni-P Film: *Yoshiharu Kariya*[1]; Kumiko Nakamura[1]; Yasunori Tanaka[2]; Masahisa Otsuka[1]; [1]Shibaura Institute of Technology, Dept. of Matls. Sci. and Eng., Shibaura 3-9-14, Minato-ku, Tokyo 1088548 Japan; [2]NEC Corporation, Mobile Comm. Div., Ikebe-cho 4035, Tsuzuki-ku, Yokohama, Kanagawa 224 Japan

Packages of the area array mounting type which are suitable for high-density mounting are receiving attention, and the production volume of fine pitch BGA (ball grid array)-type packages is consequently increasing. With diminishing solder joint sizes, characteristics of interfacial microstructure which affect the mechanical reliability of the joins are an issue that can not be neglected. Recently, electroless Ni-P/Au plating has been used for surface finishes of substrate or electrode to meet a demand on mounting technology of fine pitch BGA. However, BGA solder joint on electroless Ni-P plating often exhibits poor mechanical reliability because brittle interfacial reaction layers which are formed during soldering process. Therefore, understanding the interfacial microstructure between solder and electroless Ni-P plating and its effect on the mechanical reliability is required in order to develop highly reliable solder joints. In this paper, the interfacial microstructure between Sn-Ag eutectic and electroless Ni-P plating has been characterized using SEM and TEM. The effect of interfacial microstructure on the mechanical reliability of actual CSP (chip size package) will also be presented.

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Mechanical Behaviour of Transfusion Bonded Joints: *Tommi O. Reinikainen*[1]; Roope Nikander[2]; Jorma K. Kivilahti[2]; [1]Nokia Research Center, Nokia Grp., P.O. Box 407 FIN-00045 Finland; [2]Helsinki University of Technology, Lab. of Elect. Prod. Tech., P.O. Box 3000, TKK FIN-02015 Finland

A recently introduced novel joining method-Transfusion Bonding (TFB)-allows fluxless Pb-free microjoining by utilising metallurgically compatible low melting point metals being bicoated chemically or electrochemically (/1/). Essentially, it combines the benefits of intermetallic- and paste-free joining and bonding, and therefore it can be used also for very fine-pitch electronics assembly. This type of joining and bonding technique is becoming ever more important, when thinner diffusion barrier or adhesion layers, coated overlayers and especially smaller solder joint volumes are encountered in very high density electronic assemblies. Moreover, since there are only a limited number of new viable eutectic alloy candidates for soldering applications, the TFB technique being based on the formation of Sn-rich solid solution joints can make use of more new metallurgical options for tailoring low temperature interconnection materials. If the undercoating is pure Sn or Sn-based alloy and the topcoating is pure Bi, the bonding occurs well below 200°C and the resulting microstructure of the joints is essentially dilute Sn[Bi] solid solution, the concentration of Bi being originally somewhat higher in the middle of the joint than near the component lead or substrate interfaces. Dissolved Bi has a very strong hardening effect on Sn (/2/). However, if the amount of Bi exceeds significantly the solubility limit, about 3.5 wt-% at room temperature, pure Bi will precipitate at the grain boundaries; at low supersaturation bismuth tends to precipitate as globular particles while at the higher supersaturations it will precipitate discontinuously along the grain boundaries. In the latter case the reaction product has an embritteling effect on the joints. In this work the mechanical behaviour of several dilute Sn[Bi] solid solutions have been studied, and respective material models and parameters were assessed and implemented in a finite-element program. The effect of the Bi distribution in the solder joint is analysed by the FEM and the joining process parameters are modified accordingly to achieve optimum reliability. The simulation results are verified by experiments which are conducted by shear tests with dimensions similar to real solder joints (/3/). 1. J.Kivilahti and K.Kulojärvi, 'A New Reliability Aspect of High Density Interconnections', The Proc. of Design and Reliability of Solders and Solder Interconnections", TMS Annual Meeting, Orlando, 9-13 February 1997, USA, pp. 377-384. 2. T. Reinikainen and J. Kivilahti, ´Deformation Behaviour of Dilute SnBi(0.56at%) Solid Solutions´, Metallurgical and Materials Transactions A, Vol. 30A, January, 1999, pp. 123-132. 3. T. Reinikainen, M. Poech, M. Krumm and J. Kivilahti, ´A finite-element and experimental analysis of stress distribution in various shear tests for solder joints´, Trans. ASME J. Elec. Pack., Vol. 120, March, 1998, pp. 106-113.

4:45 PM Invited

The Effect of Solder Paste Residues on RF Signal Integrity: *Laura J. Turbini*[1]; James Brokaw[1]; John A. Williams[1]; Juergen Gamalski[2]; [1]Georgia Institute of Technology, Matls. Sci. and Eng., 778 Atlantic Dr., Atlanta, GA 30332-0245 USA; [2]Siemens AG, Dept. ZT ME6, Siemensdamm 50, Berlin D-13623 Germany

Wireless devices such as pagers and cellular phones are becoming common consumer items. These products require low loss RF signal propagation which is affected by material choices and processing conditions. This paper examines the effect of a series of no-clean solder pastes on signal integrity using an RF test circuit which sends a broadband RF signal through a gallium arsenide antenna switch and measures its transmission using a network analyzer. The test circuit also measures signal leakage. This paper reports on two different test vehicles, one that used a 900 MHz antenna switch, and the other that used a 2.0 GHz antenna switch. The transmission and leakage readings were taken daily for 20 days while the test vehicles were under accelerated aging conditions of 85°C and 85% RH. Average values for the readings for each solder paste were plotted to provide comparison among the pastes. The comparison data clearly distinguish solder pastes that provide consistency throughout the test period from those which do not.

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Microstructure and Interfacial Characteristics of Electronic Interconnects: *F. A. Khalid*[1]; S. E. Benjamin[1]; M. Rashid[1]; [1]GIK Institute of Engineering Sciences, Topi, Nwfp Pakistan

The importance of electronic interconnects have been increased with the advancement in technology due to the miniaturization of electrical components. This has also lead to an increase in the number of input/output terminations. Consequently not only the solder joints have increased but also the joint dimensions have decreased in electronic packages with increased speed and greater packaging density. However, reliability has become crucial because of their use to control operational and safety functions in aerospace and automobile applications. The present work focuses on the examination of microstructural features and their influence on the interfacial properties of substrate using optical, scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). A series of solder alloys with and without lead alloying developed were used to investigate interdiffusion

characteristics and the formation of the intermetallic phases to elucidate interfacial properties of solder alloys.

Pressure Technology Applications in the Hydrometallurgy of Copper, Nickel, Cobalt and Precious Metals: Pressure Technology Applications in the Hydrometallurgy of Copper and Zinc

Sponsored by: Extraction & Processing Division, Copper, Nickel, Cobalt Committee
Program Organizers: James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA; Norbert L. Piret, Piret & Stolberg Partners, Duisburg 47279 Germany

Tuesday PM — Room: Lincoln C
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: Gerry Bolton, Dynatech Corporation, Fort Saskatchewan, Alberta T8L4K7 Canada; James E. Hoffmann, Hoffmann and Associates, Houston, TX 77242 USA

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Chemistry and Mechanisms of High Pressure Acid Leaching (HPAL) Chalcopyrite Flotation Concentrates: *C. J. Ferron*[1]; N. McKay[1]; I. Dymov[1]; D. Butcher[2]; [1]Lakefield Research Limited, 185 Concession St., Postal Bag 4300, Lakefield, Ontario KOL-2110 Canada; [2]General Gold Australia

The feasibility of applying high pressure acid leaching (HPAL) as an alternative to smelting for chalcopyrite concentrates was tested on various concentrate samples from the Guelb Moghrein deposit in Mauritania. Initial bench scale tests on various concentrates confirmed HPAL to be technically feasible. Further bench and pilot plant tests using steady-state recycle streams, revealed the significant impact of gangue materials in the concentrate, in particular magnesium minerals on the leach process. Kinetic sampling during the pressure leach indicated an initial copper precipitation, followed by dissolution. Possible mechanisms of chalcopyrite dissolution during HPAL are briefly discussed.

3:00 PM
The Treatment of Chalcopyrite with Nitrogen Species Catalyzed Oxidative Pressure Leaching: *Corby G. Anderson*[1]; [1]Montana Tech., The Ctr. for Adv. Min. and Metallu. Process., Rm. 221, LLC Bldg., Butte, MT 59701 USA

Today, with a stringent economic and environmental climate prevailing in the copper business, there is increased interest in evaluating new processing alternatives for production. Hydrometallurgical pressure oxidation of copper concentrates is one of the more viable approaches and several technological candidates have emerged. Of these, an overlooked but, ironically the first industrially proven methodology, utilized nitrogen species catalyzation in the oxidizing pressure leach system to produce copper via SX/EW. This may prove to be a feasible process alternative for the future. In this paper, the history of the system and its application to chalcopyrite concentrates will be outlined. In particular, a novel methodology for effective recovery of precious metals from chalcopyrite concentrates will be discussed. Finally, the perceived economics of this unique industrially proven process will be delineated.

3:30 PM
Pressure Acid Leaching of Zinc and Copper Concentrates by Dynatec: K. R. Buban[1]; *M. J. Collins*[1]; I. M. Masters[1]; J. Stiksma[1]; [1]Dynatech Corporation, Metallu. Tech. Div., Fort Saskatchewan, Alberta T8L4K7 Canada

Pressure leaching of zinc sulphide concentrates has been practiced commercially for nearly two decades. The process, which includes direct leaching of concentrate with spent electrolyte in an autoclave, has been commercialized at four separate locations to date. In pilot studies carried out in the Dynatec lab in Fort Saskatchewan, dozens of additional zinc concentrates have been shown to be amenable to pressure leaching. This paper highlights recent improvements in zinc pressure leaching that have been identified through process development studies, and how the experience gained in these studies has been utilized in the development of a new copper pressure leach process.

4:00 PM Break

4:15 PM
Pressure Leaching of Copper Refinery Slimes: *Bradford C. Wesstrom*[1]; [1]Phelps Dodge Refining Corporation, 6999 N. Loop Rd., El Paso, TX 79915 USA

The extraction of metals from Copper Refinery Slimes may involve many different processes. This paper describes the use of pressure leaching for decopperizing of copper refinery slimes at the El Paso Refinery. Process equipment will be described, along with the process chemistry, unit operations and unit processes associated with the decopperizing process. Copper contained in the raw slime ranges from 15-35%. After autoclaving less than 1% copper remains.

4:45 PM
Outokumpu Process for the Precious Metal Refining from Copper Anode Slime: *Olli Jarvinen*[1]; [1]Outokumpu Wenmec Oy, P.O. Box 103, Riihitontuntie, 7 E 02200 Espoo

The capacity of the Outokumpu Pori refinery is today about 125 000 mt in 1998. The anode slime amount is about 700-800 mt. The copper is removed with pressure leaching. Selenium roasting is done with the gas roasting. The doré smelting in the furnace is done with the tilting rotating converter with oxy-fuel burner (TROF). Doré anodes are treated in the silver electrolysis. Gold is recovered from the gold mud with the quick hydrochloric acid leaching process. The important point in the leaching is to get into contact the solid copper with the oxygen and liquid. Outokumpu has developed an efficient agitator for this purpose. The GLS-agitator (GLS gas, liquid and solid) is powerful for mixing oxygen, liquid and solid together.

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

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

Tuesday PM — Room: Knoxville B
March 14, 2000 — Location: Opryland Convention Center

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

2:00 PM
Effect of Grain Size and Strain Rate on Room Temperature Mechanical Properties of a TiAl Intermetallic Alloy: M. R. Shagiev[1]; A. V. Kuznetsov[1]; *G. A. Salishchev*[1]; R. M. Imayev[1]; O. N. Senkov[2]; F. H. (Sam) Froes[2]; [1]Institute for Metals Superplasticity Problems, Russian Acad. of Sci., Ufa 450001 Russia; [2]University of

Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA

Gamma TiAl alloy samples with grain sizes varying from 0.4 µm to 17 µm were produced by an isothermal forging in the temperature range 800-1000°C followed by annealing at 770-990°C. Tensile mechanical properties of the specimens were determined at room temperature and microstructures before and after deformation were analyzed using TEM. Peak ductility of 6.9% was achieved at a strain rate around 0.1 s^{-1} and grain size of 8 µm. The increased ductility was accompanied with development of single-system deformation twins. Activation of multiple twinning in specimens with larger grain sizes and suppression of the twin development in specimens with the grain size below 1 µm led however to considerable decrease in ductility. The yield stress versus grain size followed the Hall-Petch relation with the Hall-Petch parameter k_y=0.4 MPa $m^{0.5}$, that is the dependence was about 5 times weaker as compared to the coarse-grained material.

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High Temperature, High Strain Deformation of Coarse-Grained Beta-CEZ Titanium: *Henry J. Rack*[1]; C. R. Robinson[1]; [1]Clemson University, Cer. and Matls. Eng., 110 Olin Hall, Clemson, SC 29670-0907 USA

The high temperature, high strain deformation behavior of coarse grained Widmanstatten Beta CEZ (Ti-4.8Al-2Sn-3.8Zr-3.8Mo-1.9Cr-1Fe-0.045O) has been investigated between strain rates of 1 to 2x10-4 s-1 at temperatures between 1013 and 1138K. Under these conditions the compressive stress-strain response exhibited an initial maximum followed by flow softening with increasing strain. At the lowest temperatures and highest strain rates examined this flow softening behavior was followed at the highest strains examined by strain hardening. Dynamic material modeling indicated that stable flow and therefore optimal deformation conditions can be expected above strain rates of 10-2 s-1 at temperatures between 1025 and 1100K. This presentation will discuss how establishment of this stable flow regime involves a balance between grain boundary associated deformation, resolution of the phase, dynamic spheroidization of the grain boundary and lamellae, dynamic recovery and recrystallization of lamellae and dynamic grain growth. Stable flow was associated with dynamic recovery and recrystallization of the constituent and phases, while unstable flow at the lowest temperature examined, 1013K, was associated with shearing of grain boundary and void formation at/interfaces, the former predominating at high strain rates, the later at the lower strain rates examined. With increasing deformation temperature void formation was progressively replaced by localized flow within the grain boundary region incorporating dynamic spheroidization, dissolution of the phase and dynamic recrystallization of the phase. Finally at the highest temperatures and strain rates, unstable flow was associated with flow localization and kinking of the lamellae within the near-grain boundary region.

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Low-Temperature Superplasticity of TiAl and Ti3Al Based Alloys: *R. M. Imayev*[2]; G. A. Salishchev[2]; V. M. Imayev[2]; M. R. Shagiev[2]; N. K. Gabdullin[2]; O. N. Senkov[1]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]Institute for Metals Superplasticity Problems, Russian Acad. of Sci., Ufa 450001 Russia

The data on superplastic behavior of TiAl and Ti3Al intermetallic alloys with a high ordering energy and submicron grain size are summarized. By decreasing grain size from 10 ìm to 0.1 ìm, the superplastic temperatures were decreased from 1000-1200°C to 600-900°C and the strain rate range was substantially extended towards the higher strain rates. The effects of composition, superlattices type, and grain size on low-temperature superplastic properties of titanium aluminides was investigated. An increase in the volume fraction of the Ti3Al phase in TiAl based alloys led to improvements in the superplastic properties such as ductility and strain rate sensitivity and more homogeneous deformation. The activation energies of superplastic flow were determined to be close to the activation energy of grain boundary diffusion. The considerable decrease in superplastic forming temperatures opens possibilities in cost reduction by using less expensive tools and deformation processes.

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The Influence of Solution Treatment Temperature on Martensitic Phase Transformations in IMI 550: *Henry J. Rack*[1]; Krishna Kharia[1]; [1]Clemson University, Cer. and Matls. Eng., 110 Olin Hall, Clemson, SC 29670-0907 USA

The influence of solution treatment temperature on the martensitic phase transformations observed in IMI 550(Ti-4Al-4Mo-2Sn-0.5Si) has been investigated. When solution treated of equilibrated samples is conducted at temperatures of above 960°C followed by rapid cooling a hexagonal martensite, δ', is formed. However at temperatures between 850 and 960°C, rapid cooling results in the formation of orthorombic, δ". Finally below this temperature region the β phase is stable-no martensitic transformation occurring on rapid cooling. This transition from $\delta' \; \delta_{primary} + (\delta' + \beta_{retained}) \; \delta_{primary} + (\delta'' + \beta_{retained}) \; \delta_{primary} + \beta_{metastable}$ with decreasing solution treatment temperature will be discussed by considering the effect of solution treatment temperature on alloy partitioning and the influence of this partitioning on phase stability as revealed by x-ray, transmission electron microscopy and elevated temperature neutron diffraction.

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Characterization of a Gamma-TiAl Sheet Produced from Blended Elemental Powders: *Javaid I. Qazi*[1]; Oleg N. Senkov[1]; Francis H. Froes[1]; Valadimir S. Moxson[2]; [1]University of Idaho, IMAP, Mines Bldg. Rm. # 321, Moscow, ID 83844-3026 USA; [2]ADMA Products Inc., 8180 Boyle Park Way, Twinsburg, OH USA

Gamma titanium aluminide sheet of a nominal composition of Ti-46.5Al-2Cr-3Nb-0.2W was produced from blended elemental powder. A novel loose sintering approach combined with hot rolling at temperatures near alpha-transus temperature was used. The sheet was characterized using X-ray diffraction, optical microscopy and TEM. A fully lamellar structure was produced with a colony size of around 100mm. The material consisted of two phases: gamma-TiAl and about 20% of Ti3Al. The porosity level was about 2%. Mechanical properties of the sheet were also studied.

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Ductility of a Ti3Al Intermetallic: Effect of Grain Size and Partial Disordering: *R. M. Imayev*[2]; N. K. Gabdullin[2]; O. N. Senkov[1]; G. A. Salishchev[2]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]Institute for Metals Superplasticity Problems, Russian Acad. of Sci., Ufa 450001 Russia

The effect of grain size and partial disordering on ductility and flow stress of an intermetallic Ti3Al was studied in the temperature range of 20 to 800°C using tension and compression testing. The ductility of the fully ordered material increased considerably when the grain size decreased from 27 µm to 0.1 µm, and, at the smaller grain size, an elongation of 4.8% was achieved at room temperature. Partial disordering of the crystal lattice led to a decrease in ductility at temperatures below 500°C but had no influence on ductility at higher temperatures. The critical grain size at which a brittle-to-ductile transition occurred in the fully ordered material was determined for each temperature studied. This critical grain size increased as the temperature increased. Dislocation slip changed from a localized planar mode to a more uniform fine slip, the slip line spacing decreased, cross-slip developed, and relaxation capability of grain boundaries enhanced when the grain size decreased below the critical value. The fracture mode changed from a brittle transcrystalline mode to a brittle intercrystalline mode and, finally, to a ductile mode when the grain size decreased and temperature increased. Transcrystalline fracture was observed in specimens with grain sizes above the critical grain size, while intercrystalline and ductile fracture modes occurred in specimens with grain sizes below the critical grain size. Specimens with submicron sized grains exhibited features of superplastic flow at 600°C and above.

Research and Development Efforts on Metal Matrix Composites: New Directions in MMC Research

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

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

Tuesday PM Room: Bayou A
March 14, 2000 Location: Opryland Convention Center

Session Chairs: John J. Lewandowski, Case Western Reserve University, Matls. Sci. and Eng., Cleveland, OH 44106 USA; Benji Maruyama, US Air Force, Air Force Rsch. Lab., Wright Patterson AFB, OH 45433 USA

2:00 PM Invited

Directions for Research in Discontinuously-Reinforced MMC's: *Daniel B. Miracle*[1]; Benji Maruyama[1]; [1]AF Research Laboratory, Matls. and Manu. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA

A great deal of progress has been made in the past three decades in the understanding, development, processing and implementation of discontinuously-reinforced MMC's. Significant markets are now established in the military and commercial aerospace industry, as well as automotive, electronics and recreation product sectors. However, dramatic additional benefits in affordability, performance and supportability are expected from more widespread application of this pervasive metals technology. The results of a recent analysis of potential applications in the aerospace industry will be provided. Suggestions for research that will be required to support the development of MMC's for these applications will be provided and discussed.

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New Directions in the Characterization of Composite Microstructures: *Jonathan Edward Spowart*[1]; [1]Air Force Research Laboratory, Matls. Direct., AFRL/MLLM Bldg. 655, 2230 Tenth St., Ste. 1, Wright-Patterson AFB, OH 45433 USA

Previous experimental and theoretical work has shown that there is strong correlation between the fracture toughness of discontinuously-reinforced metallic matrix composites and the spatial arrangements of the reinforcing particles. However, until recently this aspect of the composite microstructure has received little attention. Previous attempts to characterize particle distributions in composite microstructures have concentrated on the statistics of the particle-particle spacings. These approaches, based either on Radial Distribution Functions (RDF's) or tessellation schemes, reveal only limited information about the spatial arrangements of individual particles. In this paper, new characterization techniques are demonstrated whereby the inter- particle connectivity is given priority over inter-particle proximity. The importance of the connectivity of potential damage sites is discussed in terms of crack propagation phenomena. The analysis allows multi-scale clustering parameters to be defined, using fractal geometry techniques. Results are given both for experimentally obtained and artificially simulated microstructures. Cellular automata-based crack propagation simulations reveal preferential paths for crack propagation in clustered microstructures, and show strong correlations between the degree of particle clustering and selected crack path metrics. Using crack propagation simulations, fracture toughness predictions are obtained from the microstructures of two different PM-processed SiC- reinforced 7093Al materials with contrasting particle spatial distributions, following the approach of Rice and Johnson. There is good agreement between the predictions and the experimentally-obtained fracture toughness values for these materials.

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Development of a New Discontinuously Reinforced Aluminum MMC: *Aaron C. Hall*[1]; [1]The University of Illinois, Dept. of Matls. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA

A new metal matrix composite based on high aspect ratio aluminum diboride flakes (diameter/thickness > 100) has been developed. This new material is a discontinuously reinforced aluminum composite in which AlB2 flakes that are prepared in situ act as the stiffening phase. In situ preparation of the AlB2 reinforcement simplifies processing making this composite a low cost material (~ $5.00/kg). Target applications include automotive brake rotors and turbine components where a combination of excellent wear resistance and high specific properties are required. Mechanical testing and simulation of braking conditions using a laboratory scale dynamometer suggest promise for this material. Current development efforts are focused on elucidating low cost synthesis routes that will allow preparation of high aspect ratio AlB2 in the absence of AlB12 and other borides. As part of this effort the growth of AlB2 in liquid aluminum has been extensively studied. It has been found that the rate at which a liquid aluminum boron alloy is cooled dramatically affects the aspect ratio of the resulting AlB2. In addition, the aluminum rich region of the aluminum boron phase diagram has been reexamined. The peritectic temperature has been found to be significantly lower than that presented in the literature.

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In-Situ Ductile Metal/Bulk Metallic Glass Matrix Composites Formed by Chemical Partitioning: *Choongnyun Paul Kim*[1]; Chuck C. Hays[1]; William L. Johnson[1]; [1]California Institute of Technology, Matl. Sci., W.M. Keck Lab. of Eng., Mail Stop 138-78, Pasadena, CA 91125 USA

A new class of ductile metal reinforced bulk metallic glass matrix composite material have been prepared that demonstrate improved mechanical properties. This newly designed material exhibits both improved toughness and large plastic strain to failure. The remarkable glass forming ability of bulk metallic glasses allows for the preparation of ductile metal reinforced composites with a bulk metallic glass matrix via in-situ processing; i.e. chemical partitioning. The incorporation of a ductile metal phase into a metallic glass matrix yields a constraint that allows for the generation of multiple shear bands in the metallic glass matrix; this stabilizes crack growth in the matrix and extends the composite strain to failure. Specially, by control of chemical composition and processing conditions a stable two-phase composite (ductile metal in a bulk metallic glass matrix) is obtained on cooling from the liquid state. The crystalline beta-phase has a dendritic morphology with a particle size and periodicity that is determined by the chemical composition and processing conditions. These ductile metal particles impose intrinsic geometrical constraints on the bulk metallic glass matrix that leads to the generation of multiple shear bands under mechanical loading. Sub-standard Charpy specimens prepared from this new composite material have demonstrated Charpy impact toughness numbers that are 250% greater than that of the bulk metallic glass matrix alone. Bend test have shown large plastic strain to failure values of ~4%. Specimens tested under compressive loading exhibit large plastic strains to failure on the order of 8%. Another key factor to the improved behavior is the quality of the interface between the ductile metal beta-phase and the bulk metallic glass matrix is chemically homogeneous. This clean interface allows for stable deformation and for the propagation of shear bands through the beta-phase particles. X-ray diffraction, microstructural, and mechanical property results are presented for a ductile metal reinforced bulk metallic glass matrix composite based on bulk glass forming composites in the Zr-Ti-Cu-Ni-Be system.

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Metal Matrix Composites Based on Aluminum and Optical Fibers: *Ferdinando Felli*[1]; Antonio Paolozzi[2]; Michele A. Caponero[3]; [1]University of Rome "La Sapienza", Dept. ICMMPM, Via Eudossiana 18, Roma 00184 Italy; [2]University of Rome"La Sapienza", Dept.

Aerospaziale, Via Eudossiana 18, Roma 00184 Italy; [3]Enea Frascati Research Center, Via Enrico Fermi 46, Frascati, Roma 00044 Italy

Embedding optical fiber into structural components is a very promising technology for real time health monitoring. Recently the interest for producing low cost and reliable sensor is increased in the mechanical, civil and aerospace engineering community. Aluminum and polyimide coated optical fibers have been successfully embedded in different aluminum alloys by using both cast and colamination techniques. The studied samples have been subjected to the following tests: mechanical tests in order to verify the structural integrity; optical transmission tests to highlight the fiber integrity after embedding process; metallographic observation of the fiber-matrix interface; interferometric tests in order to verify that the embedded optical fiber can be usefully used as a global sensor for measuring variation of the physical properties of the hosting material. The possibility of embedding optical fibers in aluminum alloys has been demonstrated. The obtained aluminum composites show interesting performances.

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Creep and Stress Rupture Behavior of Be/Al Composite Materials: *Shihong Gary Song*[1]; J. T. Beals[1]; V. C. Nardone[1]; [1]United Technologies Research Center, 411 Silver Ln., Hartford, CT 06108 USA

Aluminum-beryllium composites are known for their ultra lightweight and exceptionally high specific stiffness and have found broad applications as static and low-stress components in aerospace industry. An increasing interest in the alloys has recently been seen for dynamic, high-temperature, and high-stress applications for aircraft and engine components. However, there is an apparent scarcity in literature of the property data of these alloys, in particular, the secondary mechanical property data such as creep, fatigue, and fracture toughness. The present investigation concerns the time dependent deformation and rupture behavior at elevated temperatures of a commercial aluminum-beryllium alloy "AlBemet 162". The creep and stress rupture properties of the alloy were measured and discussed in comparison with that of ceramic particulate reinforced Al matrix composites. Fractography of the specimens were conducted and analyzed in connection with the measured creep and stress rupture properties.

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Microstructure Control of High Strength High Conductive Cu Based In-Situ Composites: *Hirowo G. Suzuki*[1]; K. Mihara[2]; S. Sakai[2]; K. Adachi[3]; [1]National Institute for Metals Japan; [2]Furukawa Electric Company Limited; [3]Sumitomo Metal Industries Limited

Development of high strength high electrical conductive Cu based in-situ composites is potentially useful in the electric and electronic industries. Microstructure control is useful to obtain high strength and high conductivity. Based on the Cu-15wt%Cr binary alloy, the effects of microalloying elements such as Zr, Fe and Sn were examined. Processing involves ingot making by induction heat melting, hot stage forging, solution treatment, cold rolling and aging. Heavy cold rolling is effective to refine the lamellar spacing of second phase (Cr fiber) and aging treatment gives precipitation strengthening and recovery of electrical conductivity. Dynamic recrystallization occurs in the copper matrix during heavy cold rolling more than h=5.0 (here, h=lnA0/A) in a Cu-15wt%Cr binary alloy, while banded structure is formed and higher strength is attained in the Cu-15wt%Cr-0.2wt%Zr alloys, indicating the difficulty of recovery. Age hardening is remarkable in this alloy due to the acceleration of Cr precipitation. The addition of 0.2 to 0.5wt% of Fe does show work softening. Fe is scavenged to the second phase of Cr during solidification. The effect of Sn is similar to that of Zr although the electrical conductivity drops more than the case of Zr. The tensile strength follows Hall-Petch type equation with lamellar spacing of Cr. Cr phase becomes quite ductile in the Cu matrix. In the conference, the microstructure control is emphasized to optimize the strength as well as electrical conductivity.

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Preparation and Mechanical Properties of Al-MgAl2O4 In-Situ Particle Composites: *P. C. Maity*[1]; S. C. Panigrahi[1]; P. N. Chakraborty[1]; [1]National Institute of Foundry and Forge Technology, P.O. Hatia, Ranchi 834003 India

Metal matrix in-situ particle composites are being developed in recent years with the objective of producing thermodynamically stable fine particulate reinforcements in the matrix having clean interface free from reaction products. In the present work, attempts have been made to produce Al matrix MgAl2O4 particle reinforced in-situ composites by addition of reactive oxide particles in an Al-2 Mg alloy. To prepare Al-MgAl2O4 in-situ particle composites, up to 2 wt% of Fe2O3, Cr2O3 and TiO2 particles were incorporated into Al-2 Mg alloy melt by vortex method. MgAl2O4 and MgO particles formed in all the composites by reaction between the oxide particles and the alloy melt. In addition, pure Fe, Cr and Mg2Ti2O5 also formed in the respective composites. The reaction between the oxide particles and Al-2 Mg alloy melt were nearly complete. The microhardness of the composites improved due to dissolution of the reduced elements such as Fe, Cr etc. to limited extent. The reduction in microhardness in a few composites is associated with the presence of porosity and depletion of Mg from the matrix. Additional factor to increase the hardness of the composites is the dispersion of MgAl2O4 and MgO particles. Porosity resulted in poor tensile strength, except where the microhardness was substantially improved. Elongation % is higher than that of the base alloy for all the composites probably due to depletion of Mg from the matrix and presence of microporosity in the structure.

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Continuous Fiber Metal Matrix Composites at 3M: *Herve Deve*[1]; T. L. Anderson[1]; J. P. Sorensen[1]; S. R. Holloway[1]; [1]3M, Met. Matrix Comp., 3M Center Bldg. 60-1N-01, St. Paul, MN 55144-1000 USA

3M R&D efforts on continuous fiber metal matrix composites will be reviewed. In particular, the development of aluminum/alumina fiber composites wires for high voltage power transmission lines will be discussed.

Surface Engineering in Materials Science I: Coating/Films Characterization (C)-II

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

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

Tuesday PM — Room: Canal B
March 14, 2000 — Location: Opryland Convention Center

Session Chairs: D. L. Cocke, Lamar University, Gill Chair of Chem. and Chem. Eng., Beaumont, TX 77710 USA; L. J. Maksymowicz, University of Mining and Metallurgy, Instit. of Elect., Kraków 30-059 Poland

2:00 PM Invited

High Energy and Spatial Resolution XPS Analysis of Surface-Modified and Heterogeneous Polymers: *Julia E. Fulghum*[1]; [1]Kent State University, Chem. Dept., Kent, OH 44242 USA

Surface analysis of polymers has historically been viewed as being both of particular interest and of special difficulty. XPS is particularly useful in the analysis of polymers since chemical information can be

acquired, frequently with minimal damage to the sample. Recent developments in XPS instrumentation have enhanced the analysts ability to acquire interpretable chemical data from samples of increasing complexity. Along with these developments, however, come increasing demands for analysis of multicomponent or multilayered samples with ever-decreasing feature sizes. This talk will focus on the use of angle-resolved, small area, and imaging photoelectron spectroscopy for the analysis of complex polymer surfaces. The use of derivatization methods to enhance XPS sensitivity will also be discussed. Both core and valence band photoelectron spectra will be used in the evaluation of surface modifications. Lateral and vertical heterogeneities in polymer blend samples will be demonstrated using small area and imaging XPS in correlation with imaging FTIR and phase-contrast AFM. This work has been partially supported by NSF (DMR89-20147), Dow Chemical and 3M.

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Elemental and Chemical Identification of Sub-Micron Metal Precipitates in Silicon Using Synchrotron-Based X-Rays: *Scott McHugo*[1]; [1]Lawrence Berkeley National Laboratory, Adv. Light Source Ctr., Mail Stop 2-400, Berkeley, CA 94720 USA

Metal impurity precipitates in silicon were studied with a focus on the ability to retain metal impurities away from the active device region of integrated circuits by use of oxygen precipitates and their growth-related defects in the bulk of the material. With x-ray absorption spectromicroscopy (m-XAS), we have characterized the chemical state of sub-micron scale Cu and Fe impurity precipitates in silicon. Furthermore, with x-ray fluorescence microscopy (m-XRF), we have studied the dissolution rate of metal precipitates in silicon as a function of thermal treatment. Based on our results, we present theoretical analysis of metal precipitate stability in silicon and discuss the feasibility of metal contamination of device regions.

2:45 PM Invited
Spectroscopic Ellipsometry Measurements of Thin Metal Films: *Harland G. Tompkins*[1]; [1]Motorola Inc., Motorola Labs., Physical Sci. Rsch. Labs., Tempe, AZ 85224 USA

Optical methods are used to determine the thickness of thin metal films, with emphasis on spectroscopic ellipsometry and transmission. We discuss the conditions where this is possible and how to determine the optical constants for the material. The determination of the thickness of each of two metals in a bi-metalic stack is discussed. Finally, by measuring thickness with these methods and measuring weight-gain, we determine the density of platinum deposited by evaporation and deposited by a simple sputter deposition method. The resulting optical constants suggest that the microstructure of films from the two different methods will not be the same and x-ray diffraction and sheet resistance measurements verify that this is the case. Specifically, the significantly lower extinction coefficient of the sputter-deposited films correlates with a higher sheet resistance.

3:10 PM
Gas Adsoption on Highly Diffusing Surfaces of (Sn,Ti)O2 Thin Films: *M. Radecka*[1]; K. Zakrzewska[1]; [1]University of Mining and Metallurgy, Al.Mickiewicza 30, Cracow 30-059 Poland

Recently, it has been demonstrated [1] that (Sn,Ti)O2 thin films prepared by rf reactive sputtering from metallic targets are promising candidates for gas sensing devices. The sensor performance, especially its sensitivity, can be greatly improved by increasing the density of centers active for gas adsorption. Despite a quite well known method such as an appropriate doping or incorporation of catalysts, a simple creation of large surface-to- volume ratio seems to solve this problem. In general, thin oxide films grown in the reactive process, i.e. in the presence of oxygen, have smooth surfaces. The post-deposition annealing enhances the surface roughness only to some extent. However, the post-deposition oxidation of previously grown metal films results in formation of highly diffusing surfaces of (Sn, Ti)O2. This method known as RGTO (rheotaxial growth and thermal oxidation) has proved to be efficient for quite a number of oxide compounds [2]. The key feature of this technique is the substrate temperature higher than the melting point of deposited metal or alloy. In the present work we show that very rough samples could be obtained independently of the substrate temperature as long as the initial state is metallic. The light scattering experiments in the ultraviolet, visible and near-infrared ranges were performed in order to study the surface development. The correlation was found between the film morphology as determined by SEM and the level of the diffused reflection of light. Electrical resistivity measurements in changing gas atmospheres were used to study gas adsorption processes. [1] M. Radecka, K. Zakrzewska, M. Rekas, SnO2-TiO2 solid solutions for gas sensors, Sensors and Actuators B 47(1998) 194. [2] G. Sberveglieri, G. Faglia, S. Groppelli, P. Nelli and A. Camanzi, A new technique for growing large surface area SnO2 thin films (RGTO technique), Semicon. Sci. Technol. 5(1990) 1231.

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Humidity Characteristics of Thin Metal Oxide-Zeolite Films: *Jun-ichi Kodama*[1]; [1]Kinki University, Elect. Dept., 3-4-1 Kowakae, Higashi-Osaka, Osaka 577-8502 Japan

It is earnestly demanded for producing sensitive, stable and reproducible thin film humidity sensor of which resistance can respond quickly in the humidity atmosphere. It was found that the mixture of compound zeolite and metal oxide thin films such as NiO, CuO and Fe2O3 fabricated by evaporation, exhibit a good humidity characteristics. Compound zeolite itself exhibits humidity characteristics. But, it is not desirable to use it as a hygrometer by itself because of its high resistivity and its hygroscopic swelling. Metal oxides above mentioned not only prevent those troubles but also have a function to produce a conduction carrier. I will report in the paper about (1) fabrication method including how to decrease a film resistance by using surface processing, (2) humidity sensitivity by means of resistance measurement which changes from the order of 105 Ohm in 30%RH to the order of 102 Ohm in 80%RH and (3) stability and reproducibility.

4:00 PM
Construction of a New C-H-O Ternary Diagram for Diamond Deposition from the Vapor Phase: *Mahendra K. Sunkara*[1]; Sally C. Eaton[1]; [1]University of Louisville, Dept. of Chem. Eng., Louisville, KY 40292 USA

A new C-H-O ternary diagram based on radical species composition is constructed that distinguishes different regions for diamond deposition, non-diamond deposition and no deposition from the vapor phase. This construction is based on the steady state computations of gas phase and gas-surface chemistry for the data points presented in the original C-H-O ternary diagram by Bachmann et. al.1 based on feed gas compositions. The analysis of the computational domain indicates that the radical species composition do distinguish the three regions and helps explain the contradicting experimental data points with non-typical feed gas mixtures. The analysis also shows that the revised diagram based on radical species composition works only when carbon monoxide (CO) is treated as a neutral species. The effects of temperature and pressure on this new ternary diagram are also explained. Furthermore, the new ternary diagram is shown to be useful when analyzing the diamond deposition from the vapor phase inside trenches. Financial support came from NSF through CAREER award #CTS 9876251 and from KY NASA-EPSCoR program through KSGC fellowship (Sally Eaton). Reference:1. P.K. Bachmann, D. Leers and H. Lydtin, Diamond and Related Materials, 1, 1 (1991).

4:20 PM
An Analysis for Capillary Penetration Kinetics in Reactive Couples: *R. Asthana*[1]; [1]University of Wisconsin-Stout, Manu. Eng. Pgm., Menomonie, WI 54751 USA

The spreading kinetics in selected high-temperature reactive couples are revisited to identify flow regimes from plots of ln (droplet radius) versus ln (time) that are formally consistent with the classical models for simple liquids. It is suggested that while such a presentational approach could allow for identification by exclusion of complex flow regimes that are limited by interfacial reactions or diffusion, empirical models provide a more convenient approach to analyze practical spreading problems. It is shown that complex flow behavior of several reactive couples is compatible with an empirical relationship of the form: $q(t) = q0 + q0 \exp(B-At)$, where q(t) and q0 are instantaneous and equilibrium values of the contact angle, and B and A are system specific constants. This relationship is used in an analysis for capillary penetration kinetics for several cases of practical interest, such as, a

homogeneous capillary, a stripwise binary capillary with or without gravitational effects, and a homogenous capillary with shrinking pores due to reaction. Model calculations for the penetration by molten Si of C and Si3N4 capillaries (single phase or binary) show that time-dependent contact angles retard the penetration kinetics. With a binary capillary, the analysis yields stepped infiltration profiles that match the predictions based upon Cassie's effective contact angles for both gravity-free and gravity-inclusive situations. The computational outcomes for the combined effects of shrinking pore and time-dependent contact angles are assessed in light of recent experimental work in reactive systems.

Ultrafine Grained Materials: Severe Plastic Deformation Processing: II

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

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

Tuesday PM — Room: Polk A/B
March 14, 2000 — Location: Opryland Convention Center

Session Chair: S. Lee Semiatin, Air Force Research Laboratory, Wright-Patterson Air Force Base, OH 45433-7817 USA

2:00 PM Invited

Grain Refinement of Titanium Aliminides by Equal Channel Angular Extrusion: *Shankar M.L. Sastry*[1]; Rabindranath Mahapatra[2]; Dennis Hasson[3]; [1]Washington University, Mech. Eng. Dept., Campus Box 1185, One Brookings Dr., St. Louis, MO 63130 USA; [2]Naval Air Warfare Center, Aircraft, Matls. Lab., Patuxent River, MD 20670 USA; [3]U.S. Naval Academy, Mech. Eng., 590 Holloway Rd., Annapolis, MD USA

With the objectives of producing sub micron grains and determining the beneficial effects of such sub micron grained microstructures on mechanical properties, several gamma+alpha2 based Ti-Al-X (X= Nb, Cr, Mn,Mo) alloys were processed by equal channel angular extrusion (ECAE) at 1200-1500K. 6-12 mm diameter cylindrical specimens were produced by multiples passes at different temperatures and were annealed at 1300-1600K. Microstructures of as processed and annealed specimens were determined by transmission and scanning electron microscopy. Hardnesses, bend strengths, and bend ductilities were evaluated as functions of the temperature, deformation rate, and number of passes. Whereas in conventionally processed materials, the grain sizes were 10-50 micrometers, the grain sizes were < 2 micrometers in ECAE processed and annealed conditions. Furthermore there was ample evidence for the break up and conversion of gamma|alpha2 lamellar morphology to equaixed gamma and alpha2 grains. The effects of microstructural modifications on mechanical properties were determined.

2:25 PM Invited

Ultrafine Grained Materials by Equal Channel Angular Processing (ECAE): *Gary E. Korth*[1]; Thomas M. Lillo[1]; Jenya Macheret[2]; John E. Flinn[3]; [1]Idaho National Engineering and Environmental Laboratory, Met. and Cer., P.O. Box 1625, Idaho Falls, ID 83415-2218 USA; [2]U.S. Department of Energy, Idaho Operat. Off., 850 Energy Dr., Idaho Falls, ID 83415-1225 USA; [3]University of Idaho, 3450 S. 35 West, Idaho Falls, ID 83402 USA

Ultrafine grains were obtained in several alloys by processing with ECAE. Materials processed were Copper Alloy 101 (OFHC), Copper Alloy 260 (70/30 brass), Nickel Alloy 270 (99.97% pure), high purity aluminum (99.999%), and Aluminum Alloy 1100. In all cases, ultrafine grains (<1um) were produced with multiple passes through the ECAE die. Mechanical property and microstructural analysis of as-processed and annealed material showed the grain stability to be much less in the high purity materials than was observed in the more "dirty" alloys. ECAE processing produces a very high level of stored energy in the microstructure which provides a strong driving force for grain growth at relatively low annealing temperatures if grain boundaries are relatively clean. Any dispersions or impurities tend to pin the grain boundaries and stabilize the ultrafine grains.

2:50 PM

Multiaxis Deformation Methods to Achieve Extremely Large Strains and Ultrafine Grain Steels: *Wayne C. Chen*[1]; David E. Ferguson[1]; Hugo S. Ferguson[1]; [1]Dynamic Systems Inc., 323 Route 355, P.O. Box 1234, Poestenkill, NY 12140 USA

Ultrafine grain size is often achieved by severe plastic deformation. A few techniques have been developed to achieve severe plastic deformation, such as equal channel angular (ECA) pressing/extrusion, torsion straining, and accumulative roll bonding (ARB) techniques. This paper will introduce multiaxis deformation techniques which can achieve extremely large strains with constant deformation volume. Two different types of hot deformation methods, each with a different number of deformation axis, are studied. They are two-axis deformation and three-axis deformation. The two-axis deformation can be fully restrained or unrestrained lengthwise. The three-axis deformation has no restraint. The bulk volume of multiaxis full restraint compression specimens can be easily machined into mechanical test samples for mechanical property measurements and other studies. A plain carbon steel (AISI 1018) was studied using the Multi-Axis Restraint Compression system developed at Dynamic Systems Inc.. One micron grain size was achieved with the plain carbon steel. The ultimate tensile strength measured doubled, when compared to that of the conventionally hot rolled material.

3:10 PM

Fabrication of Ultra Fine Grained Bulk Iron through Mechanical Milling of Iron Powder: *Setsuo Takaki*[1]; Yuji Kimura[2]; [1]Kyushu University, Dept. of Matls. Sci. and Eng., 6-10-1 Hakozaki, Higashiku, Fukuoka 812-8581 Japan; [2]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

Mechanical milling of metallic powders is a useful technique for giving ultimate severe deformation to the metal and producing nanosized grains within the powder particles. If the mechanically milled metallic powders were successfully consolidated to bulk without loosing the fine grained microstructure, we ought to be able to obtain ultra fine grained bulk materials. In this paper, the MMC process (Mechanical Milling & Consolidation process) will be introduced for iron as a technique to fabricate ultra fine grained bulk materials and be discussed on the Hall-Petch relationship of iron materials in the ultra fine grained region.

3:30 PM

Grain-Size Evolution in Nanocrystalline Fe during Mechanical Arttrition: *Michael Atzmon*[1]; Huahang Tian[2]; [1]University of Michigan, Dept. of Nucl. Eng. & Rad. Sci. and Matls. Sci. & Eng., Cooley Bldg./North Campus, Ann Arbor, MI 48109-2104 USA; [2]University of Michigan, Dept. of Matls. Sci. and Eng., Ann Arbor, MI 48109-2136 USA

We present a study of the evolution of grain size and strain in Fe powder during low-energy ball milling. For this method of inducing mechanical attrition, local heating by impact is negligible, so that the temperature and milling intensity can be controlled independently. Using the Warren-Averbach method, the grain size and RMS strain were determined as a function of milling time for temperatures between 298K and 523K and varying milling amplitudes. Average grain diameters as small as 7 nm were observed. A model was developed to describe the evolution of the grain size, based on a rate equation which includes grain refinement and simultaneous grain growth. The contri-

bution to grain growth by nonequilibrium vacancies generated by deformation was incorporated, using concepts developed to model radiation-enhanced diffusion. The model expression fits well the temporal curves for all conditions, and the behavior of the fitting parameters as a function of temperature and milling intensity is consistent with the assumptions made. In particular, the activation energy for the kinetic coefficient for grain growth is negligible below 473K, in agreement with the dominance of nonequilibrium defects. A thermally activated contribution to the grain-growth term is noticeable above 473K. The fraction of the impact energy which is converted into grain boundaries or point defects is observed to decrease with the milling amplitude. An application of the results to observed non-monotonic precipitation in ball-milled supersaturated solid solution will be presented.

3:50 PM Break

4:00 PM Invited

Mechanisms of Ultrafine Grain Formation during Severe Plastic Deformation: *R. Kaibyshev*[1]; [1]Institute for Metals Superplasticity Problems RAS, Khalturina 39, Ufa 450001 Russia

Present work is an overview of experimental data dealt with microstructural evolution during severe plastic deformation. Two techniques were used to deform bulk materials. A magnesium alloy Mg-5.8%Zn-0.65%Zr, aluminum alloy 2219 and 15Cr25Ti steel were strained at ambient temperature by using of Bridgeman anvil. Few aluminum alloys were processed by ECAE at enhanced temperatures. Evaluation of mechanical properties, X-ray examination and TEM observations were performed to reveal operating mechanisms of ultrafine grain formation. It was shown that stacking fault energy (SFE) and temperature strongly influence mechanism of nanocrystalline and submicrocrystalline grain formation. In general the microstructural evolution consists of three stages. At first stage the subdivision if initial grains into fragments occurs. In materials with low values of SFE a deformation twinning provides this subdivision at ambient temperature. In material with high values of SFE a formation of low energy dislocations structures results in grain separation. Dynamic recrystallization may occur at moderate temperatures and refinement of initial structure takes place. At second stage of microstructural evolution a single slip occurs in fine crystallites and a strong increase of lattice dislocation density is observed. A specific mechanism for ultrafine grain formation is operative. This mechanism is considered in details. Formed structure is highly non-equilibrium due to high density of extrinsic dislocations into grain boundaries. At third stage a recovery process occurs into boundaries of ultrafine grains. It leads to decrease of internal stresses. Effects of initial grain size and secondary phases on the mechanisms of ultrafine grain formation are discussed.

4:25 PM Invited

Severe Plastic Deformation of Ti-6Al-4V via Equal Channel Angular Extrusion: *David P. DeLo*[1]; S. Lee Semiatin[2]; [1]Extrude Hone Corporation, 1 Industry Blvd., Irwin, PA 15642 USA; [2]Air Force Research Laboratory, Matls. Process. and Process. Sci., Matls. and Manufact. Direct., AFRL/MLLM, Wright-Patterson Air Force Base, OH 45433-7817 USA

The equal channel angular extrusion process (ECAE) imposes severe plastic deformation throughout a bulk section of material without changing the overall dimensions of the workpiece. Incremental deformation occurs in the form of simple shear confined to a narrow zone. Under ideal conditions, the simple shear deformation occurs uniformly throughout the workpiece refining the microstructure and affecting crystallographic orientations. Physical models were used to study the effects of ECAE processing on Ti-6Al-4V billets having either a lamellar or an equiaxed alpha preform microstructure. Microstructural features resulting from ECAE performed at various temperatures and using various processing routes are compared. The relationships between material flow properties, processing conditions, macroscopic deformation during ECAE, and the resulting microstructures are described.

4:50 PM

Effect of Large-Strain Deformation Prior to Austenitization on Austenite Grain Size in 0.3%C-9%Ni Steel: *Tomoyuki Yokota*[1]; Tetsuo Shiraga[1]; Masakazu Niikura[1]; Kaoru Sato[2]; [1]Ferrous Super Metal Consortium of Japan, NKK Corp. Matls. & Processing Rsch. Ctr., 1-1 Minamiwatarida-cho, Kawasaki-ku, Kawasaki-shi 210-0855 Japan; [2]Ferrous Super Metal Consortium of Japan, NKK Corp. App. Tech. Rsch. Ctr., 1-1 Minamiwatarida-cho, Kawasaki-ku, Kawasaki-shi 210-0855 Japan

Fundamental research project to pursue ultra fine grain size below 1micron in steel is currently under way as “Ferrous Super Metal” project supported by NEDO (New Energy and Industrial Technology Development Organization). One effort within this national project is austenite grain size refinement, which is one of the most important factors for microstructural control in heat treatment. Cold or warm deformation prior to austenitization is known to have a beneficial effect on austenite grain size, and in this paper, the effect of large-strain deformation at a temperature range just below Ac1 transformation temperature on austenite grain size after austenitization was studied using 0.3%C-9%Ni steel. Because this steel has high hardenability, once reverse transformation occurs, austenite transforms martensite with extremely high hardness. Deformation up to 70% was done in a single pass by compression of cylindrical specimens using laboratory deformation simulator. Increase in reduction ratio up to 50% continuously refined austenite grain size down to 2 micron after subsequent reheating to austenitization temperature (700°C) by induction rapid heating. Drastic change in reverse transformation behavior was found for the reduction above 70%. Spontaneous reverse transformation was induced by such a large strain deformation even without subsequent reheating. It was revealed by TEM observation that austenite grain size of the specimen was remarkably refined down to around 0.5 micron, and it showed hardness of fully quenched martensite. Adiabatic heating due to deformation was suggested to contribute to such a spontaneous transformation. Transformation mechanism and required metallurgical condition for the spontaneous transformation will be discussed.

5:10 PM

Thermal Stability of Ultrafine Grained Ferritic Structure of Iron with Oxide Particles: *Yuuji Kimura*[1]; Satoru Nakamyo[2]; Hideyuki Hidaka[2]; Hideto Goto[2]; Setsuo Takaki[2]; [1]National Research Institute for Metals, Frontier Rsch. Ctr. for Struct. Matls., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]Kyushu University, Matls. Sci. and Eng., Grad. School of Eng., 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan

Mechanical milling is very effective for charging a large strain into metals, because endless cyclic deformation can be performed through kneading reaction. Sufficient milling treatment finally causes an ultimate work-hardening based on the ultra grain refining to about few ten nm or less (charged true strain is more than 10). Moreover, we confirmed in ferritic stainless steel powders that a large amount of oxide particles, which are thermodynamically so stable, were not only dispersed but also decomposed during ultra grain refining of the matrix and then re-precipitated very finely on annealing. In this paper, iron powders with oxide particles were mechanically milled and consolidated to bulk materials. The thermal stability of ultra fine ferrite grain structures were then investigated in relation to the structural change and the Zener pinning effect of the oxide particles.

WEDNESDAY AM

Daily Personal Schedule - Wednesday - March 15

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

❖❖❖

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

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

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

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

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

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

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

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

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

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

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

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

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

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

9:50 AM Break

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

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

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

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

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

10:50 AM

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

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

11:10 AM

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

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

Alumina and Bauxite: Bayer Process Development

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

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

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

8:30 AM Invited

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

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

9:15 AM

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

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

9:40 AM

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

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

10:05 AM Break

10:25 AM

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

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

10:50 AM

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

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

11:15 AM

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

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

11:40 AM

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

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

Aluminum Reduction Technology: Environmental

Sponsored by: Light Metals Division, Aluminum Committee

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

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

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

8:30 AM Invited

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

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

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

9:00 AM

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

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

9:25 AM

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

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

9:50 AM

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

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

10:15 AM Break

10:25 AM

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

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

10:50 AM

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

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

11:15 AM

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

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

Carbon Technology: Materials Properties and Modeling

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

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

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

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

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

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

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

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

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

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

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

Cast Shop Technology: Melt Quality and Foundry

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

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

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

8:30 AM Introductory Remarks

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

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

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

9:00 AM

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

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

9:25 AM

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

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

9:50 AM

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

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

10:15 AM Break

10:20 AM

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

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

10:45 AM

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

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

11:10 AM

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

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

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

11:35 AM

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

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

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

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

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

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

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

8:30 AM

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

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

8:55 AM

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

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

9:20 AM

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

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

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

9:45 AM Break

10:10 AM

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

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

10:35 AM

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

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

11:00 AM

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

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

11:25 AM

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

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

General Recycling of Materials: Topics Related to Light Metals Recycling

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

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

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

8:30 AM Introductions and Opening Comments

8:40 AM

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

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

9:05 AM

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

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

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

9:30 AM

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

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

9:55 AM

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

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

10:20 AM Break

10:45 AM

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

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

11:10 AM

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

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

11:35 AM

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

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

12:00 PM

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

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

High Resolution Electron Microscopy in Materials Science: Interfaces

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

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

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

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

8:30 AM Invited

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

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

9:00 AM

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

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

9:30 AM

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

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

10:00 AM

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

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

10:30 AM Break

10:50 AM Invited

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

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

11:20 AM

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

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

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

11:40 AM

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

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

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

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

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

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

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

8:30 AM Keynote

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

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

9:00 AM Invited

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

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

9:20 AM Invited

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

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

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

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

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

10:00 AM Break

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

9:00 AM Invited

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

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

9:30 AM Invited

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

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

10:00 AM Invited

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

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

10:30 AM Break

10:45 AM Invited

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

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

11:15 AM Invited

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

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

11:45 AM Invited

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

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

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

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

Sponsored by: Materials Processing and Manufacturing Division,

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

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

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

8:30 AM

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

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

8:50 AM

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

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

9:10 AM

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

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

9:30 AM

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

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

9:50 AM

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

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

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

10:10 AM Break

10:20 AM

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

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

10:40 AM

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

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

11:00 AM

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

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

International Symposium on Iridium: Processing, Refining, and Chemistry

Sponsored by: Structural Materials Division, Refractory Metals Committee

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

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

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

8:30 AM

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

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

8:50 AM

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

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

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

9:10 AM

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

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

9:30 AM

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

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

9:50 AM

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

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

10:10 AM Break

10:20 AM

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

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

10:40 AM

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

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

11:00 AM

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

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

11:20 AM

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

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

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

11:40 AM

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

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

12:00 PM

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

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

Liquid Metal Atomization: Fundamentals and Practice: Gas Atomization

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

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

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

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

8:30 AM Invited

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

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

9:00 AM Invited

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

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

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

9:30 AM Invited

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

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

9:55 AM Invited

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

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

10:20 AM Break

10:35 AM

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

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

11:00 AM Invited

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

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

11:25 AM Invited

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

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

Magnesium Technology 2000: Creep Properties and Heat Treating Effects

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

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

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

8:30 AM

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

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

8:55 AM

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

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

9:20 AM

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

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

9:45 AM

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

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

10:10 AM Break

10:20 AM

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

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

10:45 AM

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

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

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

11:10 AM

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

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

11:35 AM

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

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

Magnesium Technology 2000: Solidification

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

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

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

8:30 AM

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

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

8:55 AM

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

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

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

9:20 AM

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

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

9:45 AM

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

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

10:10 Break

10:20 AM

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

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

10:45 AM

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

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

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

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

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

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

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

8:30 AM Opening Comments

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

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

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

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

9:40 AM Break

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

10:10 AM Break

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

9:55 AM Break

10:15 AM Invited

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

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

10:45 AM

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

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

11:10 AM

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

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

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

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

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

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

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

8:30 AM

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

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

8:55 AM

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

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

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

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

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

9:45 AM Break

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

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

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

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

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

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

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

Sponsored by: Light Metals Division, Reactive Metals Committee

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

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

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

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

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

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

9:00 AM

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

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

9:30 AM

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

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

10:00 AM Break

10:30 AM

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

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

11:00 AM

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

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

11:30 AM

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

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

12:00 PM

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

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

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

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

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

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

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

8:30 AM Invited

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

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

9:00 AM

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

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

9:20 AM

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

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

9:40 AM

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

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

10:00 AM

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

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

10:20 AM Break

10:30 AM

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

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

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

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

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

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

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

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

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

11:50 AM Closing Remarks

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

8:30 AM

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

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

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

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

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

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

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

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

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

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

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

10:10 AM Break

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

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

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

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

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

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

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

Ultrafine Grained Materials: Mechanical Behavior and Strengthening Mechanisms: I

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

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

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

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

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

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

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

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

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

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

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

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

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

10:00 AM

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

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

10:20 AM Break

10:30 AM Invited

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

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

10:55 AM

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

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

11:15 AM

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

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

11:35 AM

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

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

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

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

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

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

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

2:00 PM

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

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

2:20 PM

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

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

2:40 PM

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

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

3:00 PM

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

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

3:20 PM Break

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

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

4:00 PM

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

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

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

4:20 PM

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

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

4:40 PM

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

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

Alumina and Bauxite: Bayer Process Chemistry Optimization

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

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

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

2:00 PM

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

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

2:25 PM

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

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

2:50 PM

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

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

3:15 PM

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

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

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

3:40 PM Break

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

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

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

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

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

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

Aluminum Reduction Technology: Cell Operation/Innovations

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

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

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

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

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

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

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

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

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

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

3:20 PM Break

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

Carbon Technology: Raw Materials

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

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

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

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

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

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

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

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

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

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

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

3:45 PM Break

3:55 PM
Reactivity and Texture of Cokes Doped with Aluminum Compounds: *Marianne Aanvik*[1]; Morten Sorlie[2]; Harald A. Oye[1]; [1]Norwegian University of Science and Technology, Dept. of Chem. and Inorganic Chem., Trondheim N-7491 Norway; [2]Elkem ASA Research, P.O. Box 8040 Vagsbygd, Kristiansand N-4675 Norway

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

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

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

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

Cast Shop Technology: Fluxing and Filtration

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

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

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

2:00 PM Introductory Remarks

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

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

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

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

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

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

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

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

3:45 PM Break

3:50 PM
Evaluation of a Filter Developed to Remove Liquid Salts from Molten Aluminum: *Kenneth R. Butcher*[1]; Dawid D. Smith[1]; Leonard Aubrey[1]; [1]Selee Corporation, R&D, 700 Shepherd St., Hendersonville, NC 28792 USA

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

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

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

4:40 PM
2D Transient Mathematical Model of Aluminum Filtration: *Duygu Kocaefe*[1]; Rung Tien Bui[1]; Peter Waite[2]; [1]University of Quebec, Dept. of Appl. Sci., 555 Boul. De l'Universite, Chicoutimi, Quebec, Canada; [2]Alcan International Limited, P.O. Box 1250, Jonquiere, Quebec, Canada

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

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

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

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

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

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

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

2:00 PM

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

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

2:25 PM

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

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

2:50 PM

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

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

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

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

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

3:40 PM Break

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

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

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

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

4:55 PM

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

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

General Abstracts: Intermetallics II: Aluminides

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

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

2:00 PM

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Sponsored by: Structural Materials Division, Physical Metallurgy Committee

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

High Temperature Processes for Waste Treatment & Minimization: I

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

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

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

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

2:00 PM

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

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

2:30 PM

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

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

3:00 PM

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

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

3:30 PM Break

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

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

4:15 PM

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

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

4:45 PM

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

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

Honorary Symposium for Professor Oleg D. Sherby: Superplasticity A

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

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

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

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

2:00 PM Invited

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

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

2:20 PM Invited

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

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

2:40 PM Invited

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

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

3:00 PM Invited

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

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

3:20 PM Break

3:30 PM Invited

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

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

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

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

Abstract text not available

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

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

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

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

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

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

Liquid Metal Atomization: Fundamentals and Practice: Other Methods

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

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

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

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

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

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

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

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

2:55 PM Invited

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

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

3:20 PM Invited

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

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

3:45 PM Break

4:00 PM Invited

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

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

4:25 PM Invited

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

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

4:50 PM Invited

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

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

5:15 PM Invited

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

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

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

Magnesium Technology 2000: Physical and Mechanical Properties

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

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

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

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

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

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

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

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

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

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

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

3:40 PM Break

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

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

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

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

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

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

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

Program Organizers: Yellapu V. Murty, Carpenter Technology Corporation, Research and Development, Reading, PA 19612-4662 USA; Prasad Godavarti, Motorola, Austin, TX USA; Sung-Ho Jin, Bell Laboratories, Lucent Technologies, Murray Hill, NJ 07974 USA; Sung Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598 USA; John Macwilliams, US Competitors, LLC., Newark, DE 19711 USA; Mark McCormack, Fujitsu Computer Packing Technologies, San Jose, CA 95134 USA; Martin Weiser, AlliedSignal Electronic Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Wednesday PM — Room: Lincoln C
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: Sung K. Kang, IBM, Yorktown Heights, NY 10598 USA; Prasad Godavarti, Motorola, Austin, TX 78721 USA

2:00 PM Opening Comments

2:05 PM

Low Alpha Solder Electrolyte Used for Flip Chip Bump Formation: *Masayoshi Kohinata*[1]; Kiyotaka Tsuji[2]; Keigo Obata[3]; [1]Mitsubishi Materials Corporation, 12-6, Technopark Sanda, Hyogo 669-1339 Japan; [2]Ishihara Chemical Company Limited, 5-26, Nishi-Yanagihara, Hyogo-Ku, Kobe 652-0806 Japan; [3]Daiwa Fine Chemicals Company Limited, 1-17, 2-Chome Shimosawa-Dori, Hyogo-Ku, Kobe 652-0047 Japan

We have developed a low alpha solder electrolyte used for flip chip bump formation. It is generally known that the alpha particle count from the deposit immediately after plating are low, but increase with time. Using our electrolyte, the alpha particle count from the deposit can be very low and stable. The characteristics of the bump plated for 63% tin and for 5% tin solder electrolytes are given, including the uniformity of the bump height and the bump compositions over the wafer. The starting materials and electrolyte are fully developed, so our electrolyte is already in mass production. We also describe an evaluation method for the electrolyte that controls parameters influencing bump characteristics.

2:30 PM

Low-Alpha Lead for Solder Bumping Production: *Mark W. Roberson*[1]; Phil A. Deane[1]; Salvatore Bonafede[1]; Alan Huffman[1]; Sundeep Nangalia[1]; [1]MCNC, Elect. Tech. Div., 3021 Cornwallis Rd., Research Triangle Park, NC 27709 USA

Soft-errors caused by lead in solder-bumping have been a concern for many years. The problem is of special concern for high-density interconnection applications requiring solder to be placed directly over active circuitry. In that situation, alpha particles emitted by radioactive lead cause soft-errors with no possibility of shielding circuitry. For optimal cost-effectiveness, though, not all solder bumped wafers require low-alpha lead. MCNC has developed a solder bumping facility with both a research branch at MCNC and a full-scale production facility at its spin-off, Unitive Electronics Inc. We present results here of our work in incorporating low-alpha lead as part of our solder bumping process. We describe the amount of cross-contamination measured when alternating plating baths of regular lead and low-alpha

lead. We also present geometric correction factors for solder bumps measured with alpha detection counters.

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A Low Alpha Eutectic Type 6 Solder Paste for Ultra Fine Pitch Chip Bumping: *Mike Grosse*[1]; Jim Grundy[1]; Tom Herrington[1]; [1]Johnson Matthey Electronics, 10080 Willow Creek Rd., San Diego, CA 92131 USA

The rapid growth in chip level interconnection density continues to present new challenges to the suppliers of semiconductor packaging materials. Further increases in device speed and functionality with corresponding decreases in device geometry has placed tighter constraints on packaging materials and their related processes; solder paste being one of those critical materials. With the advent of bump arrays numbering in the thousands of bumps/print at ever decreasing pitch dimensions, higher and higher first pass yields will be required of the solder paste and process used for flip chip bumping. In order to achieve the higher print resolutions demanded by the flip chip process, it was necessary to optimize both the powder process and flux system. Process optimization revealed that by instituting additional controls, a type 6 powder as specified by IPC in standard J-STD-006 could be produced with reasonable final yields. Various designed experiments were conducted to identify the critical process variables and conditions that influence particle shape, surface morphology, particle size distribution and oxide content. Furthermore, as powder particle size is reduced to enhance print definition, the surface area of the powder and consequently the amount of surface oxide increases and requires careful monitoring to insure lot to lot consistency. The reduction in on-chip feature size has led to a closer proximity of logic elements and the solder bump interconnects. Industry concerns have grown over the phenomena of radioactive decay induced soft errors generated by trace isotope impurities found in lead containing solders. In order to address this reliability concern, it was necessary to obtain a lead supply with an extremely low concentration level of the isotope 210Pb. Post powder processing alpha emission flux testing is performed to determine the alpha emission rate and to gain insight into the decay characteristics of the material.

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Phase Equilibria of Ag-Sn-Cu Ternary System: *Yee-wen Yen*[1]; Sinn-wen Chen[1]; [1]National Tsing-Hua University, Dept. of Chem. Eng., #101 Sec. 2 Kuang-Fuh Rd., Hsin-Chu, Taiwan 300

Ag-Sn alloys are a prominent group of lead-free solders. Cu is the most popular substrate, and the phase equilibria of Ag-Sn-Cu ternary system is thus of importance in electronic industry. Various Ag-Sn-Cu alloys are examined and the isothermal sections at 240°C and 450°C have been experimentally determined. At 240°C, the e1-Cu3Sn phase is in equilibrium with Ag, z-Ag4Sn, and e2-Ag3Sn phases. The existence of d-Cu4Sn phase at 450°C does not change much of its phase equilibria, and e1 phase is still in equilibrium with Ag, z and e2 phases at 450°C, which indicates that the e1 phase is a very stable phase. Based on two experimentally determined isothermal sections and the knowledge of the thermodynamic models of its three binary constituent systems, thermodynamic models of the Ag-Sn-Cu have been assessed in this study. The calculated results and the experimental determination of phase equilibria are in good agreement.

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3-D Simulations of Electromigration and Stress Evolution in Interconnections: *Zhineng Fan*[1]; Sven Rzepka[1]; Matt Korhonen[1]; Che-Yu Li[1]; [1]Cornell University, Dept. of Matls. Sci. and Eng., Bard Hall, Rm. 356, Ithaca, NY 14853 USA

As IC devices scale down, the cross section of interconnect lines on the chip are reduced and the current density increases. This puts electromigration in a particular position to interconnection reliability. While the electromigration and its related phenomena are rather complex, analytical simulation methods only give the results under very simplified conditions. In most cases, those models are one-dimensional and apply the hydrostatic stress as the chemical driving force. Numerical simulation, especially finite element analysis (FEA), is much more powerful to solve the complex problems. In this paper, we demonstrate a 3-D electromigration model that was developed based on a commercial FEA code. The model is capable to include Coble creep and Nabarro-Herring creep by applying normal stress as the chemical driving force. The interconnect segment consisting a polycrystalline section with bamboo structure connected to it at both sides was surveyed. The evolution of the atomic flow, normal stress and hydrostatic stress on the grain boundary are presented in this paper. It is shown that by using hydrostatic stress as the chemical driving force will overestimate the backflow of the stress-driven diffusion.

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Characterization of Electroplated Bismuth-Tin Alloys for Electrically Conducting Adhesive Materials: *Sung K. Kang*[1]; Stephen L. Buchwalter[1]; Cornelia K. Tsang[2]; [1]IBM, Rsch. Dept., T.J. Watson Rsch. Ctr., P.O. Box 218, Yorktown Heights, NY 10598 USA; [2]MIT, Matls. Sci. & Eng., Cambridge, MA 02139 USA

Electrically conducting adhesives are promising alternatives for lead (Pb)-containing solders in microelectronic applications. However, most common silver-filled epoxy materials have various limitations to meet the requirements of the solder joints yet. To overcome these limitations, several new formulations have been developed recently. Among them, a new high conductivity Pb-free conducting adhesive developed for low temperature applications has been previously reported. This conducting adhesive contains a conducing copper filler powder coated with a low melting point metal or alloy, such as Sn or BiSn. The low melting point layer serves as a joining material among the filler particles as well as to the substrate. In this paper, characterization of electroplated BiSn alloys on a Cu substrate is reported for their microstructure, electrical properties, oxidation behavior and others. The experimental results have provided a better understanding of the joining mechanism of the newly-developed Pb-free conductive adhesive.

4:50 PM

An Evaluation of Automated Ball Indentation for Measuring the Tensile Properties of Solder Alloys: *Michael L. Santella*[1]; Tsung-Yu Pan[2]; Frank W. Gayle[3]; [1]Oak Ridge National Laboratory, Met. & Cer. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6096 USA; [2]Ford Motor Company, Ford Rsch. Lab., 20000 Rotunda Dr., MD 3135, SRL, P.O. Box 2053, Dearborn, MI 48121-2053 USA; [3]National Institute of Standards and Technology, Rm. 223-B164, Gaithersburg, MD 20899 USA

The automated ball indentation (ABI) testing technique is not well developed for alloys other than steels, but its apparent ability to measure mechanical properties in small volumes of material makes it of interest for testing solder joints. The technique was evaluated by first preparing 1.5-mm-thick tensile specimens of bulk a Sn-3.5Ag wt% alloy and testing them at room temperature. ABI tests were then done on the shoulder regions of the tensile specimens. The overall agreement of the data sets was good, with the ABI data indicating a slightly lower yield strength and slightly higher flow stresses and work hardening rate compared to the tensile test data. Subsequently, small FR4 test boards were obtained that contained arrays of solder bumps made with the same alloy. The solder bumps had nominal dimensions of 2.5 mm long x 0.8 mm wide x 0.30-0.50 mm thick. ABI test results from the solder bumps agreed well with those from the tensile specimen shoulders. Details of the data comparisons will be presented and discussed in terms of relevant alloy characteristics and microstructures. The results indicate the reasonable possibility of making valid tensile property measurements on actual solder joints.

Materials Processing in the Computer Age III: Computer Aided Instruction

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizers: Vaughan Voller, University of Minnesota, Saint Anthony Falls Laboratory, Minneapolis, MN 55414-2196 USA; Hani Henein, University of Alberta, Edmonton, AB T6G 2G6 Canada; Sulekh Jain, Ge Aircraft Engineering, MId M-89, Cincinatti, OH 45215 USA

Wednesday PM Room: Lincoln A
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Brian G. Thomas, University of Illinois, Mech. Eng. Dept., Urbana, IL 61801 USA; Garry W. Warren, University of Alabama, Metallu. & Matls. Eng., Tuscaloosa, AL 35487 USA

2:00 PM
Introducing Materials to 6th, 7th and 8th Grade Science-A First Step: *Garry W. Warren*[1]; [1]University of Alabama, Metallu. & Matls. Eng., P.O. Box 870202, Tuscaloosa, AL 35487 USA

Computer Aided Instructional (CAI) software with a materials component have been developed for 6th to 8th grades science classes. The software is a result of the educational outreach activities of the NSF sponsored MRSEC at the University of Alabama's Center for Materials for Information Technology. A primary emphasis of the MRSEC's educational outreach activities involves collaboration with a very innovative 6th to 8th grade Integrated Science (IS) curriculum based in the Center for Communication and Educational Technology at the University of Alabama. Three of the authors are teachers in this program and participated in producing three software packages dealing with (1) Simple Machines (levers), (2) The Periodic Table and (3) The Scientific Method. In many cases these software packages probably represent the students first exposure to these topics, therefore a primary objective behind their development was to present the information in a fashion that would retain student interest, convey important information accurately, and above all complement and reinforce other classroom activities. Emphasis was placed on making the software truly interactive, requiring the student to participate by answering questions, selecting options and by incorporating moving objects, sounds and rewards for correct answers. Wherever possible interesting materials and applications thereof are incorporated. The financial support of the NSF Research Experiences for Teachers program is gratefully acknowledged.

2:20 PM
Use of the Web in Materials Teaching and Research: *Vaughan R. Voller*[1]; [1]University of Minnesota, Saint Anthony Falls Lab., Mississippi River at 3rd Ave. S.E., Minneapolis, MN 55414 USA

Among the recent innovations of the computer age the World Wide Web has and will continue to have a significant impact in teaching, education and research dissemination. This paper explores some examples of use of the Web and associated resources for teaching and research in the materials processing field. Specific examples will include: An outline of authoring tools for preparing engineering learning modules. HTML for interactive Web Quizzes Preparation of simple GIF animations. Running Interactive Web programs in scripting languages (e.g., JAVA-Script). For the most part the examples will be taken directly from the experiences of the author. The central purpose will be to show the accessibility and ease of use of available tools and demonstrate how a small amount of effort can lead to a significant value added to education and research endeavors. The key messages are (1)—taking note of other computer innovations of the last 50 years (e.g., programming)—to be fully effective individual educators and researchers need to take control of the tools and not rely on others to do it for them, and (2) the need to recognize, in an educational setting in particular, that although the infrastructure of a web page is important the critical component is the hard content. The work is supported by a TEL grant from Academic & Distributed Computing Services, University of Minnesota and by the NSF under Grant NSF/EEC-9711743.

2:40 PM
Computer Aided Instruction in Materials Science and Engineering Education: *Hani Henein*[1]; [1]University of Alberta, Adv. Matls. and Process. Lab., 536 Chem.-Matls. Eng. Bldg., Edmonton, Alberta T6G2G6 Canada

The easy accessibility and power of the PC as well as the education of our engineering students in the use of these tools, are compelling reasons for developing computer aided instructional (CAI) tools. There is a wide range of areas in the engineering curriculum where students must learn concepts involving the application of judgment for problem solving. Typically, throughout North America, the instructor of a course will provide theoretical guidelines to students on how to apply the required judgment. All too often, these points are lost on the student as it is deemed too complicated and too abstract. It is often too time consuming to get students to the point where they can practice this judgment. A number of computer aided packages have therefore been developed for use in several courses in the materials science and engineering curriculum that encompass both calculational and instructional capabilities. This talk will present an overview of some of these packages on the solution of a non-linear equation, the instruction of binary diffusion couples, an introduction of crystallography and an introduction to heat transfer. The context in which these CAI Tutorials were developed and are being used will be discussed. The use of the tutorials has important implications on the expectations from an engineering education of both instructor and student.

3:00 PM Break

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Open Discussion and Software Demonstration
The Role of Computer Based Training in Materials Processing

3:50 PM Closing Remarks

Process Synthesis and Modeling for the Production & Processing of Titanium & Its Alloys: Session IV

Sponsored by: Materials Processing and Manufacturing Division, Structural Materials Division, Titanium Committee, Shaping and Forming Committee
Program Organizers: James A. Hall, Oremet-Wah Chang, Albany, OR 97321 USA; F. H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026 USA; Isaac Weiss, Johnson Matthey, USA; Kuang Oscar Yu, RMI Corporation, R&D, Niles, OH 44446-0269 USA

Wednesday PM Room: Knoxville B
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Kuang Oscar Yu, RMI Corporation, R & D, Niles, OH 44446-0269 USA; James Hall, Oremet-Wah Chang, Albany, OR 97321 USA

2:00 PM
Solar: A Numerical Software to Optimize Vacuum Arc Remelting Operations: *A. Jardy D. Ablitzer*[1]; [1]Laboratoire de Science et Genie des Materiaux Metalliques, Ecole des Mines, Parc de Saurupt, Nancy, Cedex F-54042 France

A numerical model of the VAR process has been developed at the Ecole des Mines in Nancy and applied to simulate remelting operations realized in particular at Compagnie Europeenne du Zirconium CEZUS. The transient-state model named SOLAR (which stands for SOLidification during Arc Remelting) was continuously improved, by successively accounting for heat transfer, magnetohydrodynamic effects, and solute transfer (i.e. macrosegregation) during the melting, then while solidification of the final liquid pool proceeds. An important feature of the model is its ability to take into account any evolution of the melting rate and the stirring sequence during the melting, and to allow to understand the link between such operating parameters and the intensity of macrosegregation in the ingot. SOLAR has been used with some success to simulate pilot-plant and full-scale remeltings of steels, titanium and zirconium alloys. Recently, a significant improvement has allowed to simulate a triple VAR melt, using the computed composition of each ingot after cooling as the electrode composition for the next melting operation. SOLAR has enabled, for example, to determine the influence of the stirring sequence of preliminary melts on the intensity of macrosegregation in the final third ingot. Moreover, the potential interest of using a so-called "compensated" electrode, which means an initial electrode where the alloying element repartition varies along the length, can be quantitatively predicted before performing any actual triple vacuum arc remelting operation. Results of the model are presented and discussed.

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Ti-6V-Al under Electromagnetic Stirring and Melt Current Lorentz Force in Industrial Scale VAR: Parameter Sensitivity in Simulations: *L. A. Bertram*[1]; F. Spadafora[2]; S. N. Kempka[3]; R. L. Williamson[3]; R. S. Minisandram[4]; [1]Sandia National Laboratories, 7011 E. Ave., Livermore, CA 94551-0969 USA; [2]RMI Titanium Company; [3]Sandia National Laboratories, MS 0603, P.O. Box 5800, Alburquerque, NM 87185-0603 USA; [4]Allvac, Allegheny Teledyne Co., 2020 Ashcraft Ave., P.O. Box 5030, Monroe, NC 28111-5030 USA

In experiments conducted to establish, among other things, appropriate thermal and electrical boundary conditions for the numerical simulation of the VAR of 36" Ti-6-4 ingots, the following were observed: the ingot is over half liquid at power-off from full power (i.e., no "hot-topping" power cut-back was used, as it would have been for a production ingot); the "steering" of the VAR arc by the stirring magnetic fields is clearly evident; magnetic probe data show non-zero time averaged fields and non-uniform stirring. In seeking to simulate the experimentally established solid/liquid boundaries for the above, considerable care is necessary in choosing those parameters of the process which are not established by independent measurement. Among these are model turbulence intensity, coefficient of thermal expansion, fraction of arc current entering pool surface. If solenoidal windings develop full strength stirring magnetic induction Bs when the furnace arc is operating, the simulated flows can change from cases dominated by flow down the pool sidewall to cases dominated by flows down the ingot axis (as required by experiment) for quite plausible parameter values. These results suggest that a judicious mix of experiment and simulation is crucial to making realistic predictions about thermal conditions in such VAR cases. Axisymmetric '2-1/2D' simulations run to date using boundary conditions of the same type as reported for superalloy melting indicate large liquid volumes, but have not yet reproduced details of the pool shape.

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Comparison of Induction Slag Melted and Commercial Ti-6Al-4V Alloys: *Alan D. Hartman*[1]; Karol K. Schrems[2]; Gordon R. Holcomb[2]; Edward R. Argetsinger[1]; Jeffrey S. Hansen[1]; Jack I. Paige[1]; Paul C. Turner[1]; [1]Albany Research Center-DoE, Therm. Treat. Tech., 1450 Queen Ave. S.W., Albany, OR 97321 USA; [2]Albany Research Center-DoE, Matls. Conserv. Div., 1450 Queen Ave. SW, Albany, OR 97321 USA

The Albany Research Center of the U.S. Department of Energy has been investigating a means to form useful wrought products by direct and continuous casting of titanium bars using cold-wall induction melting rather than current batch practices such as vacuum arc remelting. Continuous ingots produced by cold-wall induction melting, utilizing a bottomless water-cooled copper crucible, without slag (CaF2) additions had minor defects in the surface such as "hot tears". Slag additions as low as 0.5 weight percent were used to improve the surface finish. Therefore, a slag melted experimental alloy ingot was compared to a commercial alloy Ti-6Al-4V ingot in the areas of physical, chemical, mechanical, and corrosion attributes to address the question, "Are any detrimental effects caused by slag addition?".

3:15 PM Break

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Study and Modeling of the Electron Beam Melting Process: *J. P. Bellot*[1]; H. Duval[1]; S. Besse[2]; [1]Laboratoire de Science et Genie des Materiaux Metalliques, Ecole des Mines, Parc de Saurupt, Nancy, Cedex 54042 France; [2]SNECMA, Matls. and Process. Dept., Centre de Villaroche, Moissy-Cramayel 77550 France

Since 1991 we have undertaken a complete study of the Electron Beam Melting process, with a particular emphasis on the cold hearth melting of the titanium alloys. Resulting from this research work, three comprehensive numerical models have been set up concerning the molecular gas dynamics of the vapor phase, the thermo-hydrodynamic and solute behavior of the alloy in the cold hearth and dissolution kinetics of the low density inclusions. The paper describes the main results obtained which are focused on: the volatilization losses of the alloying elements and the reduction of these losses by addition of an inert gas, the thermo-hydrodynamic behavior of the liquid pool taken into account the Marangoni and thermal natural convections, the history of the low density inclusions in the hearth (such as hard-alpha) in terms of trajectory and dissolution, and the effects of the beam scanning frequency on the transient thermal behavior of the surface of the liquid pool.

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Mathematical Modeling of the Electron Beam Cold Hearth Refining of Titanium Alloys: *Shesh K. Srivatsa*[1]; [1]GE Aircraft Engines, Matls. and Process. Eng. Dept., One Neumann Way, Mail Drop M87, Cincinnati, OH 45215-1988 USA

Electron Beam Cold Hearth Refining (EBCHR) is used to refine titanium alloys for critical aircraft engine rotating parts. In this paper, a computational model is presented for predicting the fluid flow, heat transfer, phase change, and the motion of inclusions in an EBM hearth. The model uses a control volume method for the discretization and solution of the momentum, continuity and energy equations to predict the flow and temperature fields within the hearth. The model accounts for all the important physical phenomena influencing the process, including temperature-dependent Marangoni and buoyancy effects and radiation heat transfer. A Lagrangian tracking approach is used to predict the inclusion trajectories and their change in size due to dissolution. The model has been applied to analyze an EBM hearth operating at different power conditions. The predicted surface temperatures and the pool shape compare well with the experimental measurements. The model can be used to develop insights into the underlying physical phenomena occurring in the EBCHR process, analyze the effect of process parameters on inclusion removal effectiveness, and ultimately achieve better design and control of the overall process.

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A Spectroscopic and Electrochemical Study of Titanium Electrorefining: *Luis Ortiz*[1]; Donald R. Sadoway[1]; [1]Massachusetts Institute of Technology, Dept. Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 8-109, Cambridge, MA 02139-4307 USA

Sputtering targets for the microelectronics industry are made by electrorefining metallurgical grade titanium in a molten chloride electrolyte. The feedstock is titanium sponge produced by the Kroll or Hunter process. The kinetics of metal deposition are being investigated by spectroscopic (visible and Raman) and electrochemical (voltammetric and impedance) techniques. Sponsorship of the research from The ALTA Group, JM Electronics, is gratefully acknowledged.

Rare Earths and Actinides; Science Technology and Applications IV: Rare Earths II: Processing

Sponsored by: Light Metals Division, Reactive Metals Committee

Program Organizers: Renato G. Bautista, University of Nevada-Reno, Department of Chemical and Metal Engineering, Reno, NV 89557-0136 USA; Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA

Wednesday PM — Room: Lincoln E
March 15, 2000 — Location: Opryland Convention Center

Session Chairs: Danesh Chandra, University of Nevada, Metall. and Matls. Eng., Reno, NV 89557 USA; Seshadri Seetharaman, Royal Institute of Technology, Dept. of Metallu., Stockholm SE-100 44 Sweden

2:00 PM

The Preparation of Gd//5(Si//x/Ge//1-x/)//4/Magnetic Refrigerant Materials from Commercial Gadolinium Metal: *Karl A. Gschneidner*[1]; Alexandria O. Pecharsky[1]; Vitalij K. Pecharsky[1]; [1]Iowa State University, Ames Lab., 255 Spedding, Ames, IA 50011-3020 USA

About three years ago we discovered the giant magnetocaloric effect materials Gd//5/(Si//x/Ge//1-x/)//4/, where x≤0.5. These materials have great promise as useful magnetic refrigerant alloys for a wide range of applications-building air conditioning, supermarket chillers, frozen food processing plants, automotive climate control, etc. To date most of the samples have been prepared by arc-melting using high purity (99.8 at.%) Ames Laboratory Gd metal. The sample sizes are of the order of 50g or less. If commercialization is to be realized, one will need to use inexpensive commercial grade Gd (95 to 98 at.% pure with major impurities of O and C) and a melting process which can be used to prepare large quantities (1kg or larger) of the Gd//5/(Si//x/Ge//1-x/)//4/alloys. We found that carbon impurities are detrimental by preventing the formation of the monoclinic phase and thus destroy the giant magnetocaloric effect in Gd//5/(Si//x/Ge//1-x/)//4/for x≤0.5. This paper will discuss the development of processes to overcome these and other difficulties. Kilogram quantities of the material which exhibits the giant magnetocaloric effect have been prepared. The resultant magnetocaloric effect is slightly less than that obtained by arc-melting high purity Gd metal with Si and Ge. Supported by US DOE, Office of Basic Energy Sciences, Div. of Mats. Scis., under Contract No. W-7405-ENG-82.

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Thermal Aging Studies of $LaNi_{4.25}Al_{0.75}$ Hydrides and Tritides: *D. Chandra*[1]; W. N. Cathey[1]; D. Clare[1]; H. Mandalia[1]; J. R. Wermer[2]; J. S. Holder[3]; W. C. Mosley[3]; [1]University of Nevada, Metallu. and Matls. Eng. Div., Mackay School of Mines, Mail Stop 388, Reno, NV 89557 USA; [2]Los Alamos National Laboratory, Mail Stop C 348, Los Alamos, NM 87545 USA; [3]Westinghouse Savannah River Company, Savannah River Tech. Ctr., Aiken, SC 29808 USA

The $LaNi_5$ type hydrides are important for applications such as hydrogen/tritium storage systems and others. In this study, thermal aging of $LaNi_{4.25}Al_{0.75}$-hydride was performed in the range of 473 to 583K, and $LaNi_{4.25}Al_{0.75}$-tritide at room temperature. The hydrogen aging experiments performed at 473K, showed a rapid initial decrease in hydrogen pressure followed by gradual decreases in pressure; the initial hydrogen pressure was 1930 kPa. Isotherms taken at 389K, after hydrogen thermal aging at 473K, showed that the desorption pressure of the isotherm decreased by approximately 45 kPa. Vacuum annealing of $LaNi_{4.25}Al_{0.75}$ showed that the plateau pressure decreases were not due to metallurgical heat treatment but because of hydrogen interactions. Similar decreases in hydrogen pressures were obtained from studies on thermally aged $LaNi_{5-x}M_x$ with Sn (x=0.24) and Mn (x=0.4) substitution. Thermal aging at 563K and 583K also showed similar trends as that of 473K aging, except that desorption isotherms developed slopes at lower H/M values; the absorption isotherms were unaffected. Room temperature aging of $LaNi_{4.25}Al_{0.75}$-tritide for 6.8 years showed that the desorption isotherms developed a steep slope. During tritium aging, helium-3 formed due to radiolytic decay and remained trapped in the metal lattice. This resulted in non-uniform strain in the lattice which broadened the X-ray diffraction Bragg peaks. The thermal aging results will be discussed.

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Hydrogen Amorphization of $GdFe_2$ Laves Phase Hydride: *Dhanesh Chandra*[1]; Ricardo B. Schwarz[2]; [1]University of Nevada, Metallu. and Matls. Eng. Div., Mackay School of Mines, Mail Stop 388, Reno, NV 89557 USA; [2]Los Alamos National Laboratory, Matl. Sci. and Tech. Div., Mail Stop G755, Los Alamos, NM 87545 USA

We have investigated the effect of hydrogen interaction with $GdFe_2$ Laves phase alloy. Crystalline hydrides are formed at relatively low temperatures and pressures, and the reaction is reversible. A $GdFe_2H_{4.8}$ is formed at room temperature, which is reversible, with a H/M ratio of 1.6. However, at this temperature desorption of all the hydrogen from the hydride is difficult because of slow kinetics and, for pressures below 10^3 Pa, the desorption isotherm deviates from the absorption isotherm. An amorphous $GdFe_2H_x$ phase forms at intermediate temperatures and pressures. The absorption isotherm for this hydriding reaction is quite unusual in that, as the crystal-to-amorphous transformation is accompanied by either a gain or a loss of hydrogen. Absorption isotherms taken below 475K showed that there is an abrupt decrease in the hydrogen capacity of the alloy during the crystalline-to-amorphous hydride phase transformation. Whereas the absorption isotherms taken above 475K showed that there is an abrupt increase in the hydrogen capacity during amorphization. At temperatures above 525 K, hydrogen absorption causes to the disproportionation of the $GdFe_2$ crystal into a two-phase mixture of GdH_2 and bcc α-Fe. The formation of the crystalline and amorphous GdFe2Hx phases, phase stability regions, disproportionation of the hydride will be discussed.

3:30 PM Break

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Metallothermic Processing of Nd from NdF3: *R. S. Chiou*[1]; R. G. Reddy[2]; [1]The University of Alabama, Chem. Eng., Tuscaloosa, AL 35487 USA; [2]The University of Alabama, Metallu. and Matls. Eng., Tuscaloosa, AL 35487 USA

Production of neodymium metal by the reduction of neodymium fluoride with sodium as a reductant in presence of NaCl-NaF flux was studied. Process calculations were carried out using Gibbs energy minimization method. The effect of process parameters such as temperature, pressure, salt and feed materials composition on the yield and impurity content of the products were analyzed. The yield of Nd metal increased with an increase in temperature and the fluxing agents and so did the impurity content of the metal. An excellent agreement was obtained between the experimental and the calculated data. The results were also compared with other metallothermic processes for the production of neodymium.

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Kinetics Studies of Nitridation of Fe17-Nd2 Alloys: V. V. Hong[2]; R. E. Aune[1]; *Seshadri Seetharaman*[1]; [1]Royal Institute of Technology, Dept. of Metallu., SE-100 44, Stockholm, Sweden; [2]The Vietnam Center for Science and Technology, Instit. of Matls. Sci., Dept. of Rare Earth Met., Hoang Quoc Viet-Cau giay, Hanoi, Vietnam

The magnetic properties of Fe-Nd alloys are significantly improved by the introduction of interstitial elements like boron and nitrogen. In the processing of these property optimized magnetic alloys, it is important to have access to the kinetic information regarding the nitrogen uptake by these alloys at the process temperatures. The present work was undertaken to study the kinetics of nitridation of Fe17-Nd2 magnetic alloys in the temperature range 1173-1473 K. Thin plates of the high purity alloy (10 mm diam, initial weight between 207 and 463 mg) were used. The increase in mass of the sample was followed using a SETARAM, TAG 92 unit, as a function of time at 1173, 1273, 1373

and 1473K and nitrogen partial pressures of 100%, 75%, 50%, and 25%(vol%). Nitrogen gas was purified so that the partial pressure of oxygen was < 10 -23 bar. The incubation period was a function of temperature, followed by a rapid nitridation period, and a slow down in the reaction rate due to the formation of product layer. Oxygen impurity have a strong influence on the reaction kinetics. The activation energy for the rate controlling step during the initial stages of nitridation was evaluated. The reaction mechanism and the effect of oxygen impurity on the reaction rate are discussed.

5:00 PM
Gaseous Fluorination of Metal Hydrides: *M. L. Anderson*[1]; I. E. Anderson[1]; J. C. Foley[1]; [1]Iowa State University, Ames Lab., Metallu. and Cer., Ames, IA 50011 USA

Corrosion of the metal hydride electrode in nickel-metal hydride (Ni/MH) rechargeable batteries is a common cause of failure. The primary cause of the corrosion is the interaction with the caustic potassium hydroxide (KOH) electrolyte solution. A method has been developed to react the lanthanum contained within the metal hydride material with nitrogen trifluoride gas (NF3), creating a lanthanum fluoride (LaF3) layer at the surface of the powders. Development of a consistent passivation layer would enable manufacturing of longer lasting Ni/MH batteries. The method used to create this coating and results pertaining to the effect the coating has on the ability of the material to perform as a battery electrode will be described. The Materials Science Division of DOE/BES provided funds for this research under contract W-7405-Eng-82.

Research and Development Efforts on Metal Matrix Composites: Processing of MMCs

Sponsored by: Joint ASM-MSCTS/TMS-SMD Composites Committee; Young Leaders Committee
Program Organizers: John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; Warren H. Hunt, Aluminum Consultants Group Inc., Murrysville, PA 15668 USA

Wednesday PM Room: Bayou A
March 15, 2000 Location: Opryland Convention Center

Session Chairs: Warren H. Hunt, Aluminum Consultants Group, Murrysville, PA 15668 USA; James C. Foley, Ames National Laboratory, Ames, IA 50011-3020 USA

2:00 PM Invited
Status, Issues and Opportunities in Processing of Metal Matrix Composites: *Glenn S. Daehn*[1]; [1]The Ohio State University, Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

If one starts by considering the optimization of metal matrix composite properties, a list of technical issues that must be dealt with through processing can quickly emerge. Concerns include: uniform dispersion of reinforcement, scale of reinforcement and microstructure, thermodynamic compatibility, mismatch in thermal expansion to name a few. In some way these issues can work either with or against the composite and process designer and must be considered together when designing a composite and its manufacturing process. This presentation will consider the limiting cases of how one might create a composite (solid-consolidation, reaction processing, solidification of slurries, and direct deposition). From this an attempt will be made to point out under-exploited opportunities for reducing cost and improving performance in metal matrix composites.

2:30 PM
In-Situ Nondestructive Evaluation Method for Characterizing Sintering of Metal Matrix Composites: *James C. Foley*[1]; David K. Rehbein[1]; [1]Ames Laboratory, Metallu. and Cera. Pgm., 122 Metals Dev., Ames, IA 50011 USA

Renewed interest in the area of metal matrix composites has spurred the development of technologies to enable the production of low-cost metal matrix composites. One such technology that is being developed at the Department of Energy's Ames Laboratory is an in-situ nondestructive evaluation method to characterize sintering. The new method consists of a high temperature EMAT to measure the amplitude and velocity of an ultrasonic tone burst traveling through a sample during sintering. Samples of Al-4 wt% Cu + 10%, 20% and 30% SiC respectively were examined with the new method. In addition, the samples were examined with standard metallography, density measurement, thermal analysis and mechanical testing techniques. Comparison of obtained results demonstrates that density and the extent of sintering can now be directly observed as a function of time with the new technique. Support from an Ames Laboratory directed research and development grant and DOE-BES-DMS under contract no. W-7405-Eng-82 is gratefully acknowledged.

2:50 PM
Thermodynamics of In-Situ Reaction between TiO2 and Pure Al: *Iulian Gheorghe*[1]; Henry J. Rack[1]; [1]Clemson University, Cer. & Matls. Eng. Dept., 204 Olin Hall, Clemson, SC 29634 USA

The reaction between the matrix and reinforcement is normally considered deleterious in the fabrication of MMC's. Indeed major efforts have been made to avoid matrix-reinforcement reaction Al-SiC, Al-Graphite, etc. However it is possible to fabricate thermodynamically stable systems by recognizing the inevitability of these reactions and fabricating composites that allow the reactions to progress to completion. This presentation summarizes the thermodynamic design basis for the in-situ formation of Al2O3 reinforced (Ti3Al, TiAl, Al3Ti). Thermodynamic calculations show that the reduction of TiO2 to Al2O3 and the formation of titanium aluminum intermetallic compounds Ti3Al, TiAl, Al3Ti is possible within a large temperature range. Isothermal Ti-Al-O cross sections at 800°C and 1100°C indicate that either Ti3Al + Al2O3, TiAl + Al2O3, and Al3Ti + Al2O3 two-phase equilibrium, or Ti3Al + Al2O3 + alpha-Ti, Ti3Al + TiAl + Al2O3, and Al + Al3Ti + Al2O3 three-phase equilibrium is possible. Furthermore the calculated Al-TiO2 pseudo-binary phase diagram was calculated and is showing the stability of Al2O3 + Ti3Al, Al2O3+ TiAl, Al2O3+ Al3Ti, Al2O3+ Ti3Al + TiAl, and Al2O3+ TiAl + Al3Ti phase regions up to very high temperatures creating in the same time the base for the design of quantitative experiments. This work is supported by the Office of Naval Research and coordinated by Dr. S. Fishman, under contract No. 96PR07712-00.

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High Strain Rate Superplasticity of In-Situ ZrAl3 Fiber Reinforced Aluminum Composites: *Tsunemichi Imai*[1]; Jianfu Mao[2]; Lin Geng[2]; Sumito Kojima[3]; [1]National Industrial Research Institute of Nagoya, 1-1 Hirate-cho, Nagoya 462-8150 Japan; [2]Harbin Institute of Technology, Harbin 150001 PRC; [3]Nagoya Municipal Industrial Research Institute, 3-4-41 Rokuban-cho, Atsuta-ku, Nagoya, Japan

Nano ZrO2 particles used as reinforcement were mixed with pure aluminum powder (1N90 and sintered at 823K under the pressure of 285MPa for 20 minutes. This produced ZrAl3 fiber reinforced 1N90 pure aluminum composite with fiber diameter less than 1mm by reaction of ZrO2 with aluminum. The volume fractions of ZrO2 selected were 0.05, 0.10 and 0.15. Sintered ZrAl3/1N90 Al composites were extruded with the extrusion ratio of 44:1 at 823K. The extruded ZrAl3/1N90 composite(Vf=0.05) exhibited an m value more than 0.3 and a total elongation of about 150% at the strain rate of 0.1/sec at 923K. Hot rolling the composite after extrusion increases the total elongation to about 200% at 0.1/sec and at 913K. TEM micro-characterization clarifies that the fine fiber should be ZrAl3 and grain size of ZrAl3/1N90 composite is 2~3mm.

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Interface Design Strategies for High Temperature Composite Systems: *Joon Sik Park*[1]; John H. Perepezko[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., 1509 Univ. Ave., Madison, WI 53706 USA

A clear understanding of the basic factors controlling reproducible composite processing is critical for the high temperature application. Since the usual composite materials represent ternary or higher order systems, model ternary systems including potential reinforced materials such as B4C and SiC phases have been examined in order to provide effective strategies for the composite materials design. One attractive approach to obtain the stable phase combination (i.e. compatibility) is to produce the desired phase combination by using controlled interface reaction to achieve in-situ synthesis. For example, if the reaction between matrix and reinforcement materials does not yield a preferred phase combination, the diffusion path can be biased to produce the desired phase combination. An effective approach to control the reaction products and diffusion pathway by adding an extra component layer as a kinetic bias has been developed and investigated based upon flux calculations and a semi-empirical database. The growth kinetics of the product phases and the effect of the biasing layer during interdiffusion reaction will be presented for Ni/SiC and TiSi2/TiAl systems. The influence of the kinetic bias includes not only a control over the phase selection and sequencing, but also a control over the microstructural morphology that develops during interdiffusional reactions. An analysis of the diffusional interactions in terms of component chemical potential variations provides useful general guidance for reaction path control and the limiting kinetics. With an interface design strategy based upon a diffusion path directed by kinetic biasing to include thermodynamically stable phase combinations, the resulting in-situ synthesis offers a self-healing of internal damage to yield robust composite performance. The support of ONR (N00014-92-J-1554) is gratefully acknowledged.

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Reactive Infiltration Processing and Compression Creep of NiAl and NiAl Composites: *T. A. Venkatesh*[1]; David C. Dunand[2]; [1]MIT, Matls. Sci. and Eng., Rm. 8-139, 77 Massachusetts Ave., Cambridge, MA 02139 USA; [2]Northwestern University, Matls. Sci. and Eng., 2225 N Campus Dr., MLSB 1123, Evanston, IL 60208 USA

Reactive infiltration processing of bulk and composite NiAl was investigated with powder and wire preforms of nickel. Inhomogeneous microstructures were often obtained with powder preforms because their high surface-to-volume ratio, low permeability, and irregular infiltration paths lead to simultaneous infiltration and reaction. Homogenous NiAl could be obtained with nickel-wire preforms which had a lower surface-to-volume ratio, higher permeability, and regular infiltration paths, because infiltration was completed before the onset of reaction. Composites with continuous tungsten (W) and sapphire fibers were also successfully fabricated by reactive infiltration, while composites with molybdenum particulates and short-fibers showed significant dissolution in NiAl. The high-temperature uni-axial compression creep behavior of uni-directionally reinforced continuous fiber composite materials was investigated using NiAl-W as a model system for the case where both the NiAl matrix and the tungsten fiber underwent plastic deformation by creep. The creep behavior of the constituents NiAl and W and NiAl composites reinforced with 5-20 volume% W was characterized at 1025°C and 715°C. At 1025°C, the NiAl-W composites exhibited three stage creep behavior with distinct primary, secondary, and tertiary creep, where the composite creep-rate decreased monotonically, remained constant, and increased rapidly, respectively. At 715°C, the NiAl-W composites exhibited insignificant primary and tertiary creep but significant secondary creep. Microstructurally, primary and secondary creep were characterized by pure uni-axial compression of tungsten fibers while brooming, bulging, buckling, and kinking were four fiber deformation modes that contributed to tertiary creep. The observed secondary creep behavior correlated well with the rule-of-mixtures isostrain model developed for composites where both phases undergo creep deformation while new models were developed for the composite primary and tertiary creep and were correlated reasonably well with the experimental results.

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Excimer Laser Shock Processing on Metal Matrix Composites: *James G. Cross*[1]; Jong-Kook Park[1]; Kali Mukherjee[1]; [1]Michigan State University, Matls. Sci. & Mech., High Energy Laser Process. Lab., East Lansing, MI 48824 USA

Particulate reinforced metal matrix composites (PRMMC's) have received strong interest due to the potential advantages over monolithic metal alloys in numerous engineering applications. However, the PRMMC's retain high levels of residual stresses, due to a mismatch of coefficient of thermal expansion (CTE), between a ceramic reinforcement and a metal matrix. The residual stresses result in surface crack initiation, which limits structural application of metal matrix composites. Laser shock processing (LSP) is a unique way to locally alter the residual stress profile on a metal matrix composite. By using a very short laser pulse with high energy density, LSP can generate surface plasma that induces high-pressure stress wave propagation. The residual stresses can be relieved on the surface by incident shock waves, which are generated by the confined expansion of the high-pressure plasma. A KrF excimer laser is used for the pulsed laser irradiation, on a stir-melt processed alumina/6061 aluminum composite. An x-ray diffractometer is used for measurements of the residual stresses.

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Friction-Stir Welding of Metal-Matrix Composites: Aluminum 6061-20% Al_2O_3 and Aluminum A339-10% SiC: *Lawrence E. Murr*[1]; Ying Li[1]; Elizabeth A. Trillo[1]; [1]The University of Texas, Metallu. and Matls. Eng., 500 W. University Ave., El Paso, TX 79968-0520 USA

Friction-stir welding (FSW) involves deformation-induced dynamic recrystallization to facilitate solid-state, superplastic flow. In the joining of aluminum alloys such as 6061, heat affected areas cause reductions in residual strength and hardness of nearly 50%, and strategies to reduce this behavior have been only partially successful. However, when Al_2O_3 particles are added to aluminum alloy 6061, the initial yield stress and hardness are increased; nominally by 33% at 20% particle additions. When aluminum alloy 6061 + 20% Al_2O_3 MMC is friction-stir welded, there is a reduction in hardness just outside the weld zone of 58%. However, in contrast to 6061 Al, the minimum residual hardness is reduced by only 9%. Similar results are obtained for the FSW of aluminum alloy A339 (11% Si, 1% Cu, Mg, and Ni, 0.5% Fe, balance Al) + 10% SiC; and this MMC welded to the Al 6061 + 20% Al_2O_3. In each case the hard particles are homogeneously stirred into the weld zone, which has been dynamically recrystallized. Examination and comparison of the microstructures associated with FSW in these systems not only provides some examples of the technological potential for FSW in joining complex and dissimilar MMC systems, but also the opportunity to examine particulate flow phenomena in solid-state, extreme deformation processing. This is especially notable in the examination of intercalated flow in the Al-6061-20% Al_2O_3/Al-A339-10% SiC system which is facilitated by differential etching in optical metallography. Implications for friction processing such as friction extrusion reforming of previously formed MMC are also presented. Research supported in part by a NASA Cooperative Agreement (NCC8-137) and by a General Services Administration grant.

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Distribution of Reinforcing Particles in MMCs Produced by Spray Atomization and Deposition: *Qingzhou Xu*[1]; Enrique J. Lavernia[1]; [1]University of California, Chem. and Biochem. Eng. and Matls. Sci., Irvine, CA 92697 USA

Aluminum-based MMCs are synthesized by spray atomization and deposition combined with a co-injection method or a stirring method. The present work attempts to provide insight into the distribution of reinforcing particle in the different processing approaches. For the co-injection method, it is easy to incorporate SiC particles into the spray of metallic droplets, but the aggregation of SiC particles often is very severe as a result of the non-uniform spatial distribution of particles which come from several isolated injectors. For the stirring method, it is difficult to add SiC particles into aluminum liquid due to the effect of surface tension. However, the particle distribution is very uniform in the matrix since MMCs are generated through the accumulation of individual droplets containing SiC particles. Additionally, the

interactions between SiC particles and the moving solid-liquid solidification interface as well as their influence on the final distribution of reinforcing particles are analyzed theoretically.

Surface Engineering in Materials Science I: Coating/Films Properties Evaluation (PE)-II

Sponsored by: Materials Processing and Manufacturing Division, Surface Engineering Committee
Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Orlando, FL 32816 USA; Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Brajendra Mishra, Colorado School of Mines, Kroll Institute for Extractive Metals, Golden, CO 80401-1887 USA; John Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA

Wednesday PM Room: Canal B
March 15, 2000 Location: Opryland Convention Center

Session Chairs: John J. Moore, Colorado School of Mines, Adv. Coat. and Surf. Eng. Lab., Golden, CO 80401-1887 USA; John D. Demaree, Army Research Laboratory, Weapons & Matls. Rsrch. Directorate, Aberdeen Proving Ground, MD 21005-5069 USA

2:00 PM
Prediction and Evaluation of Mechanical Properties of Two Laser Surface Processed Low Carbon Steels: *Mary Helen McCay*[1]; Narendra B. Dahotre[1]; John A. Hopkins[1]; T. Dwayne McCay[1]; [1]University of Tennessee Space Institute, Ctr. for Laser App. MS 24, B. H. Goethert Pkwy., Tullahoma, TN 37388 USA

Two low carbon steels were coated with compositional mixes of chromium and chromium/nickel powders and laser surface processed at four different energy levels to investigate the ability to predict, and therefore control, the metallurgical properties. Metallurgical analysis consisted of metallographic observations, hardness tests, x-ray diffraction, SEM and microprobe compositional determination and wear tests. Analytical calculations using processing parameters and compositional mix parameters were employed to predict melt depths and final alloy composition. Nickel and chromium equivalents were then calculated and the phases evaluated based upon the Shaeffler diagram (for lower compositions) and the Fe, Ni and Cr phase diagrams (for higher compositions). This provided the basis for estimations of hardness and wear. Results show the possibility of predicting laser surface processed alloy layer phases and properties.

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Tribological Properties of Diamond Coatings and Their Application to a Machine Element: *Ryo Nawata*[1]; Hitoshi Tokura[1]; [1]Tokyo Institute of Technology, Dept. of Mech. Eng., 204 Ishikawadai Kenkyuzikkento, 2-12-1 O-okayama Meguro-ku, Tokyo 152-8552 Japan

Almost twenty years have past since the synthesis of diamond coatings by chemical vapor deposition (CVD) was completed. Diamond coatings have many excellent properties, such as high hardness, a low friction coefficient and low wear rate. Nevertheless, they have not many uses, except for cutting tools, because polishing diamond coatings consumes much time and money. From a point of view described above, if as-deposited diamond coatings can be used without polishing, they will be applied to many machine elements. Firstly, we examined tribological properties of diamond coatings as the basic research. Diamond coatings were deposited on cemented carbide pins and disks. Diamond coated pins were rubbed against diamond coated disk and stainless steel and titanium disks in different environments. When as-deposited diamond coatings were rubbed against as-deposited diamond coatings, we confirmed that the friction coefficient decreased rapidly. Some studies on this phenomenon have been reported and several mechanisms have been proposed. This phenomenon suggests that as-deposited diamond coatings can be used without polishing. From observation of the surface by scanning electron microscopy (SEM), the change of surface morphology caused this phenomenon. Secondly, making use of this phenomenon, we applied diamond coatings to a machine element such as bearings.

2:40 PM
Study of Corrosion Behavior of Laser Glazed and TiC Laser Coated H13 Die Steel: *Daiel Pirzada*[1]; E. G. Baburaj[1]; M. R. Govindaraju[2]; F. H. (Sam) Froes[1]; [1]University of Idaho, Instit. for Matls. and Adv. Process., 321 Mines Bldg., Moscow, ID 83844-3026 USA; [2]Karta Technology Inc., 1892 Grandstand, San Antonio, TX 78238 USA

There is a growing interest in application of intermetallic and ceramic coatings and surface treatments to extend the die-casting die life. These coatings reduce the molten metal corrosion and erosion to the die. Due to its excellent corrosion and highly abrasion resistant nature titanium carbide is a suitable candidate for coating die-casting dies. Amongst the different surface treatments and coating technologies being presently used, laser surface engineering is very attractive due to its flexibility, depth of penetration, high solidification rates (103-108 K s-1), and production of metastable phases. This paper presents the results of a study involving isothermal corrosion of laser surface treated and TiC laser coated H13 steel under accelerated corrosion conditions. The effects of grain size of the TiC coating and different laser surface treatment parameters on corrosion behavior of H13 die steel in liquid aluminum alloy A390 have been investigated. A significant improvement in corrosion resistance was achieved for H13 steel coated with TiC. The finer grain size and laser shot peening contribute significantly towards improving the corrosion behavior of steel in molten aluminum. Based on metallographic studies and energy-dispersive spectrometry (EDS) the effectiveness of the coatings along with the possible reason for their behavior are presented. The potential applications for laser surface treatment and TiC coatings to address specific industrial problems are also discussed.

3:00 PM
Effect of Deposition Temperature on the Physico-Chemical Behavior of Ti-Al-N Thin Films: *S. Seal*[1]; A. Kale[1]; V. Desai[1]; D. Jimenez[1]; K. Sundaram[2]; N. Dahotre[3]; [1]University of Central Florida, AMPAC & MMAE, Eng. 381, Orlando, FL 32816 USA; [2]University of Central Florida, Elect. and Comp. Eng. Dept., Orlando, FL 32816 USA; [3]University of Tennessee Space Institute, Ctr. for Laser App., Dept. of Matls. Sci. & Eng., MS-24, B. H. Goethert Pkwy., Tullahoma, TN 37388 USA

Nitride based hard coatings are of prime interest in today's cutting tool technology. Ternary (Ti,Al)N with a 1:1 Ti:Al ratio seems to be a promising alternative candidate to the widely used titanium nitride. The major concern of using TiN in high-temperature applications is due to the fact, that it oxidizes rapidly at temperatures above 500°C. In contrast, (Ti,Al)N coatings are characterized not only by high microhardness and dense microstructure, but also by their greater thermal stability. In this study, (Ti,Al)N coatings were deposited onto 316SS substrates under ambient and liquid nitrogen temperatures by dc-magnetron sputtering. Both structure and morphology are greatly affected by the deposition temperatures. The as deposited films were oxidized in a vertical fused-silica tube furnace in pure O_2 atmosphere at 850°C for 3.5 and 7.5 hours. Because of their outstanding properties with respect to hardness, wear resistance, oxidation resistance and corrosion resistance, it seems to be desirable to study the crystal structure, mechanical and surface chemical properties of (Ti,Al)N thin films in detail by using SEM, XRD, AES and XPS.

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Corrosion Resistance of Ion Nitrided AISI 304 and 316L Stainless Steels: *R. Vallerio*[1]; M. Landis[1]; R. Hidalgo[1]; K. Marchev[2]; B. C. Giessen[1]; [1]Northeastern University, Barnett Instit. and Dept. of Chem., Boston, MA USA; [2]Saint Gobain Industrial Ceramics Inc., Norton Diamond Film, Northboro, MA USA

As the technology of ion nitriding stainless steels progresses, it becomes imperative to examine the effect of nitriding on the corrosion resistance of the surface of the treated steel. This work will give some insight into the relationship of the conditions of the nitriding treatment to the corrosion resistance of the surface layer through their effect on its structure. By varying the nitriding conditions, single or multi-phase nitride layers can be obtained, leading to dramatic changes in the corrosion resistance. As reported earlier, a textured tetragonal martensitic phase (mphase) can be produced by nitriding under mild conditions. The corrosion resistance of m phase was compared and found to be superior to that of a mixture of phases (ε and γ) present after higher temperature treatments.

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Wear Behavior of Cr3C2-NiCr Detonation Spray Coating: *Jun Wang*[1]; Sun Baode Li[1]; Yaohe Zhou[1]; [1]Shanghai Jiao Tong University, School of Matls. Sci. & Eng., Shanghai, China

Coatings can be applied to surfaces to improve the surface characteristics over those of the bulk properties and are widely used in tribological applications either to reduce wear and/or to modify friction during contact. One of the foremost coating methods for combating wear is thermal spraying, however, despite its widespread industrial use, little is known about the basic friction behavior and the mechanisms by which such coatings wear. Thus, most thermal spray wear coating applications and developments are based on empiricism. In order to prolong the conticaster roll's life, Cr3C2-NiCr detonation spray coating has been processed on the roll surface in the steelmaking plant of Bao ShanSteel Company. The wear behavior of the coating was studied systematically in this paper. The abrasive and dry frictional wear testing were performed on a pin-on-disk tester. Experiment results show that the wear resistance of the coated samples without the risk of seizure are much better than those of the uncoated at room and elevated temperature with any load and sliding velocity. And the coating wear mechanisms under variation test condition were discussed.

4:15 PM

Development and Testing of Corrosion-Resistant Properties of CVD Mullite Coatings for Silicon Nitride: *Svetlana M. Zemskova*[1]; James A. Haynes[1]; Matthew K. Ferber[1]; Kevin M. Cooley[1]; David P. Stinton[1]; [1]Oak Ridge National Laboratory, Met. and Cer., P.O. Box 2008, 1 Bethel Valley Rd., Oak Ridge, TN 37831-6063 USA

Recently it has been demonstrated that thin (3-5mm) chemical vapor deposited (CVD) mullite 3Al2O3x2SiO2 provides excellent oxidation protection for Si3N4 and SiC in high-pressure steam. However, CVD mullite microstructure and composition may substantially influence the coating properties. It was found that the Al:Si ratio in CVD coatings with "mullite-like" structures can be varied from 1:1 up to 9:1 depending on the deposition conditions. The present investigation is aimed at development of CVD parameters for fabrication of dense, uniform, crystalline mullite coatings with controlled Al:Si ratios. The mechanical properties of as-coated Si3N4 specimens were tested by four-point bending and compared with the properties of non-coated material. The effect of mullite Al:Si ratio on coating microstructure, mechanical properties and oxidation resistance was evaluated.

4:35 PM

Influence of Boron Ion Implantation on the Mechanical Properties of TiN Coatings Deposited by Cathodic Arc Evaporation: *Yao-Can Zhu*[1]; Y. Matusmoto[1]; K. Fujita[1]; N. Iwamoto[1]; N. Nagasaka[2]; T. Kataoka[3]; [1]Ion Engineering Research Institute Corporation, 2-8-1, Tsuda-yamate, Hirakata, Osaka 573-0128 Japan; [2]Ebara Research Company Limited, 2-1,Honfuzisawa 4-chome, Fujisawa 251-8502 Japan; [3]Ebara Corporation, 20-1 Nakasode, Sodegaura, Chiba-ken 299-0296 Japan

TiN coatings were deposited on SUS420J2 steel substrates by cathodic arc evaporation. Boron ions were implanted into TiN coatings at 75keV with a dose range of 1.0 ~ 8.0x1017 ions/cm_2. The composition and crystallographic structure of as-deposited and implanted TiN coating were characterized by X-ray photoelectron spectroscopy and Glancing-angle X-ray diffraction. A nanoindentor was employed to measure the hardness of TiN coatings. Ball-on-disc wear tests were carried out to evaluate wear resistance of as-deposited and implanted TiN coatings. The results of wear tests showed that boron ion implantation resulted in a significant improvement of the wear resistance of TiN coatings.

4:55 PM

Performance Comparison Between Various Carbide & Metal Co-Sputtered Coatings: *F. M. Kustas*[1]; B. Mishra[2]; J. Zhou[2]; [1]Engineered Coatings, P.O. Box 4702, Parker, CO 80134-4702 USA; [2]Colorado School of Mines, Dept. of Metallu. & Matls. Eng., Golden, CO 80401 USA

Carbide coatings have excellent potential for wear applications if their toughness, wear resistance, and durability can be increased. Addition of metals to ceramics is a well know method to increase toughness, as demonstrated by traditional cermet (ceramic/metal) bulk materials technology. Using a similar approach, several different carbide & metal cermet systems were fabricated in coating-form using bias-assisted unbalanced magnetron co-sputtering from a single target. Cermet systems that were fabricated for subsequent evaluation included boron carbide (B4C) & molybdenum (Mo), titanium carbide (TiC) & tungsten (W), TiC/titanium diboride (TiB2) & W, TiC/chromium carbide (Cr3C2) & metals, and silicon carbide (SiC) & metals. Coating elemental composition (by XPS) and structure (by XRD) were measured, while scratch adhesion, microhardness, and wear tests were performed to enable performance comparisons between the different cermet systems. In general, unique performances were measured for different cermet systems. For example, the TiC&W system exhibits excellent wear resistance (non-measurable wear against WC-Co at an initial stress of 1.3 GPa (192 ksi), whereas the B4C & Mo system has extremely high hardness (>5000 HKN). Reasons for the different performances of the cermet systems are proposed and candidate applications for these unique cermet coatings are discussed. Work partially supported by a Department of Energy grant under the Entrepreneur's Technical Assistance (ETAP) Program.

Ultrafine Grained Materials: Mechanical Behavior and Strengthening Mechanisms: II

Sponsored by: Materials Processing and Manufacturing Division, Powder Metallurgy Committee, Shaping and Forming Committee

Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; S. L. Semiatin, Wright Laboratory, Materials Directorate, Dayton, OH 45440 USA; C. Suryanarayana, Colorado School of Mines, Department of Metal and Materials Engineering, Golden, CO 80401 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA

Wednesday PM — Room: Polk A/B
March 15, 2000 — Location: Opryland Convention Center

Session Chair: Ruslan Z. Valiev, Institute of Physics of Advanced Materials, Ufa 450000 Russia

2:00 PM Invited

Creep Inhibition of Ceramic/Ceramic Nanocomposites: *Tatsuki Ohji*[1]; [1]National Industrial Research Institute of Nagoya, Superplastic Nanoscience Lab., Hirate-cho, Kita-ku, Nagoya 462-8510 Japan

The dispersion of nanometer-sized silicon carbide particles into alumina or silicon nitride matrix results in significant improvements in creep resistance. The creep rates of the nanocomposites are about several orders of magnitude lower than those of the monolithic ceramics. In this paper much attention is paid to the interfaces between the intergranular nanoparticles and the matrix, and its role in creep inhibition. It will be shown that the intergranular nanoparticles are rigidly bonded to the matrix by several approaches. The rigid bonding of the interfaces causes the inhibition of grain boundary sliding, leading the remarkably improved creep resistance. The importance of change in grain boundary chemistry by doping the nanoparticles is also emphasized. In addition, below specific stresses, the creep rates are remarkably decayed, suggesting the presence's of the threshold stresses below which creep stops. The estimated threshold stress ranges from ten to several ten MPa, depending on the volume fraction of the nanoparticles. These stresses agree with those predicted from the Ashby's model, where motion of grain boundary dislocations responsible for vacancy nucleation and annihilation is considered to be pinned by hard particles.

2:25 PM Invited

Deformation Model During Equal-Channel Angular Pressing: *Patrick B. Berbon*[1]; [1]Rockwell Science Center, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA

In the last few years, the equal-channel angular (ECA) pressing technique has allowed remarkable achievements in the production of submicrocrystalline (SMC) materials with superb superplastic properties, particularly at low temperatures and high strain rates. Although some of the observed mechanical properties can be explained by the small grain size resulting from the process, other results are more puzzling. It appears they are caused by the specific mode of deformation occurring in ECA processing. In this paper, we are first reviewing the specificity of this metal working technique, and we are then proposing a possible deformation mechanism to explain the observed microstructures, their thermal stability, and the superb superplastic properties.

2:50 PM Invited

Microstructure and Mechanical Properties of Aluminum 5083 Processed by Equal Channel Angular Extrusion: John W. Sinclair[1]; *K. T. Hartwig*[1]; R. E. Goforth[1]; [1]Texas A&M University, Mech. Eng., 800 Swan Place, Worland, WY 82401 USA

Commercial grade Al-5083 was processed in bulk form by equal channel angular extrusion to a submicron sized microstructure with boundaries ranging in size from 0.3 to 0.50 μm and the room temperature strength and superplastic performance at 510°C and 350°C investigated. Processing involved hot working the material at 300°C followed by warm working at 200°C and 170°C to a cumulative processing strain of ~ 13.8. Inclusion of an intermediate solution-heat treatment at 530°C following a processing strain of ~ 9.2 was found to enhance superplasticity. Particle sizes greater than 0.56 μm were found to have an adverse effect on superplastic performance. Microtexture evolution indicated a progressive increase in misorientation angle of the boundaries as deformation strain increased. Conventional superplasticity was achieved at 510°C with elongation to failure over 400% and a strain-rate sensitivity index of 0.35. Marginal superplasticity was achieved at 350°C with elongation to failure over 250% and a strain-rate sensitivity index of 0.23. The measured particle volume fraction of 0.02 was insufficient to adequately stabilize the submicron-size microstructure against grain growth at both temperatures.

3:15 PM

Creep Behavior of Carbon Doped Nanocrystalline Nickel: *W. M. Yin*[1]; *S. H. Whang*[1]; R. Mirshams[2]; C. H. Xiao[2]; [1]Polytechnic University, Dept. of Mech. Eng., Six Metrotech Ctr., Brooklyn, NY 11201 USA; [2]Southern University and A&M College, Dept. of Mech. Eng., P.O. Box 9987, Baton Rouge, LA 70813 USA

Nanostructured nickel processed by pulse plating exhibits an excellent combination of full density, uniform grain size and ultrahigh tensile strength. Nevertheless, it shows room temperature creep under high stress conditions. It appears that the room temperature creep might be associated with high grain boundary diffusion. From this prospective, it is interesting to investigate effect of a particular solute element on the creep of this material, in that the solute plays a significant role in the grain boundary characteristics. In this presentation, we will report tensile properties and creep behavior of nanostructured nickel doped with 250ppm carbon. The experimental results showed that Young's modulus has been improved significantly by adding 250ppm carbon while the tensile ductility and yield strength decreased at room temperature in contrast with the solute hardening in the polycrystalline nickel. The tensile creep tests showed enhanced creep strain rate at room temperature and 373K under the stress ranging from 200MPa to 800MPa compared with that of pure nanocrystalline nickel. Further creep tests will be performed at low loads 50-100MPa and higher temperatures. The conventional TEM has been employed to investigate microstructures of the crept specimens. Effect of carbon on creep mechanisms at various conditions will be discussed based on mechanical testing results and microstructure characterization.

3:35 PM Break

3:45 PM

Superplastically Sinter-Forged Si_3N_4 and Si_3N_4-SiC Ceramics: *Naoki Kondo*[1]; Yoshikazu Suzuki[1]; Tatsuki Ohji[1]; [1]National Industrial Research Institute of Nagoya, Superplastic Nanoscience Lab., Hiratecho, Kita-ku, Nagoya 462-8510 Japan

The microstructures and mechanical properties of Si_3N_4 and Si_3N_4-SiC, produced by a superplastic sinter-forging technique from submicron Si_3N_4 or Si-C powders, were investigated. Both the obtained materials exhibited highly anisotropic microstructures, where rod-shaped Si_3N_4 grains tended to be aligned perpendicularly to the forging direction. In addition, for the Si_3N_4-SiC material, β-silicon carbide grains with micrometer-size and nanometer-size were found at the grain boundaries and within the Si_3N_4 grains, respectively. Very high bending strength as well as high fracture toughness were achieved when a stress was applied perpendicularly to the pressing direction.

4:05 PM

Superplastic Microstructure of Modified AA-5083 Aluminum Alloy Processed by Equal Channel Angular Extrusion: *Darrell R. Herling*[1]; Mark T. Smith[1]; [1]Pacific Northwest National Laboratory, Matls. Process., 902 Battelle Blvd., Mail Stop: P8-35, Richland, WA 99352 USA

Current processing methods that are used to develop fine-grained superplastic microstructures in aluminum alloys involve extensive hot and cold deformation steps, usually in the form of hot and cold rolling. This approach has distinct limitations that can have a significant influence on the cost and quality of superplastic forming (SPF)-grades of aluminum sheet. First, the extensive cold rolling required for SPF aluminum sheet typically results in substantial edge cracking and overall yield losses. The second limitation is that the high levels of hot and cold work necessary to achieve the desired microstructure requires starting with very large ingot size, while the final product is usually limited to thin gage sheet. In addition, through conventional rolling thermal-mechanical-processing (TMP) schedules, the microstructure is typically limited to 5-10 micrometer grain size. An improvement in SPF performance can be achieved with the development of smaller-grain equiax microstructure ~1 micrometer. The Equal Channel Angular Extrusion (ECAE) process offers several potential advantages in the processing of SPF-grade aluminum alloys. The ability of the ECAE process to achieve high levels of work through localized shearing can develop a well defined subgrain structure and provide a mechanism for distributing the eutectic constituent particles and dispersoids that play a critical role in the recrystallization process and resulting thermally stable fine-grain size. In addition, with ECAE there is the unique ability to achieve these desirable microstructures in bulk form, without reducing the dimensions of the starting material, as is the case in conventional processing of SPF materials. The objective of this work was to process, via ECAE, a 5000-series aluminum alloy in bulk form to produce a fine-grain (~1 micrometer), thermally stable SPF microstructure. Previous work performed at Pacific Northwest National Laboratory on modified 5000-series alloys identified several compositional features that assist in developing a fine, thermally stable microstructure required for SPF. These modification, which include an increase in Mn level and the addition of Zr, develop fine dispersoids that assist in grain refinement and control excessive grain growth at SPF temperatures. Mechanical

tensile testing was conducted to evaluate the SPF properties of the ECAE processed materials.

4:25 PM

An Evaluation of the Applicability of Theoretical Models for Elevated Temperature Plasticity to Ultrafine Grained Materials: *Rajiv S. Mishra*[1]; [1]University of Missouri, Dept. of Metallu. Eng., 218 McNutt Hall, Rolla, MO 65409-0340 USA

In the last ten years synthesis of ultrafine grained materials, including nanocrystalline, has opened up the possibility of extending our knowledge of grain size dependent phenomenon to a much finer microstructural scale. Superplasticity and creep deformation are known to be grain size dependent and the mechanistic understanding in the microcrystalline range is fairly good. The emerging experimental data on elevated temperature plasticity in the ultrafine grain size range allows us to evaluate the applicability of these mechanisms in this new domain. The present analysis shows that the kinetics of grain boundary related deformation processes are significantly slower than the theoretical model predictions. Some thoughts on the reason for these discrepancies are presented.

4:45 PM Discussion Break

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				
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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.