10TH INTERNATIONAL SYMPOSIUM ON EXPERIMENTAL METHODS FOR MICROGRAVITY SCIENCE: Session IV

Sponsored by: ASM International: Materials Science Critical Technology Sector, Thermodynamics & Phase Equilibria Committee, Jt. Electronic, Magnetic & Photonic Materials Division/Structural Materials Division, Alloy Phase Committee, NASA Microgravity Sciences

Program Organizers: Robert Schiffman, R.S. Research Inc., Crystal Lake, Barton, VT 5822; Carlo Patuelli, Universita di Bologna, Depatrmento di Fisica, Bologna I-40126 Italy

Tuesday PM Room: Plaza Room A
February 17, 1998 Location: Convention Center

Session Chair: B. Q. Li, Washington State University, School of Mechanical and Materials Engineering, Pullman, WA 99164

2:00 PM

NUMERICAL SIMULATION OF A MONOTECTIC SOLIDIFICATION FRONT: *Y. Arikawa*[1]; J. B. Andrews[1]; [1]University of Alabama at Birmingham, Department of Materials and Mechanical Engineering, Birmingham, AL 35294

Abstract not available.

2:20 PM

A STATISTICAL ANALYSIS OF IDGE/USMP-3 SPACEFLIGHT DATA: *A. Lupulescu*[1]; M. E. Glicksman[1]; M. B. Koss[1]; J. C. LaCombe[1]; L. T. Tennenhouse[1]; [1]Rensselaer Polytechnic Institute, Materials Science & Engineering Department, Troy, NY 12180-3590

Dendritic growth is a solidification phenomenon of considerable interest to materials scientists and processing engineers. Two microgravity spaceflights of the Isothermal Dendritic Growth Experiment (IDGE), to provide convection-free dendritic growth data, were completed on USMP-2 (March 1994) and USMP-3 (Feb./March 1996). Rigorous comparison between these experiments and theoretical predictions should be based on a statistically valid analysis. The first IDGE results from space (USMP-2) indicated some departures from Ivantsov's thermal diffusion solution. Flight data from USMP-3 at a few selected supercoolings (0.26, 0.48, 0.65K) provided dendritic growth data suitable for statistical analysis. We present a comparison between terrestrial and microgravity data using a statistically developed interpretation of dendritic velocities, radii, and Pèclet numbers. Special attention is given to the dendritic scaling factor, $\sigma^*=2\alpha d_o/VR^2$, and its associated mean and standard deviation. USMP-2 data originally suggested that gravity does not influence the value of σ^*, whereas USMP-3 data, when analyzed statistically show a small but distinct dependence on gravity.

2:40 PM

THE MEASUREMENT OF THE G-JITTER AFFECTING AN EXPERIMENT IN MICROGRAVITY: *G. Poletti*[1]; F. Magni[1]; [1]Università di Milano, Istituto Fisiologia Generale e Chimca Biologica, Milano 20134 Italy

The instruments used to measure the microgravity and the, g-jitter on board of many different Space Mission are reviewed. Their characteristics and performances are briefly discussed and compared. The attention is focused on the values of the "on board" microgravity as measured by the above instruments and their relationship with the "local" microgravity really affecting an experiment. This on board microgravity is only a rough first approximation value of the microgravity really acting on the experiment that is critically dependent on the position within the payload. The possibility to derived the local microgravity from the values measured for the on board microgravity put forward by some researcher has been ruled out by the conclusions of the studies performed by MUSC on the data recorded during the EURECA Mission. A set of microgravity values drawn out from the data recorded by the SFA experiment on board of EURECA are discussed and compared with the data from the Microgravity Measurement System on board of EURECA. They clearly support the above conclusion and are in qualitative agreement with the operational phases of the mission. In addition they come out to depend on the particular phase of the operation of the SPA instrument itself.

3:00 PM

LIQUID METAL PROCESSING USING THE DROP TOWER AT ZARM, BREMEN, GERMANY: V. Vekris[1]; *R. W. Smith*[1]; [1]Queen's University at Kingston, Department of Materials and Metallurgical Engineering, Ontario K7L 3N6 Canada

Queen's University and the University of Bremen are collaborating in a series of experiments designed to process liquid metals using the ZARM Drop Tower. A flux encapsulation technique has been adopted to examine the influence of gravity on microstructure generation in droplets and so permit deep undercoolings to be achieved. Using the flux encapsulation technique, the specimen is first repeatedly melted and frozen in lg in a halogen lamp furnace to give reproducible undercooling results, the flux acting as a sink for nucleating inclusions, i.e. the specimen develops a nucleation "memory". The specimen is installed in the drop-tower, remelted and then frozen to check its "memory" i.e. to determine whether the earlier obtained undercooling had changed, and finally permitted to cool. The capsule containing the experiment is released approximately 0.2 sec before nucleation so that most of the 4.6 sec of free fall is used to permit the 4-5 mm droplet to freeze under reduced gravity conditions. The results of recent ZAR M-Queen's activities will be described.

3:20 PM Coffee Break in Exhibit Hall

3:40 PM

INFLUENCE OF CONVECTION DURING DIRECTIONAL SOLIDIFICATION OF IMMISCIBLE AL-1N ALLOYS: *L. J. Hayes*[1]; J. B. Andrews[1]; [1]University of Alabama at Birmingham, Department of Materials and Mechanical Engineering, Birmingham, AL 35294

During directional solidification of hypermonotectic Al-In alloys under interfacially stable conditions (high thermal gradient to growth rate ratios), a solute depleted boundary layer is expected to form. Convection in the melt may result due to the destabilizing density gradient brought about by the solutal field. This study will investigate the effect of this convection on the ability to achieve and maintain coupled growth in these alloys. In addition to processing hypermonotectic Al-In alloys under one-g conditions, samples have also been processed on the KC-135 under alternating gravity level conditions. Results from experiments will also be discussed in which the furnace was inverted during directional solidification resulting in a stabilizing solutal field and destabilizing thermal field. Variations in both composition and morphology for all of these processing conditions will be discussed and compared with those reported in off-eutectic coupled growth experiments.

4:00 PM

COMBINED TEMPERATURE AND GRAVITY EFFECTS ON SOLID-LIQUID INTERFACIAL FREE ENERGY: C. Patuelli[1]; *R. Tognato*[1]; [1]Università di Bologna, Dipartimento di Fisica, Bologna I-40127 Italy

The solid-liquid interfacial free energy is influenced by temperature. If the interface is considered a two-dimensional system containing domains, temperature decreases involve interfacial free energy increases. Due to the presence of domains also gravity might have effects on the interfacial free energy: a model to foresee possible combined tempera-

ture and gravity variation effects, in undercooling conditions, on the interfacial free energy has been proposed.

4:20 PM
STUDY OF POWDER DISPERSION PROCESS UNDER MICROGRAVITY: *A. Pyatenko*[1]; H. Takeuchi[1]; S. Chiba[1]; Y. Ohyama[1], [1]Hokkaido National Industrial Research Institute, Sapporo 062 Japan

Particle handling is an essential element of many modern technologies that deal with solid powders. Many steps of this handling, such as particle feeding from hopper or the vertical motion through the reaction zone use gravity as the driving force. Thus they have to be studied under microgravity to find the suitable substitution for absent gravitational force. Although some other steps like pneumatic transportation or classification do not directly depend on gravity, microgravity could provide the unique experimental conditions for more profound study of these processes. The latter situation is valid for powder dispersion. The dispersion process caused by accelerated motion of the gas-solid flow through the orifice was studied. Superficial gas velocity and orifice diameter were varied as the parameters in different experiments. Particle size distribution was measured under microgravity conditions by a cascade impactor and compared with the original one. Special experiments were conducted to prove the possibility of using a cascade impactor in drop capsule. Experiments were carried out using the drop facility of Japan Microgravity Center (JAMIC). As a result, the efficiency of dispersion process was found. It ranged from 0.2 to 0.6 for different experimental conditions.

4:40 PM
AN OPTICAL NUCLEATION MODEL FOR GROWING A SINGLE CRYSTAL IN SPACE: *Hossin Abdeldayem*[1]; Donald O. Frazier[1]; [1]NASA-Marshall Space Flight Center, ES 76, Space Science Laboratory, Huntsville, AL 35812

We have demonstrated that an optical trapping force on the molecules of several organic solutions can be exerted at the focus of a lens by a single low power Ar^{+} laser beam. Formation of clusters on the order of 250μm is diameter were obtained. We, herein, describe a technique for growing protein crystals in space. A two counter propagating beams are focused at one point to induce an optical potential well, where the light of maximum intensity is generated. The molecular condensation at the center creates a highly supersaturated region that initiates nucleation automatically to start crystal growth. A starting seed for crystal growth is not needed. A seed crystal that has been formed in a normal gravity, might not be free of defects and thereby still hinders the growth of a perfect crystal in space. The continuous presence of the optical field during the whole duration of the experiment, causes a continuous migration of the molecules toward the trapping site. This will eliminate the creation of a depletion region and will enhance the crystal growth rate. The protein solution is not required to be supersaturated. The optical field will induce a single site of super saturation to initiate the nucleation. This will eliminate the chance of unwanted nucleation to take place at other sites. The system is safe to operate, can be designed to a compact size and is inexpensive to build.

5:00 PM
NASA'S MICROGRAVITY MATERIALS SCIENCE PROGRAM: *Donald C. Gillies*[1]; [1]Marshall Space Flight Center, Space Sciences Laboratory, Huntsville, AL 35812

Materials Science research programs are funded by NASA through the Microgravity Research Division. Such programs are normally designated as flight definition or ground based and can be awarded initially for up to four years. Selection is through a peer review process with proposals being submitted in response to a biennial NASA Research Announcement (NRA). The next announcement is due in November 1997 with proposals due in March 1998. Topics of special interest to NASA are described in the guidelines for proposal writing within the NRA. NASA's interest in materials is wide and covers a range which includes metals and alloys, ceramics, glasses, polymers, non-linear optics, and nanostructures. With increasing interest in the Human Exploration and Development of Space (HEDS) program, the materials research funded will not be exclusively devoted to processes dependent on microgravity, but will also support materials of strategic interest in meeting NASA's long range plans of interplanetary travel.

ADSORPTION, ION EXCHANGE, AND SOLVENT EXTRACTION: Ion Exchange II

Sponsored by: Extraction & Processing Division, Aqueous Processing Committee, Copper, Nickel, Cobalt Committee, Lead, Zinc, Tin Committee, Precious Metals Committee
Program Organizer: Courtney Young, Montana Tech, Metallurgical Engineering, Butte, MT 59701

Tuesday PM — Room: Plaza Room C
February 17, 1998 — Location: Convention Center

Session Chair: David B. Dreisinger, University of British Columbia, Metals and Materials Engineering, Vancouver, BC V6T 1Z4, Brian Green, Mintek, Mineralogy and Process Chemistry Division, Randburg, 2125 South Africa

2:30 PM Plenary
ANION EXCHANGE RESINS IN GOLD PROCESSING AND CYANIDE RECOVERY - RECENT DEVELOPMENTS AND FUTURE TRENDS: *Chris Fleming*[1]; [1]Lakefield Research, Lakefield, ON K0L 2H0 Canada

Much research and process development work has been performed over the last 50 years to produce anion exchange resins and engineer extraction processes (in solution or pulp) for the recovery of gold complexes from leach solution. Efficient methods of eluting and regenerating the resins have also been developed, allowing them to be recycled in the process. Extensive testwork has shown that resins, if properly handled and treated, are physically very robust in these applications, and resin make up rates can be as low as 10 to 50% of inventory per annum. Most gold leach solutions contain high concentrations of other anions, both simple inorganic anions, as well as anionic metal complexes, and the extent to which these other anions are extracted or rejected by the resins has a great influence on the efficiency of gold extraction. Considerable progress has been made in recent years in the engineering of specific internal resin structures, that significantly alter the selectivity characteristics of resins for gold and other anions. This has enabled the development of processes that not only extract gold quite selectively and very efficiently at the front end of the flowsheet, but also extract cyanide and other toxic anions quite efficiently at the tail end of the flowsheet, producing clean tailings for discharge to the environment. Recent developments in these areas will be discussed. It is apparent that in a number of operations, as well as new projects, these processes could produce more favourable economies than conventional carbon-based or Merrill Crowe processes, and yet the gold mining industry has generally been slow to embrace this technology. The reasons for this will be discussed.

3:15 PM INVITED
RESIN-IN-PULP - AFTER GOLD, WHERE NEXT?: *B. R. Green*[1]; M. H. Kotze[1]; [1]Mintek, Randburg 2125 South Africa

During the past two decades, Mintek has developed various aspects of the resin-in-pulp (RIP) process for gold. The rather complex task of coupling the technical developments to commercial production of a special resin and financing of a full scale demonstration of the technology is about to become reality. It is time to look forward to the potential for re-application of the technology to other situations because the successful implementation of this demonstration will mark the conclusion of RIP's development phase. The main attraction of RIP (i.e., no need to filter or clarify) qualifies situations, where there is some disadvantage to filtration of a pulp, as possible candidates. Some such cases have been identified and have merited preliminary testwork including

(1) recovery of manganese that is difficult to wash from filter cakes, in the production of electrolytic manganese, (2) recovery of zinc from leach residues prior to disposal to slimes dams, in the production of zinc, (3) recovery of nickel from a low grade pulp after precipitation of Iron III with limestone, and (4) removal of metal cyanides from pulps after gold has been recovered. The discussion will include a description of the problem, factors that affect the choice of resin, and factors that will affect the economics of the process.

3:40 PM Coffee Break in Exhibit Hall

3:50 PM INVITED
RECOFLO ION EXCHANGE TECHNOLOGY: *Michael Sheedy*[1]; [1]Prosep Technologies Inc., Subsidiary of Eco-Tec Ltd., Pickering, ON L1W 3T9 Canada

Recoflo ion exchange technology utilizes fine mesh resin beads, a fully packed resin bed, and counter-current regeneration. These unique features help to improve exchange kinetics, reduce regenerant consumption, and increase the concentration of strip solutions. Commercially, Recoflo technology has been extensively used in the following areas: the separation of strong mineral acids from dissolved metal salts; the purification, removal and recycle of dissolved metals in the surface finishing industries; and the production of high purity water. In the mining and metallurgical industries, Recoflo ion exchange systems have been installed to treat a copper refinery bleed stream at Falconbridge's Kidd Creek plant and to treat a nickel and cobalt bearing ammonium sulphate effluent stream at another primary metal producer's facility. In the first application, an APU-Recoflo system is used to separate the sulphuric acid at 200 g/l from the dissolved copper and nickel sulphate salts at 80 g/l. The adsorbed sulfuric acid is recovered from the resin by elution with water and is then returned to the refinery electrolyte circuit. In the second application, a chelating ion exchange resin is used to remove nickel and cobalt at 0.01-3.0 g/l from process streams containing 120-240 g/l ammonium sulphate. This Recoflo system has been designed to reduce the nickel and cobalt levels to 0.001-0.002 g/l while recovering cobalt and nickel at concentrations up to 30 and 15 g/l, respectively. Case studies for both these metallurgical applications and other Recoflo processes are presented in this paper.

4:15 PM INVITED
ION EXCHANGE TREATMENT OF COPPER ELECTROWINNING ELECTROLYTES FOR IRON REMOVAL USING EICHROME'S DIPHONIX RESIN: *D. B. Dreisinger*[1]; R. Shaw[2]; M. J. Gula[3]; [1]University of British Columbia, Metals and Materials Engineering, Vancouver, BC Canada; [2]Eichrome Industries, Inc., Fife, Scotland; [3]Eichrome Industries, Inc., Darien, IL

The conventional approach to iron control in copper electrowinning circuits is to bleed electrolyte. This approach is generally successful but can be quite costly due to the loss of (1) cobalt which is added to electrowinning solutions to protect the lead anodes from excessive corrosion and to lower the anodic oxygen overvoltage, (2) acid which must be replaced with concentrated sulfuric acid especially at sites where acid is not consumed in leaching, i.e., acid producing ores, (3) water which must be of high quality to keep impurities at low concentrations and control the pH upon acid addition, and (4) copper which must be re-extracted from the bled electrolyte. Cobalt and acid make-up costs can be significant. Pure water production and copper re-extraction requires both capital and operating expenses. The Eichrome Iron Control System has been designed to remove iron from copper electrowinning solutions as an alternative to bleeding. The system utilizes Diphonix resin to selectively remove ferric iron. Elution of the resin is accomplished using sulfurous acid to reduce the ferric ion on the resin to ferrous. Ferrous ions are weakly held by the resin and therefore easily eluted. The system provides significant savings because bleeding is reduced by a much as 90%. Additional benefits are realized from lower iron concentrations including improved copper plating current efficiency and therefore lower electrical costs per unit of copper produced. The first commercial installation of the Eichrome Iron Control System has recently been commissioned at Cananea, Mexico. Recent operational data will be presented and discussed along with the control of other impurities such as chloride.

4:40 PM INVITED
IMPLEMENTATION OF A COMMERCIAL-SCALE ION EXCHANGE PROCESS FOR THE CONTROL OF IRON IN THE COPPER ELECTROWINNING CIRCUIT AT MEXICANA DE CANANEA: *Michael J. Gula*[1]; Jose Hector Figueroa[2]; [1]Eichrome Industries, Inc., Darien, IL 60561; [2]Mexicana de Cananea (Sonora, Mexico), Douglas, AZ 85608

During 1997, the first commercial-scale implementation of an ion exchange process utilizing Eichrome's Diphonix resin for the control of iron in the copper electrowinning circuit at Mexicana de Cananea was accomplished. This unit is designed to remove approximately 1 tonne of iron per day from the copper electrolyte. The presentation will discuss our experiences in starting up this new process. The initial operating data from the iron control resin plant and the impact on the copper electrowinning process at Mexicana de Cananea will also be presented.

ALUMINA & BAUXITE: Impurity/Red Mud

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Jean Doucet, Alcan International, PO Box 6090, Montreal, Quebec H3C 3A7 Canada

Tuesday PM Room: Fiesta A
February 17, 1998 Location: Convention Center

Session Chair: TBA

2:00 PM
ENHANCED OXALATE REMOVAL UTILIZING THE MULTIFUNCTIONAL PUROX PROCESS: *Dr. D. Perrotta*[1]; [1]Aluminium Co. of America, Alcoa Technical Center, Point Comfort, Texas 77978-0101 USA

Previous published work by the authors has shown that layered double hydroxides and activated carbon have the capability of removing organic compounds from Bayer liquor. Removal of the effected organic compounds results in the enhanced precipitation of sodium oxalate from treated liquors. The commercial application of this technology has been called the Purox Process. The previous work has been extended in the present study to show that interestingly further enhancement of oxalate precipitation can be obtained by the synergistic effect of adding more than one treating agent.

2:25 PM
SIZING AN ORGANIC CONTROL SYSTEM FOR THE BAYER PROCESS: *Mr. Benny E. Raahauge*[1]; [1]FLS Minerals A/S, Copenhagen Denmark

Organic materials are introduced to the Bayer process by almost all bauxites. Over time, the accumulation of organic matter in some Bayer liquors decreases liquor and plant productivity. The organics level can be limited by some form of liquor purging, or by operating an organics control system. This paper presents a Mass-Balance Model for predicting the rate of increase and decrease of organic concentration levels in response to changes in bauxite, purge rate and/or the commissioning of an organic destruction system. Correct selection and sizing of the organic destruction unit is the key to improve Bayer plant productivity and environmentally acceptable operation. The first Solid-Liquid Calcination (SLC) plant for organic destruction has just been installed at Alumina Espanola, Spain. The performance criteria of this technology will be highlighted.

2:50 PM
REMOVAL OF ORGANIC SUBSTANCES FROM BAYER PROCESS LIQUORS BY MAGNESIUM COMPOUNDS: *Dr. Vadim A. Lipin*[1]; [1]VAMI, Aluminium-Magnesium Institute, St. Petersburg, 199026 Russia

The organic substances passed into spent liquors have an impact on different operations in Bayer process substantially. The main problems of alumina production are associated with color organic substances. Because of this, the color organic substances should be removed from spent liquors primarily. The alkaline earth compounds are attractive as sorbents of organic substances and reusable. The possibility of application a number reasonable magnesium compounds for purification of Bayer process evaporated liquors was considered. The great bulk of organic impurities in liquors was presented by high molecular compounds, carboxylic acids and phenols. The influence of temperature, duration of the treatment, concentration of the putting magnesium compounds on results of the sorbtion organic substances and losses of aluminium was studied. It was established that amount of puttling sorbent have great impact on the sorbtion, and with magnesium aluminate the losses of aluminium are minimum. The optimum parameters of purification for the evaporated liquors of Nikolaev Alumina Plant were found.

3:15 PM Coffee Break in Exhibit Hall

3:30 PM
ADVANTAGES OF PRESSURE FILTRATION OF RED MUD-SAND MIXTURES: *Mrs. Vicenta Roccaro*[1]; [1]Laboratorio de Separaciones Mecanicas, Universidad Central de Venezuela, Caracas 1015-A Venezuela

Red mud must be disposed in great quantities every day in an alumina plant. If only countercurrent settler-washer trains are used, the waste consistence is very low and mud has to be sent to open ponds to promote a proper evaporation of the liquid. Most of the plants using this principle are going in environmental troubles due to this practice. High efficient thickeners as well as vacuum and overpressure filters are sometime used after settler-washer systems in order to dispose the mud with less liquid content. The fine particle content of the red mud leads to low permeabilities of the formed cakes. Experiments were conducted at the Mechanical Separations Lab to determine the cake building and dewatering characteristics of red mud produced from Pijiguaos Bauxite (CVG-Bauxilum). Mixtures of sand and red mud in ratios below the ratio in which both wastes are industrially produced were filtered and dewatered with differential gas pressure, using the transportable filter cell Filtratest-UCV. Although the s and acts as a filter aid, there was almost any improvement in the red mud production when sand was added, being the high permeability compensated by the great quantity of solids to be filtered. Nevertheless, an important reduction of the moisture content during short dewatering time was found to take place. High sand/mud ratios lead to undesirable sedimentation and segregation effects. The use of pressure differences over the vacuum range show to be very useful until 2 bar, but greater pressure differences are uneconomic. Intermediate compressibility index was determined.

3:55 PM
ADVANCES IN RED MUD DEWATERING AND DISPOSAL TECHNOLOGIES: Mr. Ronald F. Nunn[1]; [1]ICF Kaiser Engineers, Oakland, CA 94612-3430 USA

Red Mud is the number one environmental concern of alumina plants today. A typical alumina plant can produce upwards of a million tons of red mud slurry each year. The large volume and the causticity of the red mud slurry are the principal problems. The search for ways to reduce the cost of mud disposal and the advent of stricter environmental regulations had led to many advances in the technology of red mud dewatering and disposal. A history of these advances is presented. A review and comparison of vacuum filtration, deep thickeners, super thickeners, and hyperbolic filtration is presented in terms of technology, economics and environmental impact. Advances in red mud disposal are also reviewed and compared including transportation to the disposal site, deposition of the mud, landscaping and rehabitability of the site.

4:20 PM
ENHANCING OPERATION OF A DESANDING CIRCUIT WITH HYDROCYCLONES: *Mr. Gerald P. Kelton*[1]; [1]Krebs Engineers, Tucson, Arizona 85743 USA

The sands separation area at Alcan's Burntisland Plant utilized a two stage rake classifier system. Problems, primarily mechanical breakdowns, scale problems, and ineffective performance of the rake classifiers, suggested an alternative approach. After considerable study, hydrocyclones were identified as a solution. The initial phase was to bypass the second stage "wash" classifier and replace it with a single hydrocyclone with underflow washing using an elutriation device and regulated underflow density. These attachments are standard in other mineral processing applications but are novel in Bayer plants within the alumina industry. Installation experience is reported.

4:45 PM
THE LEACHING OF ALUMINIUM IN SPANISH CLAYS, COAL MINING WASTES AND COAL FLY ASHES BY SUPHURIC ACID: *Dr. Miguel A. Llavona*[1]; [1]University of Oviedo, Dept. of Science, U.S. Mining & Topographic Eng., Mieres Spain

The acid leaching of aluminium of several non traditional ores, bayerite, kaolinite, different clays, coal mining wastes and coal fly ashes, and the kinetic of their dissolution are described. The effects of time, temperature, acid concentration, sample calcination, particle size were examined. The leaching of aluminium is dependent on acid concentration and strongly on temperature. Generally, the time to reach a fixed percentage of dissolution decreases with increasing acid concentration in the range of 6% to 40% acid by weight. On clays and coal mining wastes a good relation between Al removal and ratio kaolinite/illite was also observed at all temperatures and acid concentration tested. Coal fly ashes are particles that were heated at very high temperatures in the power station and Al compounds were transformed into mullite and so Al recovery was minor. Several rate equations describing the kinetics of the leach reaction were discussed and Kinetic parameters and activation energy values of samples are presented.

ALUMINUM ALLOYS FOR PACKAGING III: Session I - Fundamental Research

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Subodh Das, Arco Aluminum Company, PO Box 32860, Louisville, KY 40232

Tuesday PM Room: Fiesta B
February 17, 1998 Location: Convention Center

Session Chair: Gyan Jha, ARCO Aluminum, Inc., Louisville, KY 40232

2:00 PM
COMPOSITION AND PREHEATING EFFECTS ON DISPERSOID AND INSOLUBLE CONSTITUENT PARTICLE EVOLUTION IN 3XXX ALLOYS: *Thomas N. Rouns*[1]; [1]Alcoa Technical Center, Alcoa Center, PA 15069

Several 3XXX alloys containing varying amounts of Fe, Mn, and Mg were cast and fabricated into full hard sheet at two commercial operations. Automated quantitative image analysis using a scanning electron microscope equipped with an energy dispersive X-ray detector allowed both the composition and amount of the insoluble constituent phases ($Al_{12}[Mn,Fe]_3Si$ and $Al_6[Mn,Fe]$) to be measured. From this analysis the relationship between the bulk chemical composition and the amount of Al_{12} $[Mn,Fe]_3Si$ after preheating can be determined. There is also a significant increase in the amount of Mn contained in both the $Al_{12}[Mn,Fe]_3Si$ and $Al_6[Mn,Fe]$ insoluble constituent phases after preheating. Additionally, there is an increase in the size of the insoluble constituent phases. This analysis coupled with electrical conductivity measurements were used to calculate the amount of dispersoid. The combination of these techniques, therefore, results in a complete description of the constitution of 3XXX.

2:30 PM

MODELLING OF DISPERSOID AND CONSTITUENT PARTICLE EVOLUTION IN 3XXX ALLOYS: *J. P. Suni*[1]; R. T. Shuey[1]; [1]Alcoa Technical Center, Alcoa Center, PA 15069

A kinetic model is developed for constituent and dispersoid particle evolution during preheating of 3XXX alloys. The constituent modelling combines the parallel processes of growth (or dissolution) and transformation from Al_6[Fe,Mn] to Al_{12}[Fe,Mn]$_3$Si phases that result in evolution equations for volume fraction of the two constituent phases, as well as the Mn/Fe ratio. The dispersoid modelling combines the parallel processes of nucleation, growth and coarsening, resulting in evolution equations for particle size, number density and volume fraction of dispersoid. Dispersoids can either be Al_6Mn or $Al_{12}Mn_3Si$ phase, depending on the silicon content, but numerical fitting work has only been done for silicon contents large enough that dispersoids are calculated as being exclusively the $Al_{12}Mn_3Si$ type. The evolution equations for constituents and dispersoids are coupled to each other through the matrix concentrations of manganese and silicon. Depleting these by the formation of new dispersoid reduces the growth and transformation of the constituents, and vice versa. Model parameters are related to diffusivity, interfacial energy, nucleation kinetics and equilibrium solvi for the Al_6[Fe,Mn] and Al_{12}[Fe,Mn]$_3$Si phases. The model is fit to conductivity date, as well as to measured values for the portion of constituent which is Al_{12}[Fe,Mn]$_3$Si and to Mn/Fe ratios in either phase. Data used in numerical fitting and testing this model includes internal Alcoa data as well as published, external results. This data contains variations in solidification rate, alloy composition, namely Fe, Mn, Si and Mg (i.e., either 3003 or 3004), as well as time and temperature of thermal treatments. The resulting model can be used to predict the effects of process excursions, or to the search for new combinations of alloy composition and thermal practice based on physical principles and prior data.

3:00 PM

PRECIPITATION BEHAVIORS AND PRECIPITATION HARDENING IN AA3004 ALUMINUM ALLOYS: Shixi Ding[1]; Jiun Qiu[1]; *James G. Morris*[1]; [1]University of Kentucky, Light Metals Research Labs, College of Engineering, Lexington, KY 40506-0046

The present study was undertaken to investigate the aging behaviors, the precipitation hardening potentials and the corresponding strengthening mechanisms of the AA3004 alloy system. The results of this study show that precipitation can occur at temperatures below 260 °C in properly processed AA3004 alloys. This precipitation can result in a hardening effect which is associated with the formation of intermediate precipitates of Al_2 CuMg and Mg_2Si phases. For an AA3004 alloy with a normal composition, an increase of 90-100 Mpa in the tensile yield strength and 70 Mpa in the ultimum tensile strength can be achieved at the O-temper state through a precipitation hardening mechanism. The precipitation behaviors of the alloys were studied by means of hardness tests, tensile tests, electrical resistivity measurements, and TEM observations. The phase identification by X-ray diffraction techniques (XRD) was carried out on selected samples and the detailed analyses of XRD spectra were made by employing a non-linear curve fit software (Peakfit).

3:30 PM Coffee Break in Exhibit Hall

4:00 PM

TEXTURE CAUSING 0°-180° DEGREE EARING IN CAN BODY STOCK: *Baolute Ren*[1]; [1]Reynolds Metals Company, Manufacturing Technology Laboratory, Corporate Research & Development, Muscle Shoals, AL 35661-1258

It is well known that Goss texture is a recrystallization texture and that it causes two ears on a cup at 0° and 180° to the sheet rolling direction (RD) when the cup is formed. However, for final gauge can body stock, Goss texture is unlikely to be highly developed because it is not a stable end orientation during cold rolling. For the purpose of earing control, it is important to understand what kinds of texture cause 0°-180° ears on D&I cans. In the present work, texture analysis was carried out on can body stock with various earing behaviors. The results indicated that a high density of texture orientations {Okl} <100> could be developed during cold rolling which caused high 0°-180° earing in can body stock. Typical texture examples corresponding to high and low 0°-180° earing are shown. A mechanism for these texture orientations to enhance two ears in the RD is discussed. Possible metallurgical and processing factors affecting the formation of the {Okl} <100> textures are proposed.

4:30 PM

STRUCTURAL AND PROPERTY CHARACTERIZATION OF DEFORMED ALUMINUM: *Niels Hansen*[1]; [1]RISO National Laboratory, Roskilde DK-4000 Denmark

Orientation image microscopy techniques (OIM) are developing rapidly for studies of thermomechanical processes both by Scanning Electron Microscopy (SEM) and Transmission Electron bilicroscopy (TEM). These techniques give statistical information about key parameters characterizing the evolution of deformation microstructures. These parameters are the misorientation angle and the spacing associated with dislocation boundaries. This microstructural information can be used in the prediction in the stress-strain behaviour of metals and to quantify the energy stored in the dislocation structure. That energy is a basis for their recrystallization behaviour. The application of this research will be demonstrated for aluminium and aluminiurn alloys deformed in rolling, plain strain compression and tension where the target is to develop physical based models for the relationship between process parameters and materials properties especially strength, anisotropy and recrystallization behaviour.

5:00 PM (ORAL ONLY)

EFFECT OF HOMOGENIZATION TREATMENT ON THE MICROSTRUCTURAL AND TEXTURE EVOLUTION OF ALUMINUM ALLOY AA3104 DURING HOT DEFORMATION: *Mr. Chetak Gandhi*[1]; Dr. Mary A. Wells[1]; [1]University of British Columbia, Metals and Materials Engineering, Vancouver, British Columbia V6T 1Z4 Canada

As customer demands become more stringent for canbody stock, it becomes essential to understand the complex interaction between the processing conditions and resulting product properties. This research focused on the homogenization process used for canbody stock and its effect on the microstructural and texture evolution during hot rolling. Samples were taken from an industrial DC Cast ingot and were homogenized in a computer controlled laboratory furnace under various thermal profiles. (i.e. homogenization temperatures 540, 570 and 600°C and homogenization soak times of two, six and twelve hours.) The samples were then hot deformed under plain strain conditions and the resulting microstructural (percent recovery, percent recrystallization and particle size distribution) and texture evolution was quantified.

ALUMINUM REDUCTION TECHNOLOGY: Computer Process Control Systems

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Alton Tabereaux, Reynolds Metals Company, 3327 East Second Street, Muscle Shoals, AL 35661-1258

Tuesday PM Room: Fiesta D
February 17, 1998 Location: Convention Center

Session Chair: Gary Tarcy, Aluminum Company of America, Alcoa Center, PA 15069

2:00 PM

REDUCTION CELL CONTROL TECHNOLOGY: *Mr. Joseph N. Rieg*[1]; Mr. Robert G. Nichol[1]; Mr. Gilbert G. Fryer[1]; [1]Intalco Aluminum Corp, Reduction, Ferndale, WA 98248-0937 USA

In 1997, Intalco Aluminum Corporation replaced the original 1971 potline control system with a modern centralized control architecture that opposes the trend of individual pot computers. System design,

implementation, and operation are the result of cooperation between Process Control, Operations, and Management Information Systems (MIS) departments. Incorporated into the system is a seamless interface between the centralized potline control system and MIS databases. One centralized potline computer controls individual pots and mirrors real-time data to the corporate network, making it available to all system users. Included within the reflected data are pot specific alarms that are sent to pot operators via an in-plant pager system.

2:25 PM

ELYSESEM AN EDUCATION AND INFORMATION TOOL: *Peter M. Entner*[1]; [1]Alusuisse Technology & Management Ltd., Technology Center, CH- 3965 Chippis Switzerland

The instruction of the personnel which operate electrolytic pots is conventionally done orally in seminars or by written documents. ElyseSem extends these methods by transmitting the information using the help routines of the standard operating system of a personal computer. The content covers the practical aspects and theoretical background of aluminum production like heat balance, magnetohydrodynamics, pot startup and operation, handling of disturbances and pot life. The user finds and displays information by clicking with the mouse pointer on the keyword in the content or index window. Separate windows are opened simultaneously with the body text to display figures, tables or animated sequences (for instance to demonstrate the operation of a point feeding device). After activating so called hot spots in the text or figures popup windows show additional information about the item. The user may also execute calculation with his own input from inside the system applying relations discussed in the seminar.

2:50 PM

RESPONSE OF A HALL-HEROULT CELL TO STEP CHANGES IN OPERATING CONDITIONS: MEASUREMENTS AND "DYNAMIC" SIMULATION: *Dr. H. Q. Tang*[1]; Dr. Nobuo Urata[1]; Mr. C. Mark Read[1]; Ms. Susan Stejer[1]; [1]Kaiser Aluminum and Chemical Corporation, Primary Aluminum Business Unit, Pleasanton, CA 94566-7769 USA

Operating cell responses to step changes in cell voltage and excess Aluminum Fluoride concentration were measured on 70 kA prebake aluminum reduction cells. Various operating parameters, including superheat, were monitored semi-continuously. In the first four hours subsequent to a 0.4 V cell voltage increase, superheat and bath temperature increased at an average rate of 2.5 C/hour and 4.5 C/hour, respectively. In a separate test, in response to the addition of a known weight of AlF3, the superheat and bath temperature decreased at an average rate of 2.7 C/hour and 4.5 C/hour, respectively. A proprietary "Dynamic Cell Simulator", controlled by commercially available Kaiser cell control software, was used to simulate the response of the cell to the above step changes in operating conditions. Significant events which occurred during the measurement period, e.g., anode change, were simulated. The relative error of the simulated response compared with the measured values of bath temperature, bath chemistry and superheat were 0.5%, 2% and 8%, respectively.

3:15 PM

LIQUIDUS TEMPERATURE DETERMINATION IN MOLTEN SALTS: *Ove Kobbeltvedt*[1]; Sverre Rolseth[1]; Paul Verstreken[1]; [1]SINTEF Materials Technology, Trondheim Norway

Three different ways of determining the liquidus temperature of Hall-Heroult baths are discussed: - bath analysis and application of liquidus equations - thermal analysis in laboratory furnaces -using liquidus probes or sensors. These methods are compared in both synthetic and industrial baths. Basically, all three methods make use of thermal analysis. Taking bath samples for chemical analysis only introduces more errors and seems to be the least reliable method.

3:40 PM Coffee Break in Exhibit Hall

3:50 PM

A LABORATORY PROBE FOR ACIDITY OF CRYOLITE-ALUMINA MELTS.: *Dr. Geir M. Haarberg*[1]; Mr. Frode Olufsen[1]; Mr. Henrik Gudbrandsen[1]; Dr. Sverre Rolseth[1]; Dr. Åsmund Sterten[1]; [1]Norwegian University of Science and Technology, Department of Electrochemistry, Trondheim, Trondheim N-7034 Norway

Potential measurements between aluminium and a sodium alloy electrode were performed in cryolite-alumina melts. This emf method was used to test acidity probes in laboratory experiments. The potential was found to respond to changes in the bath acidity during controlled additions of AlF3 and NaF. Several sodium alloys were tried. Effects of additions of CaF2, LiF and MgF2 as well as temperature on the measured potential were studied.

4:15 PM

PROBLEM OF CRYOLITE RATIO AND BATH TEMPERATURE STABILIZATION IN THE CELL: *Victor H. Mann*[1]; Vladimir V. Yurkov[1]; Peter V. Polyakov[1]; Victor Y. Buzunov[1]; [1]Krasnoyarsk Aluminium Smelter, Krasnoyarsk Russia

It is well known that there is a strong correlation between temperature and cryolite ratio in an aluminum reduction cell. In this paper we propose another hypothesis for this relationship, that is significantly affected by the potroom process control system. As well, we will outline the main directions of investigations carried out by Krasnoyarsk Aluminum Smelter's experts in the field of creation of a two interrelated control systems for cryolite ratio and cell temperature control in a reduction cell, based on the above mentioned hypothesis.

4:40 PM

TITANIUM DIBORIDE PLASMA COATING OF CARBON CATHODE MATERIALS, PART I: COATING PROCESS AND MICROSTRUCTURE: *Dr. Katharina Seitz*[1]; Frank Hiltmann[2]; [1]Hoechst AG, CR&T, D-65926 Frankfurt Germany; [2]SGL Carbon AG, Griesheim Plant, D-65933 Frankfurt Germany

The interest in using titanium diboride (TiB_2) as cathode coating material is high because of its possible potential for significant energy and cost savings in the production of primary aluminum. In the present work vacuum plasma spraying has been used to fabricate TiB_2 coatings on different carbon materials. The influence of different process parameters, e.g., spray distance, spray powder quality and chamber pressure on microstructure of the TiB_2 deposits were studied. The density and pore size distributions measured by image analysis changed sensitively with chamber pressure. The deposits have a high hardness, good adherence to the carbon material, and a good wettability by aluminum. The electrical resistivity which significant depends on the oxygen impurity is lower than that of the carbon material. Further characterization of the coated materials is reported in Part II of this paper.

5:05 PM

TITANIUM DIBORIDE PLASMA COATING OF CARBON CATHODE MATERIALS, PART II: CHARACTERIZATION: *Frank Hiltmann*[1]; Katharina Seitz[2]; [1]SGL Carbon AG, Grieheim Plant, D-65933 Frankfurt Germany; [2]Hoechst AG, CR&T, D-65926 Frankfurt Germany

Carbon cathode materials which had been coated with titanium diboride according to the plasma spraying procedure described in Part I were submitted to several laboratory tests in order to examine the suitability of application in aluminium electrolysis cells: Rapoport tests exhibited a lower expansion due to reduced sodium attack and a good wetting of the coating by molten aluminium. Abrasion measurements showed a considerably improved mechanical stability vs. Uncoated samples. Fast heat-up to 1200 degrees C yielded no negative effect on the internal structure or the adherence of the coating. Chemical resistance to attack by molten aluminium was high. These results indicate that TiB_2 plasma-coated cathodes possibly could give significant benefits to the aluminium smelting process in terms of energy cost saving and extended cell life.

5:30 PM

SIMULATION OF DYNAMIC CELL RESPONSE UNDER DIFFERENT TYPES OF CELL CONTROL LOGIC: *Marc Dupuis*[1]; Imad Tabsh[1]; [1]GeniSim, Jonquiere, Quebec G7S 2M9 Canada

A comprehensive program (ARC/Dynamic) was developed to simulate the dynamic behavior of aluminum reduction cells during operation. It was demonstrated in previous papers that the program could be used to study the impact of design changes and to assist in the training

of new pot operators. This paper focuses on the third intended application of the program, i.e., using the model to test the dynamic pot response to different types of cell control logic. The first part of the paper will discuss the ability of a cell to self regulate its concentration of dissolved alumina. It will show that this is dependent on the relationship between the current efficiency and the concentration of dissolved alumina. The paper will then look at the case where a cell cannot self regulate itself and show that the continuous tracking scheme is the best control logic to use.

ATOMIC-LEVEL SIMULATION OF MATERIALS: NEW METHODS & NOVEL APPLICATIONS: Bulk Simulations

Sponsored by: ASM International: Materials Science Critical Technology Sector, Computer Simulation Committee

Program Organizers: Jim Adams, Arizona State University, Dept. of Chemical Bio and Materials Dept., Tempe, AZ 85287-6006; Vaclav Vitek, University of Pennsylvania, Dept. of Materials Science & Eng., Philadelphia, PA 19104

Tuesday PM Room: 203
February 17, 1998 Location: Convention Center

Session Chair: David Srolovitz, Ann Arbor, MI 48109-2136

2:00 PM

THE INFLUENCE OF SOLID SOLUTIONS ON FLOW BEHAVIOR IN γ-TiAl: *Dr. Christopher F. Woodward*[1]; Dr. Scott A. Kajihara[1]; Dr. Satish I. Rao[1]; Dr. Dennis M. Dimiduk[2]; [1]UES Inc., Materials Research Division, Dayton, OH 45432 USA; [2]Materials Directorate, Wright Laboratory, MLLM, Wright Patterson AFB, OH 45433-7817 USA

Transition metal intermetallic alloys, such as TiAl and NiAl, are an important class of structural materials due to their low density, high melting temperature and excellent strength retention at high temperature. Modifications of alloy chemistry are often used to tailor the intrinsic flow behavior of these structural materials. Models of solution strengthening, high temperature yield stress and creep must relate the effects of chemistry to the mechanisms which are responsible for a specific material property. In ordered alloys, additional information regarding the crystallographic site occupancy of ternary elements is required. Relaxed structures and energies for intrinsic and substitutional point defects are calculated using a plane-wave-pseudopotential method. The calculated defect energies are used to predict the density and site preferences of solid solutions (Si, Nb, Mo, Ta and W) in γ-TiAl. Size and modulus misfit parameters are calculated and the interaction of these defects with a dissociated ordinary screw dislocation evaluated within anisotropic elasticity theory. The derived interaction strength is then related to solid solution strengthening for these defect centers. Predicted solid solution effects are in good agreement with experimental observations for the binary alloy. Several defects in the ternary alloys are also predicted to produce solid solution strengthening at intermediate temperatures.

2:35 PM

DESIGN OF LITHIUM-METAL-OXIDES CATHODE MATERIALS FOR RECHARGEABLE LITHIUM BATTERIES BY MEANS OF FIRST-PRINCIPLES MODELING.: *Prof Gerbrand Ceder*[1]; Dr Kadri M Aydinol[1]; Mr Anton Van der Ven[1]; [1]MIT, Dep. of Materials Science and Engineering, Cambridge, MA 02139 US

First-principles methods have become sufficiently accurate that they can predict some properties of materials with high degree of confidence. Because they require no experimental input, and offer full control over the applied boundary conditions, these methods can be used to conduct "virtual experiments". We demonstrate how this approach can be effectively used to study the factors that influence the Li-intercalation voltage in lithium-metal-oxides. Strong interest exists in these materials since they are used as cathodes in lightweight rechargeable Li-batteries. The pseudo potential method is used to calculate the Li-intercalation voltages for a series of compounds with various structures and metal chemistries. From these results design criteria for higher voltage compounds can be established. Such compounds have now been evaluated with our quantum mechanical methods, synthesized and tested.

2:55 PM

DYNAMIC FRACTURE IN NANOPHASE CERAMICS: *Dr. Rajiv K. Kalia*[1]; Dr. Aiichiro Nakano[1]; Dr. Shuji Ogata[2]; Dr. Andrey Omeltchenko[1]; Dr. Kenji Tsuruta[1]; Dr. Priya Vashishta[1]; [1]Louisiana State University, Concurrent Computing Laboratory for Materials Simulations, Dept. of Physics and Astronomy, Baton Rouge, LA 70803-4001 USA; [2]Yamaguchi University, Department of Applied Sciences, Ube, Yamaguchi 755 Japan

Multimillion atom molecular dynamics simulations are performed on massively parallel machines to investigate fracture in nanophase silica and silicon nitride [1,2]. In the nanophase ceramics, structural correlations in interfacial regions are found to have a strong influence on mechanical properties. Intercluster regions tend to deflect cracks and give rise to local crack branching. Implications of crack deflection and meandering on fracture will be discussed. Simulation results for the morphology of fracture surfaces will also be presented. Work supported by DOE, NSF, AFOSR, USC-LSU Multidisciplinary University Research Initiative, Army Research Office, and Louisiana Education Quality Support Fund. 1. R. K. Kalia, A. Nakano, K. Tsuruta, and P. Vashishta, Phys. Rev. Lett. 78, 689 (1997). 2. R. K. Kalia, A. Nakano, A. Omeltchenko, K. Tsuruta, and P. Vashishta, Phys. Rev. Lett. 78, 2144 (1997).

3:30 PM

AB INITIO STUDY OF H IN AMORPHOUS SILICON: *Dr. Blair Tuttle*[1]; Dr. James Brewster Adams[2]; [1]UIUC, Urbana, IL 61801; [2]Arizona State University, Chem, Bio and Materials , Tempe, Arizona

The presence of hydrogen is essential for producing device quality films of hydrogenated amorphous silicon (a-Si:H). Despite the importance of hydrogen, microscopic models for hydrogen bonding and diffusion are not well developed. We use ab initio density functional methods to calculate the structure and energetics of hydrogen in realistic models of a-Si:H. Our results are successfully applied to several experiments including tracer diffusion and thermal evolution. In addition, we clarify the role of hydrogen during the creation of electronic defects. Our results suggest the isolated dangling bond is not a viable candidate for mid-gap defects. We describe other defect models which are consistent with our calculations and the experimental constraints.

3:50 PM Coffee Break in Exhibit Hall

3:55 PM

AB-INITIO STUDY OF SYMMETRICAL TILT GRAIN BOUNDARIES IN BODY-CENTERED CUBIC TRANSITION METALS.: *Dr. Christian Elsaesser*[1]; [1]Max-Planck-Institut fuer Metallforschung, Stuttgart D-70174 Germany

Atomistic simulations of grain-boundary structures in body-centered cubic transition metals have revealed that angle-dependent contributions to interatomic interactions are essential (see, e.g., [1,2]). However, results of presently available empirical potentials are not yet always sufficiently accurate or unique for quantitative theoretical predictions about defect structures like grain-boundaries, which are consistent with experimental observations, e.g., by HRTEM. Ab-initio local-density-functional calculations, on the other hand, offer the possibility to determine the structures of some special grain boundaries with high accuracy, which can be used as a data base for the development of empirical potentials. Results of such ab-initio calculations, using a mixed-basis pseudopotential method and grain-boundary supercells, are presented for the symmetrical Σ 5 (310) [001] tilt grain boundaries in Niobium and Molybdenum. [1] G. H. Campbell et al., Phys. Rev. Lett.

70, 449 (1993).[2] A. G. Marinopoulos et al., Phil. Mag. A 72, 1311 (1995).

4:30 PM

ATOMISTIC SIMULATION OF TRIPLE JUNCTION MIGRATION: *Mr. Moneesh Upmanyu*[1]; Prof. David J. Srolovitz[1]; Prof. Lasar S. Shvindlerman[2]; [1]University of Michigan, Materials Science & Eng., Ann Arbor, MI 48109-2136 USA; [2]Academy of Sciences, Institute of Solid State Physics, Cherngolovka, Moscow District 142432 RUSSIA

The effect of triple junctions on grain boundary migration is studied using a molecular dynamics simulation technique. Initially, the ratios of the grain boundary energies are determined by equilibrating the three grain boundary/triple junction at high temperature (> 0.5 Tm) and measuring the equilibrium triple junction angle. The steady-state triple junction mobility is extracted by monitoring the trajectory, for several special and non-special boundaries. The triple junction angle remains fixed during its motion. The effect of the triple junction on boundary migration is monitored by comparing the rate at which the triple junction moves as compared with the mobility of the constituent boundaries measured in separate simulations. Atomic motions in the vicinity of the triple junctions are observed to arrive at mechanisms which contribute to the triple junction mobility. The implication of incorporation of finite triple junction mobility into grain growth models is discussed.

4:50 PM

INFLUENCE OF GRAIN BOUNDARY MISORIENTATION ON THE GALLIUM PENETRATION OF ALUMINUM: *Mr. Richard C. Hugo*[1]; Dr. Richard G Hoagland[1]; [1]Washington State Univ, Mech and Matls Engr, Pullman, WA 99164-2920 USA

We present the application of atomistic simulation to the liquid metal embrittlement of aluminum. Liquid gallium penetration of aluminum grain boundaries is observable in the Tranmission Electron Microscope (TEM). In-situ TEM studies show that the speeds of the penetration fronts can differ by two orders of magnitude between different grain boundaries. This suggests that, among other things, penetration rates depend on the structure of general grain boundaries. We employ this phenomenon to elucidate the nature of general grain boundaries in aluminum. From misorientation data obtained during TEM experiments, atomistic grain boundary models are constructed and relaxed using Embedded Atom Method potentials. The grain boundary energies and excess volumes derived from the models were compared with the gallium penetration speeds for a set of observed grain boundaries. We find that boundaries with higher calculated energy and excess volume are penetrated more rapidly; however, the variation in these calculated properties is smaller than the variation in penetration speeds. Other factors that may explain the large variation in penetration speed, such as channels of high excess volume, are discussed. This research is supported by the US Dept. of Energy under grant DE-FG06-87ER45287

5:10 PM

APPLICATION OF THE KINETIC MONTE-CARLO METHOD FOR ATOMISTIC SIMULATIONS OF INTERFACIAL DIFFUSION: *Dr. Yuri Mishin*[1]; [1]Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061-0237

A new application of the kinetic Monte Carlo method for the simulation of atomic transport along interfaces is illustrated. The simulation scheme includes the following steps: (1) molecular statics calculation of the underlying interfacial structure at 0K; (2) calculation of point defect binding energies to different interfacial site, and thus the respective point defect concentrations; (3) static calculation of point defect migration energies between different pairs of interfacial sites; (4) calculation of the atomic jump frequencies between different interfacial sites (the attempt frequency is evaluated in the local harmonic approximation); (5) calculation of the interfacial diffusion coefficient by means of kinetic Monte Carlo simulations. Two variants of the Monte Carlo method are considered and compared with one another: (1) the direct kinetic Monte Carlo method, known from previous simulations of lattice diffusion, and (2) combined matrix-Monte Carlo method developed specifically for interfacial diffusion. In the latter case the diffusion coefficient is expressed in terms of partial correlation factors associated with individual atomic jumps at the interface. The partial correlation factors are determined from a matrix equation in terms of next jump probabilities. In turn, the next jump probabilities are calculated by Monte Carlo simulations of individual encounters between a point defect and a diffusing atom. Recent applications of this simulation scheme are discussed.

CARBON TECHNOLOGY: Materials and Properties

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Ron Barclay, Alumax, PO Box 1000, Goose Creek, SC 29445

Tuesday PM Room: Fiesta E
February 17, 1998 Location: Convention Center

Session Chair: Sheralyn Hume, Boyne Smelters, Gladstone 4680, Queensland Australia

2:00 PM

PETROLEUM AND COAL TAR PITCH CORROSION EVALUATION: *Carola Acuna*[1]; Roger Marzin[1]; Maria Specht[1]; [1]INTEVEP, S.A., Caracas 1070A Venezuela

Comparative performances of Venezuelan petroleum pitch and coal tar pitch, in terms of corrosion, were evaluated at laboratory scale under different test conditions. Corrosion for both liquid and gaseous phases was determined using carbon steel coupons. The experimental methodology is described in this paper. Pitch chemical structure and gas phase composition were used to explain the observed corrosion rates for both types of pitch.

2:25 PM

VIBROCOMPACTING MACHINES FOR THE MOULDING OF GREEN ANODES-PROCESS DEVELOPMENT FROM THE EQUIPMENT SUPPLIER'S POINT OF VIEW: *Mr. Manfred Beilstein*[1]; Mr. Manfred Spangehl[1]; [1]KHD Humboldt Wedag AG, D-51057 Cologne Germany

In green anode production, since the late nineteenseventies vibrocompacting is the only accepted green anode moulding technology for any new carbon plants to be built, and -due to economics- hydraulic block pressing, which was previously in use, has been completely abandoned. Also in a number of existing aluminum smelters, block presses have been replaced by vibrocompacting machines for the smelter operators to be able to mould larger anodes needed for modernized smelter operation. The basic principles of design of anode vibrocompacting machines being in industrial use are presented along with the development over the years of core equipment components. The main objective of this work was to improve equipment reliability and maintainability, reduce emissions of vibrations, noise and fumes to the environment, and last not least improve anode quality. Such improvements are being described and supported with plant data.

2:50 PM

PITCH FUME TREATMENT FOR A GREEN CARBON PLANT: *Mr. Malcolm Gale*[1]; [1]Hosokawa Micron Limited, Runcorn, Cheshire WA7 2TY England

-Introduction- The following summarizes a dry scrubbing system to extract and scrub pitch fume generated within the green carbon area of an aluminum plant. Equipment served includes:- * Pitch weigh pots * Anode paste mixers * Paste conveyor * Anode forming tanks * Pitch storage vessel -Emission Measurement- -Concentration levels of total tar, benzine, total fluorides and PAH's will be tabulated both before and after installation of the scrubbing system for comparison. -Description of System- A comprehensive description of the dry scrubbing system will be given supported by flow diagram, G.A. drawing and

calculations. Particular reference will be made to the design of the combined reactor/filter inlet. Gas and adsorpent enter the filter vertically upwards into the reactor section where thorough mixing and velocity reduction occur. Internal louvres are positioned prior to the bag chamber to ensure a very low velocity and laminar flow pattern, to eliminate abrasion and re-entrainment.

3:15 PM

DETERMINATION OF THE LENGTH OF CRYSTALITES IN THE PETROLEUM COKE (PARAMETER Lc) THROUGH THE TECHNIQUE OF X-RAY DIFFRACTION: *Leonir Gomez*[1]; Mokka N. Rao[1]; [1]Universidad Nacional Experimental De Guayana, Puerto Ordaz-8015A, Estado Bolivar Venezuela- S.A.

The petroleum coke proceeding from the refinery Punta Cordon-Maraven, obtained by delayed coking process is subjected to the process of calcination at different temperature levels in a controlled laboratory oven. This work consists of study of structural changes of the calcined petroleum coke utilized in the fabrication of anodes in the electrolite aluminium reduction cells. Technique of X-Ray diffraction is utilized for the determination of the length of the crystallites (Lc) with the object of establishing models which permit obtaining the basic parameters of optimum quality of calcined petroleum coke for the fabrication of anodes. The values of Le are correlated with the temperature of calcination within the range of 900° - 1500°C in the quadratic form in agreement with the model:$Lc = 84.726 - (0.131)\ T\ (6.733 \times 10^{-5})T^2$

3:40 PM

THE INFLUENCE OF PITCH IMPURITY CONTENT ON REACTIVITY OF BINDER COKE IN ANODES: *Mr. Morten Sorlie*[1]; Mr. Trygve Eidet[1]; [1]Elkem, N-4602 Kristiansand S. Norway

Controlled carbonization of anode binder pitches under 15 bar inert gas pressure in a specially built reactor, and subsequent heat treatment, has been developed as a method to produce uniform "binder cokes" for laboratory study. A number of industrial anode pitches together with some alternative binders have been studied in order to determine the reactive properties towards CO_2 at 960°C and air at 475°C of the pitch binder coke in anodes. Simple empirical expressions of gas reactivity versus binder coke impurity content can be used to describe the oxidation properties of the coked and calcined pitches. The sodium impurity content in the binder coke dominates the air reactivity results. Measurements of CO_2 reactivity, air reactivity and carbon dust generation on baked anodes give a qualitative verification of the binder coke tests.

4:05 PM

DEVELOPMENT OF ANODE BINDER PITCH LABORATORY CHARACTERIZATION METHODS: *E. R. McHenry*[1]; J. T. Baron[1]; K. C. Krupinski[1]; [1]Koppers Industries, Inc., Pittsburgh, PA 15238 USA

Typical anode binder pitch characteristics, such as softening point and quinoline insoluble, are used to monitor quality assurance. More meaningful laboratory characterization methods that can be used to predict the performance of binder pitches are being developed. A pitch carbon yield measurement designed to follow pilot-scale anode baking rates is proving to be more informative than routine laboratory methods. The pitch carbon from this measurement is used to determine relative reactivities of carbons from a variety of pitches. A four-inch diameter vibrated anode is permitting comparison studies of binder pitches and anode forming variables; for example, temperature, vacuum vs. atmospheric pressure and binder level. The results from these nonroutine characterization procedures are assisting in the development of feedstock sourcing and processing.

CAST SHOP TECHNOLOGY: Session IIB - Solidification Structures

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Diran Apelian, Worcester Polytechnic Inst., 100 Institute Rd., Worcester, MA 01609-2280

Tuesday PM — Room: River Room B
February 17, 1998 — Location: Convention Center

Session Chair: Martin A. Kearns, London & Scandinavian Metallurgical Co., Fullerton Road, Rotherham South, S60 1DL England

TUESDAY PM

2:00 PM

THE EFFECTS OF Ti AND Sr AFFECTING THE MICROSTRUCTURES OF Al-11.3WT% Si ALLOYS BY THE CONTINUOUS CASTING PROCESS.: *M. H. Kim*[1]; T. S. Song[1]; H. H. Jo[1]; C.S. Kang[1]; C. R. Loper, Jr.[2]; [1]Chungbuk National University, Dept. of Materials Eng., Cheogju Korea; [2]University of Wisconsin, Madison, WI

The horizontal continuous casting process with the heated mold for obtaining unidirectional solidification at a high cooling rate (300-2000 Degrees C/s), comparing with the conventional casting process, has been applied to study the effect of grain refiner or modifier such as Ti or Sr, respectively, in the Al-11.3wt%Si alloy. The results represented that the addition of 0.02%Si or 0.2-0.4%Ti promoted the directionality of primary alphadendrite, whereas the addition of 0.6%Ti suppressed the directionality. Also, the addition of 0.02%Sr suppressed the occurrence of feathery grains, while the addition of 0.2%Ti promoted the occurrence of feathery grain. The addition of Ti over 0.4% suppressed the occurrence of feathery grain, resulting from the increasing grain-refining effect. The addition of Ti also increased the coarsening effect of eutectic silicon phase in the Al-11.3wt%Si alloy.

2:25 PM

EFFECT OF CA ON THE MICROSTRUCTURE OF A CAST ALUMINUM ALLOY: *Prof. Rafael Colas*[1]; Dr. Eulogio Velas; co[2]; Dr. Salvador Valtierra[2]; Dr. Juan Francisco Mojica[2]; [1]FIME, UANL, San Nicolas, N.L. 66451 MEXICO; [2]Corporativo Nemak, S.A. de C.V., Garza Garcia, N.L. 66221 MEXICO

A series of experimental trials in a casta aluminum alloy (A319) were conducted. The experimental design was planned to investigate the effect that Ca exerts on porosity and modification of the alloy under study. Grain refiners and modifier elements, as well as Ca, were added in a controlled manner into the liquid Al-Si alloy (which was degassed with nitrogen before pouring into specially designed moulds). Study of the solidification reactions was carried out by thermal analysis, evaluation of the resulting microstructure was done by image analysis. It was found that addition of Ca in quantities higher than 90 ppm exerts a positive influence in the decrease in size and number of pores encountered in the cast piece. This element also contributes to modification when the material was previously treated with Sr.

2:50 PM

THE EFFECT OF STRONTIUM CONCENTRATION ON POROSITY IN 319 ALUMINUM ALLOY CASTINGS: *Mr. Jacob W. Zindel*[1]; [1]Ford Motor Company, Materials Science Department, Dearborn, MI 48121 USA

Strontium (Sr) is added to hypoeutectic Al-Si alloys to alter or modify the morphology of the Si phase in the Al-Si eutectic constituent. Several investigators have also shown that the addition of Sr alters the amount and nature of porosity that forms during the solidification of castings. Castings made without Sr tend to have large local pores or cavities while the addition of 200 ppm Sr tends to disperse porosity throughout the casting. The goal of this work was to determine if the concentration of Sr has an effect on the amount of porosity in a directionally solidified casting. Eight castings with Sr concentrations

ranging from <20 ppm to 180 ppm were cast with controlled levels of hydrogen and sectioned for porosity measurements. The castings are in the shape of a wedge and were designed to provide a wide range of solidification times. Porosity was calculated from densities measured using the Archimedes technique. In all cases, the porosity increased from the thin to the thick sections of the wedge while the Sr concentration did not have a significant effect on the amount of porosity measured in the castings.

3:15 PM Coffee Break in Exhibit Hall

3:30 PM

ESTIMATION OF BOUNDARY CONDITIONS FOR THE MODELING OF METALS CASTING: *Stavros A. Argyropoulos*[1]; Mike Trovant[1]; [1]University of Toronto, Department of Metallurgy and Materials Science, Toronto, Ontario M5S 3E4 Canada

The mathematical modeling of casting processes has undergone evolutionary development within the last decade. A current stage in this maturation process involves the coupling of boundary conditions with all associated governing equations. A novel technique is proposed, which incorporates the relationship between the heat transfer coefficient and the formation of and 'air' gap at the mold boundary of a casting to improve the boundary conditions input to the energy equation. The correlation is experimentally derived, and attempts to express the heat transfer coefficient as a function of air gap size, mold and metal interface temperatures, the thermophysical properties of the gas in the gap, the geometry of the interface, and an interfacial roughness parameter. The general form of the equation is given for molds of differing heat diffusivities, and error analysis is used to express the reliability of the correlation with respect to the above mentioned variables. A comparison illustrating the improved performance to a typical casting simulation will be also presented.

3:55 PM

DEVELOPMENT OF A TECHNIQUE FOR THE DETERMINATION OF THE THERMAL CONTACT RESISTANCE BETWEEN A METAL SOLIDIFYING UNDER PRESSURE AND A METALLIC MOLD: *Mark C. Carroll*[1]; Makhlouf M. Makhlouf[1]

The heat transfer coefficient at the metal-mold interface is a critical parameter in solidification modeling. Unfortunately, the development and accumulation of a complete data base of heat transfer coefficients is extremely difficult since the magnitude of the heat transfer coefficient can take on an infinite number of values depending on a variety of factors. Specifically, it is well established that the magnitude of the interfacial heat transfer coefficient between a metal mold and a solidifying metal alloy depends on the casting process and the casting conditions, the metal and mold materials, the type and thickness of the mold coating, the geometry of the mold, and the magnitude of the applied pressure. To further complicate matters, an air gap usually develops between the solidifying metal and the mold wall. The presence and size of the air gap affects the magnitude of the interfacial heat transfer coefficient. Moreover, the above-mentioned factors interact strongly with one another so that isolating the effect of one parameter on the magnitude of the heat transfer coefficient is, in most cases, impossible. Nevertheless, because of the considerable interest in computer modeling of the casting process, and the importance of the heat transfer coefficient to the accuracy of computer simulations, extensive research efforts have been devoted to the measurement and determination of the coefficient. However, the majority of these measurements were performed under limiting conditions, and as such are of limited usefulness. Thus, there exists a need for a method that allows the accurate, repeatable, and reproducible determination of the interfacial heat transfer coefficient between a metal mold and a solidifying metal. This method should be general enough to permit characterizing the effect of all the significant variables an the magnitude of the interfacial heat transfer coefficient. This research is conducted in two phases: During phase one, emphasis is on developing the test apparatus and demonstrating its effectiveness in determining the interfacial heat transfer coefficient as a function of mold temperature, metal temperature, applied pressure, and type and thickness of mold coating. Also during phase one effort is made to demonstrate the repeatability and reproducibility of the results obtained using the apparatus and procedure developed. During phase two, a method for determining the heat transfer coefficient from data produced using the apparatus and procedure developed in phase one is developed. Calculation techniques investigated in phase two include reverse calculation of the heat transfer coefficient from the empirical cooling curves using an iterative module in Pro-Cast, and the inverse method for calculating heat transfer coefficients from empirical cooling curves.

4:20 PM

EMPIRICAL MODELS FOR CALCULATING Mn RETAINED IN SOLID SOLUTION AND CONSTITUENT PHASE PARTICLE SIZE IN AS-CAST ALUMINUM ALLOY3004: *P. N. Anyalebechi*[1]; [1]James Madison University, College of Integrated Science and Technology, Harrisonburg VA 22807

Empirical models for predicting the effects of solidification rate and alloy composition (specifically Mn, Cr, Fe, and Si contents) on Mn retained in solid solution and constituent phase particle size in cast aluminum alloy 3004 have been developed. Using directionally solidified laboratory size ingots, the study involved a range of solidification rate similar to that observed in a wide range of cast ingots and slabs. Within the range of composition and solidification rate investigated, the models correctly predict the variation of constituent phase particle size and Mn retained in solid solution through the thickness of commercial size ingots and continuously cast slabs.

DEFECTS IN CRYSTALS: A SYMPOSIUM HONORING THE CONTRIBUTIONS OF JOHN P. HIRTH: Damage and Failure

Sponsored by: Jt. Electronic, Magnetic & Photonic Materials Division/Structural Materials Division, Chemistry & Physics of Materials Committee, Physical Metallurgy Committee, Shaping and Forming Committee

Program Organizers: Craig S. Hartley, National Science Foundation, 4201 Wilson Blvd Room 545, Arlington, VA; Brent L. Adams, Carnegie Mellon University, Dept. of Materials Sci & Eng., Pittsburgh, PA 15213-3890; Richard G. Hoagland, Washington State University, Dept. of Materials Sci & Eng., Pullman, WA 99164-2920; Rob Wagoner, The Ohio State University, Dept. of Materials Sci & Eng., Columbus, OH 43210

Tuesday PM Room: 102
February 17, 1998 Location: Convention Center

Session Chair: Brent Adams, Carnegie Mellon University, Dept. of Materials Sci. and Eng., Pittsburgh, PA 15213-3890

2:00 PM

EDGE DISLOCATIONS EMITTED FROM A MODE I CRACK: *James C. M. Li*[1]; [1]University of Rochester, Department of Mechanical Eng., Rochester, NY 14627-0133

Edge dislocations are emitted from the crack tip as well as from nearby sources. The size and shape of the plastic zone depend on the order of dislocation emisssion, namely whether the dislocations are emitted one at a time or many simultaneously. They depend also on the availability of slip systems, the lattice friction stress for dislocation motion, the applied stress intensity factor (SIF) and the critical SIF for dislocation emission from the crack tip. The applied SIF determines the size of the plastic zone and the critical SIF determines the size of the dislocation-free zone (DFZ). The shielding effect is less when the dislocations are more uniformly distributed around the crack tip. When the nearby sources are available, the DFZ is reduced or eliminated. The shape of the plastic zone is comparable to that of von Mises or Tresca

criterion. Work supported by NSF through DMR 9623808 monitored by Dr. Bruce MacDonald.

2:30 PM

THE ROUGH FRONTS OF PITTING CORROSION: *Torstein F. Jossang*[1]; [1]University of Oslo, Institute of Physics, Blindern, Oslo 0316 Norway

A two dimensional computer model for the pitting corrosion process that includes passivation and depassivation was developed and extended to include the increased aggressiveness of the local environment due to hydrolysis of corrosion products as they move within the pits by diffusion and Coulomb interactions. The pitting corrosion process has also been studied experimentally. A characterization of their rough front morphologies by fractal geometry was performed. The computer model produced results that were qualitatively as well as quantitatively very similar to those obtained from the analysis of the experimental corrosion fronts.

2:50 PM

THE RELATIONSHIP BETWEEN SURFACE ROUGHENING AND LOCALIZED NECKING IN AL 6022 T4 SHEETS: *S. P. Lee*[1]; L. Hector[1]; G. W. Jarvis[1]; H. Hampel[1]; Brent L. Adams[1]; Henry R. Piehler[1]; Anthony L. Rollett[1]; [1]Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

The development of localized necks in sheet metal deformation has been the subject of numerous theoretical and experimental investigations. We report here on the less well understood precursor to localized necking, i.e. surface roughening. The experimental results for surface roughening and localized necking will be interpreted in terms of the underlying microstructure and to predictions of heterogeneity of deformation. The development of surface roughness in AL 6022 T4 sheets has been studied after deformation in uniaxial tension and near plane strain tension. Characterization was by optical microscopy, x-ray texture analysis, optical interference microscopy, capacitance gage topography, and scanning electron microscopy. The texture of the T4 sheet combined rolling and annealing components, with the strongest component being <100>(001) at 13 times random. Little surface roughening could be seen until localized necking developed at a local strain of about 0.3, at which point roughening of varying wavelengths of tip to several hundred micrometers was observed.

3:10 PM

ON THE MIXED-MODE IMPACT TOUGHNESS OF MATERIALS: *Dr. M. Manhoran*[1]; [1]Nanyang Technological University, Division of Materials Engineering, Singapore 639798 Republic of Singapore

A considerable amount of work on mixed mode I/III fracture toughness of materials using proportional loading methods is now available largely due to contributions by John Hirth and his co-workers. The superposition of mode ITT loading results in drastic reduction in fracture toughness in some materials whereas in other materials it has little effect or even results in an increase in the fracture toughness. Fracture mechanism maps delineating regions of susceptibility to tensile and shear loads have been proposed to explain such differences. Most of this work has, however, been conducted at slow strain rates. Recently, we have attempted to extend the proportional loading concept to explore the mixed-mode impact toughness of a variety of steels and aluminum alloys. This paper will summarize these results and show that the general concepts of the fracture mechanism map are in principle extendible to the case of dynamic impact fracture.

3:30 PM

IN-SITU TEM STUDY AND DISLOCATION MODELING OF NANOCRACK NUCLEATION AND GROWTH: *Scott X. Mao*[1]; Xiangping Li[1]; [1]University of Calgary, Department of Mechanical Engineering, Calgary, Alberta T2N 1N4 Canada

During the last two decades extensive experimental and theoretical work has been done to study the brittle-to-ductile transition, lead to the fundamental concepts on crack-tip dislocation emission, dislocation free zones(DFZ) and local equilibrium models of ductile or brittle fracture. Not only is the understanding of the global stress field needed to evaluate the probable crack nucleation site, but more local scales of stress distribution and environmental interactions are required within this region for an appropriate failure criterion. The transmission electron microscope (TEM) in situ tensile observations have been performed on many metals, alloys and intermetallic compounds. An excellent review on the results of the TEM observations and the development of the dislocation theory of fracture have been made by Ohr. Both experiments and previous theoretical analyzes revealed a mechanism of quasi-cleavage—it proceeds by forming a nanocrack in the DFZ of large elastic distortion and then by linking the nanocrack with the main crack, and this quasi-cleavage process is driven by dislocation pileups against the DFZ. In this paper, the initiation and propagation of nanometre scale cracks have been investigated in detail in mechanics modeling and in situ TEM observations for the intermetallic compound Fe_3Al under Mode I loading. A discrete dislocation model is proposed to assess the quasi-static equilibrium, crack tip emission, shielding of near-tip dislocations and the nanoscopic growth of the main crack. The equilibrium locations of individual dislocations and the equilibrium number of dislocations are determined by a minimum energy requirement. Three stage responses are revealed: When cracks propagate directly from the thin edge of a double-jet hole, no dislocation is detected and no dislocation emission is found. In thicker regions of the foils, thinning takes place because a great number of dislocations are emitted from the crack tip, and then an electron semitransparent region is formed in front of the crack tip region but not at the crack tip. Accurate dislocations/FEM calculation enables insight into how dislocation shielding led to microcrack nucleation and indicates the emergence of a tensile stress peak ahead of the crack tip, as the dislocations pile up against the DFZ. Distances between discontinuous nanocracks and the main crack tip were 4-70 nm which depends on the applied loading. The distances increase with the tensile loading increases. A gigantic super-dislocation and a mini-dislocation array are used to simulate the effect of more stringent barrier such as grain boundary, second phase particles, and brittle ductile interfaces. It was found that at the higher K_I the number of dislocations needed for shielding are in agreement with analytical and numerical models, the stress peak decreases slightly with the applied loading increases. This reflects the effect of stress relaxation in the entire DFZ as a result of the accumulation of dislocations in the plastic zone. The experiment and theoretical results is qualitatively consistent.

3:50 PM

DEFECTS AND FAILURE IN ULTRA-FINE COPPER MAGNET WIRE: *Lawrence E. Murr*[1]; R. D. Flores[1]; [1]University of Texas at El Paso, Department of Metallurgical and Materials Engineering, El Paso, TX 79968-0520 USA

Copper magnet wire drawn to 52 or 56 gauge (17 to 10 micrometer diameters, respectively) from standard precursor rod (0.79 cm diameter) represents a continuous wire drawing true strain of >13. The microstructure evolution is characterized by equiaxed grains containing dislocation cells in the precursor rod and highly elongated dislocation cells and a recrystallized core in the heavily drawn, fine magnet wires. These microstructures are interrupted by entrained, sub-micron copper particles which are carried along during wire drawing, and contribute to failure in a significant way; often limiting the drawability. Dislocations and LEDS evolution do not contribute to wire failure and in fact provide what is effectively a superplastic core which may facilitate the drawing process. Techniques for observing microstructures in both longitudinal and transverse sections of fine copper magnet wires in transmission electron microscopy will also be illustrated. The implications for microinductor and fine-wire sensor applications, etc. will be discussed briefly. Supported by a Murchison Chair Endowment.

4:10 PM

INTERNAL STRESSES AND CRACK TIP FORCES: *K. Sadananda*[1]; A. K. Vasudevan[1]; [1]Naval Research Laboratory, Washington, DC 20375 USA

Crack tip driving forces depend on the remote applied stresses and local internal stresses. The internal stresses arise from internal boundaries, elastic inhomogeneities, inhomogeneous deformations that give rise to strain gradients or dislocation density gradients. All internal stresses can be expressed by appropriate distribution of dislocations (continuum or crystalline) and their effect on crack tip driving forces, in principle, can be quantified. We use a simplified super dislocation

model to illustrate the effects of internal stresses in terms of shielding and anti-shielding effects on cracks and show how the internal stresses can either accelerate or retard the crack growth process in a material. Application of these concepts to acceleration of short cracks and retardation by overloads under fatigue will be presented.

4:30 PM

BRITTLE FAILURE INDUCED BY EMISSION OF SESSILE DISLOCATIONS: *Dr. Diana Farkas*[1]; [1]Virginia Tech., Department of Materials Science and Engineering, Blacksburg, VA 24061 USA

We present a study of the atomistic mechanisms of crack propagation in NiAl. A new failure behavior showing cleavage crack growth after crack-tip dislocation emission is demonstrated using atomistic simulations for an embedded-atom model of NiAl. Dislocations emitted from crack tips normally blunt the crack and inhibit cleavage, inducing ductile behavior. When the emitted dislocations stay near the crack tip (sessile dislocations), they do blunt the crack but brittle cleavage can occur after the emission of a sufficient number of sessile dislocations. Removing the dislocations from the simulation cell just prior to cleavage prevents cleavage and allows further dislocation emission to occur, explicitly demonstrating the embrittling effects of the sessile dislocations. These results suggest a new intermediate mechanism for the brittle-ductile transition that (i) is intrinsic to the crack tip but not controlled by crack-tip dislocation emission and (ii) depends on dislocation mobility but does not require the existence of extrinsic dislocation sources or sharp crack tips.

GENERAL ABSTRACTS: III - Innovations in Titanium

Sponsored by: Light Metals Division, Aluminum Committee, Department of Energy

Tuesday PM Room: Patio A
February 17, 1998 Location: Convention Center

Session Chair: Paul Turner, Albany Research Center, U.S. Department of Energy, Albany, Oregon USA

2:00 PM

DOE TITANIUM INITIATIVE FOR ENERGY EFFICIENCY : *Ms. Toni Grobstein Marechaux*[1]; [1]U.S. Department of Energy, Washington, DC 20585-0121 USA

The Department of Energy is interested in titanium for a wide range of applications in industries and transportation. The ultimate goal of these initiatives is improved understanding leading to more valuable materials applications and better energy efficiency. Programmatic coordination and synergism in DOE's Office of Energy Efficiency and Renewable Energy will be discussed.

2:25 PM

CONTINUOUS PRODUCTION OF TITANIUM POWDER USING CIRCULATING MOLTEN SALT, TiLAC: *Mr. Guy Elliot*[1]; [1]Santa Fe Alloys, Albuquerque, NM 87120-5419 USA

Santa Fe Alloy's TiLAC process seeks continuous production of titanium powder. The TiLAC process, which uses circulating molten salt, moves through two principal stages: Stage 1, where molten salt holding Ti ions reacts with magnesium to produce titanium powder; and Stage 2, where further molten salt holding titanium ions is prepared. It is anticipated that using this process will reduce oxygen, magnesium and other impurities currently found with traditional Kroll production. The TiLAC system is anticipated to be low-cost, safe, and less energy intensive than the traditional systems.

2:50 PM

TITANIUM AND TITANIUM ALUMINIDE POWDERS BY THE FLASH REDUCTION OF TITANIUM CHLORIDE VAPORS OR TITANIUM CHLORIDE/ALUMINUM CHLORIDE VAPOR MIXTURE: *Hong Yong Sohn*[1]; [1]University of Utah, Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA

This concept addresses the production of titanium or titanium aluminide powder directly from titanium chloride vapor or a mixture of titanium chloride and aluminum chloride vapors. The concept will be demonstrated by reducing the vapors by magnesium vapor in a pilot-sized flash reactor. The vapors will react in the furnace shaft to form fine titanium or titanium aluminide powder and magnesium chloride. This process is a continuous production process avoiding some of the traditional batch process problems such as high labor costs, excessive downtime between batches, and difficult quality control.

3:15 PM

PRODUCTION OF LOW-COST TITANIUM POWDERS: *Dr. F.H. (Sam) Froes*[1]; Dr. Oleg Senkov[1]; [1]University of Idaho, Institute for Materials and Advanced Processes (IMAP), Moscow, ID 83844-3026 USA

An innovative method of direct titanium powder production from titanium or with the use of a metal hydride reduction (MHR) process will be developed. The method will lead to virtual elimination of the inherent salt and chloride present in conventional sponge fines powder and therefore to a significantly improved powder quality. The method allows direct production of both commercially pure titanium and prealloyed powders as well as intermetallic titanium aluminide based powders.

3:40 PM Coffee Break in Exhibit Hall

3:55 PM

PRODUCTION OF MICRON-SIZE TITANIUM POWDERS AND NEAR-NET-SHAPE FABRICATION OF TITANIUM COMPONENTS USING SOLID-STATE SPRAY FORMING: *Mr. Ralph Tapphorn*[1]; Mr. Howard Gabel[1]; [1]Innovative Technology, Inc., Menlo Park, CA 94025 USA; [1]Innovative Technology, Inc., Las Cruces, NM 88012 USA

Extremely fine, oxide-free titanium powder is produced from commercial particle feedstock. Powder is produced with a minimum of energy and no environmental pollution. The powder may be continuously formed into near-net shapes with Solid-State Spray Forming or other consolidation processes. This paper will also discuss an energy-efficient, ecologically-sustainable process that employs a low temperature spray forming process to fabricate titanium components which are expected to exhibit exceptionally high ductility and strength.

4:25 PM

UTILIZATION OF A COLD GAS SPRAY COATING PROCESS FOR THE ENHANCEMENT OF TITANIUM: *Mr. Albert E. Segall*[1]; Mr. Anatolii N. Papyrin[1]; [1]Pennsylvania State University, University Park, PA 16804 USA

The CGSM is a promising lower temperature thermal spray method that rapidly and efficiently creates a coating through a process related to friction welding by exposing a metallic or dielectric substrate to a high-velocity jet of solid-phase particles. The particles are accelerated by a supersonic jet of gas at temperatures much lower than the melting or softening temperature of the coating and substrate materials.

4:50 PM

PLASMA ENABLED RECOVERY OF TITANIUM METAL FROM TITANATE SLAGS : *Dr. Harold Larson*[1]; Dr. Eagar Thomas[1]; [1]Massachusetts Institute of Technology, Dept. of Materials Science and Engineering, Cambridge, MA 02139 USA

The concept is to directly reduce titanium metal from a specific titanate slag at significantly lower costs using a reducing plasma. The titanium will be recovered as a dendritic solid in the slag. It is expected that the plasma sponge titanium will have a relatively high oxygen level, but the cost of production is expected to be significantly lower than the traditional processes.

GENERAL ABSTRACTS: VIII - Materials Processing II

Sponsored by: TMS

Tuesday PM Room: Patio B
February 17, 1998 Location: Convention Center

Session Chair: Satyanarayana Kudapa, Michigan State University, Dept. Mat Sci & Mechanics, East Lansing, Michigan 48824

2:20 PM

EVALUATION OF THE STRUCTURE AROUND THE INTERFACE OF BONDED REFRACTORY METALS BY PLASMA ACTIVATED SINTERING: *Mr. Shin-ichi Sumi*[1]; Mr. Yoshiki Mizutai[1]; Dr. Toshihiko Abe[1]; [1]Tohoku National Industrial Research Institute, Materials Engineering Division, Sendai 983 Japan

Plasma activated sintering (PAS) is a kind of hot pressing method. This sintering method is usually for powder sintering. But, it is very effective method for bonding of refractory metals to refractory metals such as tungsten too. For PAS, there are two types electric current during pressing. The surface of the metals is cleaned and activated by activating electric current. The metals are heated due to Joule effect by sintering electric current. The bonded joints of tungsten at the different bonding conditions (temperature, without interlayer, with interlayer of tungsten powder or wire) were evaluated the structure around the bond interface by a microscope and an ultrasonic imaging. This joints will be used for the gun parts of ion implantation devices.

2:40 PM

CHARACTERIZATION OF METALLIC SUBSTRATES WITH BIAXIAL TEXTURE FOR Y-123 COATED CONDUCTORS: *Dr. J. L. Reeves*[1]; D. C. Larbalestier[1]; C.L.H. Thieme[2]; M. L. Jowett[2]; E. Thompson[2]; L. G. Fritzemeier[2]; [1]University of Wisconsin, Applied Superconductivity Center, Madison, WI 53706 USA; [2]American Superconductor Corporation, Westborough, MA 01581 USA

Metallic substrate materials with excellent biaxial texture were made for Y-123 thin film deposition. Texture was measured on samples taken along long lengths of substrates and across their width to establish the statistical relevance of their quality, using pole figure analysis. Orientation Imaging Microscopy was also used to characterize these samples, and revealed that low angle grain boundaries dominated the microstructure. The “rogue” grains with large misorientation angles were usually twin boundaries which might limit Jc in epitaxially deposited Y-123 layers. Differences between pure metals and alloys will be discussed, as will the effect of a reduced grain size on grain boundary angles.

3:00 PM

A NOVEL METHOD FOR PROCESSING LAMINATED TI/AL INTERMETALLIC STRUCTURES: *Viola L. Acoff*[1]; [1]The University of Alabama, Dept. of Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202

The processing and fabrication of certain metal alloys and layered composites can be expensive due to the sophisticated manufacturing equipment that is required for some existing processing techniques. For example, highly reactive metals such as titanium can be difficult to process in the molten state because molten titanium reacts with oxygen and nitrogen in the air. Special processes requiring vacuum systems and water-cooled copper crucibles must be used to produce titanium and titanium alloy ingots. Several other materials are difficult to process using conventional techniques, including some of those classified as intermetallic compounds. As a class of materials, intermetallic compounds and alloys are of technological interest because they are light in w eight (low density) and maintain high strengths at elevated temperatures (>1100ºC). Limited breakthroughs in cost-effective processing techniques for intermetallics are partially responsible for the lack of their wide spread use in industry. This study investigates roll bonding as a novel, cost-effective, simplistic processing technique for the fabrication of Ti/Al intermetallic compounds.

3:20 PM

THE EFFECT OF CASTING CONDITIONS ON THE OCCURRENCE OF SURFACE CONNECTED POROSITY IN INVESTMENT CAST Ti-48Al-2Nb-2Cr, Ti-48Al-2Nb-2Cr + 0.5 AT. % B AND Ti-45Al-2Nb-2Cr + 0.9 AT. % B PLATES: *Linda L. Rishel*[1]; Tresa M. Pollock[1]; Alan W. Cramb[1]; Donald E. Larsen[1]; Paul A. McQuay[1]; [1]Carnegie Mellon University, Materials Science and Engineering, Pittsburgh, PA 15213-3890 USA

Cast Gamma alloys are currently being critically evaluated as an alternative material to nickel based superalloys for aerospace applications. A series of experiments were conducted to study the effect of processing parameters on the development of surface connected porosity in investment cast Ti-48Al-2Nb-2Cr, Ti-47Al-2Nb-2Cr+0.5 at. % B and Ti-45Al-2Nb-2Cr+0.9 at. % B (XD?) alloy plates. Mold preheat, mold insulation, section thickness, and boron level were varied to examine the effect of cooling rate, solidification sequence, and as cast morphology on the distribution and occurrence of defects, most notably surface connected porosity. Surface connected porosity and total porosity were quantified and will be presented as function of processing parameters and gas content. Conditions promoting and minimizing surface connected porosity will be discussed along with key mechanisms for the formation of porosity and surface connected porosity.

3:40 PM Coffee Break in Exhibit Hall

3:50 PM

MECHANISMS OF SHOCK INDUCED SYNTHESES OF INTERMETALLICS: *Fernand D.S. Marquis*[1]; [1]South Dakota School of Mines and Technology, Department of Metallurgical and Materials Engineering, Rapid City, SD 57701

The in situ synthesis and densification of advanced materials is of considerable scientific and technological interest because it makes possible the formation of near net shape parts of ceramics, intermetallics and composites. Shock Induced Synthesis (SIS) is characterized by the simultaneous generation of extremely high pressures, very high temperatures and enhanced particle reactivity, all within a few microseconds, which makes it possible to synthesize phases with unique properties. Because of the very short duration of the reaction synthesis a fully dense material, of very fine grain size, and of very high purity can be obtained. However, the achievement of microstructural control, through the achievement of good experimental parameter control is necessary, together with a good understanding of the synthesis mechanisms. This paper discusses the role of parameters such as: shock pressure, thermal energy and deformation energy and mechanisms, on the synthesis of metal disilicides and correspondent microstructural control.

4:10 PM

THE SYNTHESIS AND RESEARCH OF PLATINUM-IRIDIUM DIOXIDE THIN FILM: *D. V. Malevich*[1]; A. F. Masez[1]; V. B. Drozdovich[1]; I. M. Zharskii[1]; [1]Belorussian State Technological University, Department of Electrochemistry, Minsk 220630 Belarus

The thin films which contain of Pt and Ir dioxide on Ti-support were synthesized by method of thermal decomposition of substances. These films have large interest for electrochemistry as anodic electrocatalysts in reaction of oxygen reception, microelectronics etc. It was established, that oxides of Ir had a form of film (thickness < 1000 nm) on a surface of the support. Platinum was contained as a individual phase (size of particles was 10-100 nm), and as component in the oxide film. We observed reduction of activity of mixed Pt-Ir films in oxygen evolution reaction in comparison with pure films. The assumption about catalytical action of a platinum on the iridium dioxide oxidation was made. For check of this assumption we had put a platinum on the surface of a pure Ir dioxide film. The reduction of activity at increase of the contents on a surface was observed. However activity of electrodes was restored at increase of the contents of a platinum on the surface is higher then 0.2 ?/?2. In

this case the platinum participates in the electrode process as the catalyst of oxygen evolution.

4:30 PM
THE EFFECTS OF BORON AND CARBON ADDITIONS ON THE PROCESSING OF A BETA TITANIUM ALLOY: *Mr Chen Li-Hung*[1]; [1]University of Birmingham, IRC in Materials, Birmingham B15 2TT England

Small additions (01-0.4wt%) of carbon and boron have been added to a Ti-15Mo-based b-Ti alloy for grain refinement. The as-cast, as-forged and solution treated microstructures have been characterized. In the as-cast state the grains remained columnar though a significant reduction in DAS was observed. The additions had little effect on the forgeability but the as-forged microstructure was much refined and more homogeneous. Grain growth after solution treatment at various temperatures (850-1200°C) was strongly inhibited by precipitate particles, the mean grain size being 2-4 times smaller than that of the base alloy. A series of isothermal compression tests between 800-1100°C at strain rates of 3x10-2 - 3 x 10-4s-1 were also carried out to establish the relationship between forging conditions and the microstructure. The overall implications of these particles on the processing and grain size will be discussed.

4:50 PM
COLUMNAR TO EQUIAXED TRANSITION STUDIES IN LEAD TIN ALLOYS: *A. E. Ares*[1]; C. E. Schvezov[2]; [1]CONICET, Quimicas y Naturales, (3300) Posadas, Misiones Argentina; [2]Facultad de Ciencias Exactas, Quimicas y Naturales, (3300) Posadas, Misiones Argentina

The columnar to equiaxed transition was studied in Pb-Sn alloys solidified directionally from a chill face. The reported results are the temperature gradients and solidification velocity. The transition was observed to occur when the temperature gradient in the melt ahead of the front between the liquid phase and solid plus liquid (mushy zone) decreased to -0.8 deg C/cm to 1.2 deg C/cm. In addition there was an increase of the size of the mushy zone. The effect of composition, number of columnar grains, heat extraction was determined and analyzed. The results were compared with available models.

TUESDAY PM

GENERAL RECYCLING OF NON-FERROUS METALS: General Recycling II - Thermal Processing

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

Program Organizers: Courtney Young, Montana Tech, Metallurgical Engineering, Butte, MT 59701; John P. Pickens, Alumitech Inc., 10380 Route 43, Streetsboro, OH 44241

Tuesday PM Room: Plaza Room B
February 17, 1998 Location: Convention Center

Session Chair: Ray D. Peterson, Muscle Shoals, AL 35661-1258, John W Pickens, Streetsboro, OH 44241

2:30 PM
RECENT DEVELOPMENT OF WAELZ KILN PROCESS FOR EAF DUST TREATMENT AT SUMITOMO SHISAKA WORKS: *Atushi Kaikake*[1]; [1]Sumitomo Metal Mining, Shisaka Works, Niihama, Ehime 792 Japan

Sumitomo Shisaka Works has been treating EAF (Electric Arc Furnace) dust to recover zinc and lead by Waelz Kiln process. Since its commencement of operation in 1977 many technical improvements have been successfully made to increase the production capacity and zinc recovery rate. The EAF dust charge to the Waelz Kiln was increased from 35,000t/y to 120,000t/y, and production of zinc oxide now amounts to 40,000t/y. Zinc content in clinker was decreased from 5% to less than 1%, by the optimization of kiln heat pattern with no accretion trouble. And the clinker has become more valuable raw material for steel making. This paper describes the above progress of SMM Waelz Kiln process operation at the Shisaka Works.

2:55 PM
INERTIZATION AND RECYCLING OF WASTES CONTAINING ASBESTOS WITH THE CORDIAM METHOD: *Giralamo Belardi*[1]; A. Imerito[2]; D. Maccari[1]; A. M. Marabini[1]; P. Plescia[1]; [1]Consiglio Nazionale delle Ricerche, Istituto per il Trattamento dei Minerali, Roma 7-00138 Italy; [2]Ecotec S.R.L., Roma 2 - 00100 Italy

Wastes containing asbestos are a specific sort: they second only to urban solid wastes in volume and first among toxic wastes in quantity. This contribution examines a method called the "CORDIAM procedure" for the inertization and the treatment of wastes containing asbestos through low-temperature, metamorphic-like reactions and the consequent transformation of thus gained inerts for the production of ceramics of different quality. Among the methods that are being used for the inertization include dilution of cement matrices, vitrification through plasma techniques, vitrification through standard fusion with fluxes, clinkerization, pyroceramization, and ceramization. Obtained products may not be recycled though, or are only recycled for a minimum part in the industry and are generally disposed of in landfills. The first operative example of this technology process, foresees a transformation of wastes containing asbestos in their solid state with a natural catalyst at a temperature between 850°C an d 950°C, thus permitting to recycle different sorts of inorganic wastes with reactivity features suitable to produce highly refractory and very low thermal expanding ceramics, ceramics for frits and tiles, according to the sort of in-going wastes and to the size of the natural catalyst. The thermal behaviour of asbestos minerals is being examined as well as the behaviour of mixtures with natural catalysts used for the transformation process in order to have useful data for the planning of furnaces and in general for the industrial scale-up of this technology. A comparison with several of the most generally used techniques for the inertization of wastes containing asbestos is then carried out.

3:20 PM INVITED
THERMODYNAMIC CONDITIONS FOR THE FORMATION OF DIOXIN DURING THE RECYCLING OF NON FERROUS METALS FROM ELECTRIC AND ELECTRONIC SCRAP: *Dr. Nourreddine Menad*[1]; Dr. Bo. Bjorkman[1]; Dr. Eric Forssberg[2]; Mr. Shunli Zhang[2]; [1]Lulea University, Process Metallurgy, Lulea, Lulea S-971 87 Sweden; [2]Lulea University, Mineral processing

Plastic materials have been associated with electric and electronic applications since the early days of the electrical industry. Their amount in the present study is about 30 %. Generally, they are treated with flame retardants such as halogenated ones and they can result in different toxic chemical compounds. These plastics can be used as combustibles to recover copper and some precious metals. However, during their combustion, halogenated flame retardants can produce dibenzo-p-dioxins and dibenzo-furans. The estimated thermodynamic data of several organic compounds resulting from combustion of electric and electronic scrap, has been used in conjunction with the program HSC to calculate the conditions for dioxin and furan formation. The results show that the formation of the dioxins can be dependent on the quantities of carbon, hydrogen, chlorine and oxygen reacting in a given system, as well as on parameters such as temperature and pressure. Keywords : Electric and Electronic Scrap, Plastics, Flame Retardant, Combustion, Thermodynamic.

3:45 PM Coffee Break in Exhibit Hall

4:15 PM INVITED
RESOURCES AND RECYCLING PROCESSES IN NON-FERROUS METALS INDUSTRY OF JAPAN: *T. Nakamura*[1]; M. Maeda[2]; [1]Kyushu Institute of Technology, Materials Science and Technology, Kitakyushu 804 Japan; [2]University of Tokyo, Tokyo Japan

Metal production industries consume a large amount of energy and resources and also produce a large amount of waste. Metals and their alloys, however, are one of most important materials to support hu-

man life. This means that all metallurgists and materials scientists need to make an effort to save energy and minimize waste not only in production of metals but also in use of metals. In other words, recyclability has to be considered when new alloy compositions for the next generation are developed to achieve a sustainable development of Japan. Resource and material accountabilities of non-ferrous metals such as Al, Cu and Zn in Japan have been investigated. In this paper, recycling processes for those metals in the present time are briefly reviewed and guide-lines for new alloy design are proposed from a thermodynamic point of view.

4:40 PM
FORMATION OF HARD SPOTS IN SECONDARY BRASSES: *Mrs. Isrun Bohlinger*[1]; Prof. Dr.-Ing. Wolfgang Wuth[1]; [1]TU Berlin, Institute of Metallurgy, Berlin D-10623 Germany

Resources recovery of brasses for semifinished products often results in the formation of hard spots impairing the quality of following abrasive and/or surface finishing processes. A short survey is given on the recycling processes, followed by the actual knowledge on genesis, composition, and possibilities of avoidance of hard spots. Informations on genesis and composition of hard spots are completed by data from recent experiments.

5:05 PM INVITED
NEW MODEL FOR ASSESSMENT OF METAL PRODUCTION AND RECYCLING SYSTEM: *Kiyoshi Shibata*[1]*;* Dr. Yoshio Waseda[1]; [1]Tohoku University, Institute for Advanced Material Processing, Sendai, Miyagi 980-77 Japan

A new assessment model for metal production and recycling system has been developed. The model consists of three processes, smelting of ore, cascading and upgrading of metal scrap. The present approach is to assess the total energy consumption of the system, by introducing two equations for describing the relationships between critical impurity content and the mass of production or scrap recycling. The total energy consumption in the system can be described as a function of recycling rate and performance of the upgrading process. The model calculation results suggest that the impurity removal efficiency and yield in the upgrading process are essential to reduce the energy consumption in the systems. Especially, such points should be explicitly controlled in the aluminum recycling. The potential capability of this model, such as effectiveness for the development and operation control of beneficial recycling of metals by obtaining several quantitative information, will be discussed.

HARD COATINGS BASED ON BORIDES, CARBIDES & NITRIDES: SYNTHESIS, CHARACTERIZATION & APPLICATIONS: Session II

Sponsored by: Materials Design and Manufacturing Division, Surface Modification & Coatings Technology Committee
Program Organizers: Yip-Wah Chung, Northwestern University, Dept. of Materials Sci & Eng., Evanston, IL 60208; Ray W.J. Chia, Western Digital Corporation, 2109 Tasman Dr., Santa Clara, CA 95054; Ashok Kumar, University of South Alabama, Dept. of Electrical & Comp Eng., Mobile, AL 36688-0022

Tuesday PM Room: Centro Room C
February 17, 1998 Location: Convention Center

Session Chair: J. Narayan, North Carolina State University, Department of Materials Science and Engineering, Raleigh, NC 27695, R. Alexandrescu, National Institute for Lasers, Bucharest Romania

2:00 PM
DEPOSITION, STRUCTURE, AND MECHANICAL PROPERTIES OF NITRIDE-BASED SUPERLATTICE THIN FILMS: *Scott A. Barnett*[1]; Anita Madan[1]; Ilwon Kim[1]; [1]Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208

In this talk, mechanical properties of superlattice thin films are reviewed. The deposition of multi-layered films by reactive magnetron sputtering, and the details of their resulting structures, is described. Three types of superlattices will be discussed The first type consists of two rocksalt-structure transition-metal nitrides, e.g. TiN/NbN. These materials exhibit high hardness (~50 GPa) for bi-layer repeat periods ~ 5 nm, compared with ~ 20 GPa for the corresponding homogeneous films. In this case, dislocation glide across layers is impeded due to shear modulus differences and coherency strains, leading to the hardness enhancement. Miscibility of the layers leads to broadened interfaces, limiting the hardness. The second type consists of immiscible layers with different structures. One example is the combination of BCC metals with a transition-metal nitride. For example, Mo/NbN superlattices show substantial hardness enhancements (~ 30 GPa) at periods of 1-2 nm, versus 3 (Mo) and 16 (NbN) GPa . Because the dislocation glide systems of the layers are different, dislocations should be effectively confined within layers. The maximum hardness is limited by some other mechanism however, perhaps by the metal reaching its theoretical strength limit. In the third type of superlattice, one layer is forced into a metastable structure by the surrounding layers, providing a broad new range of materials to be explored. One example in AlN/TiN, where AlN layers are in the rocksalt structure for thicknesses up to 2 nm, but transform to the equilibrium hexagonal structure for larger thicknesses.

2:30 PM
COMPOSITIONAL TAILORING OF Cr-N THIN FILMS FOR WEAR APPLICATIONS: *A. Michael Peters*[1]; J. J. Moore[1]; B. Mishra[1]; [1]Colorado School of Mines, Advanced Coatings and Surface Engineering Laboratory, Golden, CO 80401

In a continuing study, Cr-N thin films were produced over a range of compositions. Films were deposited onto mill annealed and heat treated M4 tool steel to examine the effects of substrate hardness. Properties of hardness, critical load and multi-pass scratch testing were done on films. Bias and nitrogen pressure were varied systematically to produce desired properties. Preliminary results have indicated that films rich in Cr2N have higher harnesses while films rich in Cr-N exhibit higher critical loads.

2:50 PM
CHARACTERIZATION OF TITANIUM NITRIDE FILMS PRODUCED BY PHYSICAL VAPOR DEPOSITION TECHNIQUES: *M. A. George*[1]; J. J. Weimer[1]; Ashok Kumar[2]; [1]University of Alabama in Huntsville , Materials Science Ph. D. Program, Huntsville, AL 35899; [2]University of South Alabama, Electrical Engineering Department, Mobile, AL 36688

The success of titanium nitride (TiN) as a protective coating is attributed to its exceptional properties, such as its hardness, chemical stability, adhesion and wear resistance, and corrosion resistance, In this work, TiN film were deposited on silicon substrates using either pulsed laser deposition or magnetron sputtering. The resulting polycrystalline films were subsequently characterized using surface microscopic and spectroscopic techniques. The surface morphology of the films was mapped with atomic force microscopy (AFM). Surface roughness and film grain sizes were determined based on images taken at various resolutions down to submicron in scale. The surface chemistry of the films was characterized by x-ray photoelectron spectroscopy(XPS). Differences in the Ti/N composition ratio and the oxidation states of the Ti were determined. The results are presented in terms of a comparative analysis between the two deposition techniques. This research was supported by Alabama NASA EPSCoR Program.

3:10 PM

MICROSTRUCTURE AND WEAR BEHAVIOR OF HIGH-NITROGEN 304L SS COATINGS PRODUCED BY FLAME SPRAY TECHNIQUES: *J. W Simmons*[1]; J. A. Hawk[1]; [1]Albany Research Center, U.S. Department of Energy, Albany, OR 97321

In this study, the microstructure and wear behavior of high-nitrogen (0.07 to 2.63 wt %N) 304L stainless steel coatings produced using a flame spray system are presented. The stainless steel powders used to make the coatings were solid state nitrided in pure N2 gas using mechanical fluidized vacuum (MFV) technology (i.e., a mechanical fluidized bed). The coatings were produced on 304 stainless steel substrates with and without Ni-Al bond coats, using both H2 and acetylene as fuels. The microstructures of the coatings have been characterized through bulk and microchemical analyses, X-ray diffraction, and optical and electron microscopy. The phases and phase morphologies present in the coatings are compared to the original high-nitrogen 304L starting powder. Abrasive wear behavior of the coatings were determined at ambient conditions using two- and three-body abrasion test methods. The severity of the two- and three-body abrasive particle-coating interaction, and the mechanisms of material removal for each tribo-environment have been determined using optical and scanning electron microscopy. The wear behavior of high-nitrogen coated materials have been compared to 304SS.

3:30 PM Coffee Break in Exhibit Hall

3:45 PM

IN-SITU CHARACTERIZATION OF THE VACUUM TRIBOLOGY OF DIAMOND-LIKE CARBON COATINGS: *James Arps*[1]; Paul Lacey[1]; Michael Miller[1]; Darrel Dunn[1]; [1]Southwest Research Institute, San Antonio, TX 78228

The Southwest Research Institute is actively involved in the characterization and development of solid lubricant coatings. Of particular interest has been the application of diamond-like carbon(DLC) coatings by ion beam assisted deposition (IBAD) to address a variety of friction and wear problems. Using an organic precursor, IBAD of DLC can produce hard (12 - 20 GPa) conformal coatings on metal, ceramic, or polymeric substrates with minimal stress (< 50 MPa) and low friction in air over a range of relative humidity. A brief overview of the process, coating facilities, and some specific applications will be provided. Recently, efforts have focused on the development and use of a novel ball-on-ring tribometer with an in-situ scanning electron microscope capability to study the performance of DLC coatings in vacuo.. Comparison of DLC performance at different vacuum pressures, the effect of composition and microstructure, and the influence of the counterface material will be discussed.

4:15 PM

HARDNESS, BONDING, AND SURFACE ROUGHNESS OF HYDROGEN-FREE DIAMOND-LIKE CARBON FILMS SYNTHESIZED BY DIRECT ION BEAM DEPOSITION: *Ian Widlow*[1]; Murat Guruz[1]; Laurence D. Marks[1]; Vinayak P. Dravid[1]; Yip-Wah Chung[1]; [1]Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208

Recent molecular dynamics simulation showed that when carbon atoms are deposited onto a surface with controlled kinetic energies, there is a strong correlation between the local bonding configuration of carbon and its arrival energies. In particular, the sp3 content of the resulting film is highest at arrival energies ~ 100 eV. This implies that carbon films deposited at these energies should be the hardest. This was observed to be the case in a recent study using direct ion beam deposition of negative carbon ions. We are extending this study not only to confirm the previous hardness results, but also to explore how the local bonding and surface roughness vary with incident carbon ion energies. The local bonding will be determined from electron energy loss measurements, while the surface roughness will be measured by atomic force microscopy. These studies indicate that it is possible to synthesize smooth and ultrahard diamond-like carbon films without hydrogen under optimum process conditions, and that these films are likely to be useful as ultrathin protective overcoats for the next generation computer disk drives.

4:35 PM

ADHESION OF DIAMOND ON WC/CO BASED SUBSTRATES: Dong-Gu Lee[1]; *Rajiv K. Singh*[1]; [1]University of Florida, Department of Materials Science and Engineering, 311 MAE, Gainesville, FL 32611

Adhesion of diamond films on cemented carbide tools has been a critical issue due to large mismatch of thermal expansion coefficients. Adhesion is mainly influenced by stresses at the interface, chemical interaction between diamond film and WC-Co substrate, and surface roughness. We focused on the surface modification of cemented carbide tools using KrF pulsed laser to give surface roughness, leading to mechanical interlocking. Laser conditions such as fluence, number of pulses, and processing atmospheres were changed to observe the surface roughness, phase changes, and adhesion of cemented carbide tools to diamond films. Diamond films were characterized by Raman spectroscopy, atomic force microscopy, Rockwell hardness indentation, and scanning electron microscopy.

4:45 PM

CHARACTERIZATION OF HARD SURFACE IMPURITY FILMS ON Al-5% MG ALLOY PREPARED BY THERMOMECHANICAL PROCESSING: Fenglin Liu[1]; *M. Shamsuzzoha*[1]; [1]The University of Alabama, Department of Metallurgical and Materials Engineering, Tuscaloosa, AL 35487

Microstructure and hardness of surface impurity films of an Al-5% Mg alloy prepared by drawing and annealing have been investigated using transmission electron microscopy, x-ray diffraction, and nano-indentation techniques. The impurity film is distributed nonuniformly on alloy surface. Investigations on films of thickness ranging between 0.13 to 0.50 υm revealed the existence of a bilayer film structure. The interior layer, which lies next to matrix, is constituted of phases such as Al, Mg, Al_2O_3 and MgO_2, all of which are mainly crystalline and exhibit granular microstructure. The film present in outer layer appears featureless and is mainly comprised of an amorphous Al_2O_3 Phase. The measured hardness of impurity layer is somewhat smaller than that observed in bulk Al_2O_3, but is larger than that of the matrix.

HIGH TEMPERATURE SUPERCONDUCTORS: YBCO Superconductors

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

Program Organizers: U.Balu Balachandran, Argonne National Laboratory, 9700 S. Cass Ave. Bldg. 212, Argonne, IL 60439; Pradeep Haldar, Intermagnetics General Corp., 450 Old Niskayuna Rd., Latham, NY 12110; Paul McGinn, University of Notre Dame, Center for Materials Science, Notre Dame, IN 46556

Tuesday PM Room: Fiesta C
February 17, 1998 Location: Convention Center

Session Chair: K. Salama, Univ of Houston, Texas Center for Superconducting, Houston, Texas 77004

2:00 PM INVITED

STUDY OF THE SINGLE DOMAIN STABILITY IN MELT TEXTURED YBCO LEVITATORS: *Dr. K. Salama*[1]; Dr. Sherif Salib[1]; [1]University of Houston, Texas Center for Superconductivity, Houston, TX 77204-4792

To study the single domain stability in bulk YBCO superconductors a large number (>500) of melt textured YBCO disk-shaped levitators were manufactured using a seeding technique. The trapped magnetic field of these levitators range between 0.45 and 0.65 Tesla, while the levitation forces are between 25 and 35 Newtons. In order to determine the extent of single domain structure along the thickness of the

levitator, we studied the effects of changing the height of the disk and changing the thickness of the 211 base on its superconducting and microstructural properties. The results of these experiments show that regardless of the initial conditions of the sample, the thickness of the single domain remains the same, indicating that the growth stability in these disks is independent of their original heights and thickness of the 211 bases. Microstructural examinations on these disks after cutting support these findings.

2:20 PM INVITED

THE EFFECT OF PLATINUM ADDITIONS ON Y_2BaCuO_5 PRECIPITATION AND COARSENING DURING MELT TEXTURING OF $YBa_2Cu_3O_7$: *Prof. Paul J. McGinn*[1]; Thomas J. Meignan[1]; [1]Univ. of Notre Dame, Chemical Engineering, Notre Dame, Indiana 46556 USA

The effect of Pt additions on the various stages of Y_2BaCuO_5 (211) development was studied through the use of $YBa_2Cu_3O_{7-d}$ (Y-123) pellets coated with $Ba_4CuPt_2O_9$ (0412). It is observed that the 0412 compound is readily dissolved in the peritectic melt. Pt in solution promotes nucleation of 211 by decreasing the critical nucleus radius. Pt also reduces the 211 coarsening rate by decreasing the 211/liquid interfacial energy. This behavior is similar to that observed in pellet coated with $BaCeO_3$.

2:40 PM INVITED

GROWTH ANISOTROPY AND ELECTROMAGNETIC PROPERTIES IN SEEDED MELT GROWN $YBa_2Cu_3O_X$: *Dr. Donglu Shi*[1]; Dr. D. Qu[1]; Dr. Brian Tent[1]; [1]University of Cincinnati, Cincinnati, OH 45221-0012 USA

By varying cooling conditions, we have obtained different growth rates in seeded melt growth of $YBa_2Cu_3O_x$. We have found that the planar growth of solid Y123 interface exhibits anisotropy, particularly at high growth rates. In the two groups of samples studied, we have found that the growth rate is not necessarily dependent on undercooling applied, but rather dependent on the overall bulk growth rate which may be considered a more fundamental parameter. A physical model is proposed to explain the growth anisotropy. Thermal conditions that favor planar growth will maximize growth rate along both the a and c axis, thus causing a pronounced anisotropy. Slow planar growth due to poor thermal conditions will limit R_a more effectively (the variation in R_c is relatively small under all conditions), therefore reducing the anisotropy. Effect of processing on electromagnetic properties such as flux pinning and levitation force will also be discussed.

3:00 PM INVITED

THE INFLUENCE OF INTRAGRANULAR DISSIPATION AND BOUNDARY STRUCTURE ON CURRENT TRANSPORT ACROSS GRAIN BOUNDARIES IN $YBa_2Cu_3O_7$: *Dr. Dean J. Miller*[1]; Dr. Michael B. Field[1]; Dr. Kenneth E. Gray[1]; [1]Argonne National Laboratory, Materials Science Division, Argonne, IL 60439 USA

The transport behavior across grain boundaries in $YBa_2Cu_3O_{7-x}$ (YBCO) has been studied by a variety of researchers using both thin film and bulk bicrystals. While intragranular regions in YBCO thin films typically exhibit very high critical current densities (J_c), the intragranular Jc in bulk samples are generally much lower. At the same time, the grain boundary structure of these two types of samples can be very different. As a result, the relative contributions of intragrain and grain boundary dissipation to the total dissipation measured across a bicrystal sample may be different in each case. Understanding the relative contributions of these two sources of dissipation is a significant issue in evaluating the potential of various conductors. We will compare transport measurements across grain boundaries in bulk bicrystals with those of thin film samples and irradiated bulk bicrystals to elucidate the relative contribution of these two factors and to illustrate role each of these may play in transport within a practical conductor. This work was partially supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under contract #W-31-109-ENG-38 and by the National Science Foundation through the Science and Technology Center for Superconductivity under contract #DMR 91-20000.

3:20 PM

PROCESSING OF $Y_1Ba_2Cu_3O_X$ FILMS BY SOLUTION TECHNIQUES USING METAL ORGANIC DECOMPOSITION.: *Mr Srivatsan Sathyamurthy*[1]; Dr. Kamel Salama[1]; [1]Texas Center for Superconductivity, Houston, Texas 77204 USA

Processing of YBCO films by decomposition of metal trifluoroacetate precursors and its application to the development of coated conductors is investigated. The technique involves preparation of the solution, deposition, and a two stage heat treatment. A stoichiometric mixture of the acetates of Y, Ba, Cu and trifluoroacetic acid are used as the starting materials. The glassy residue of the trifluoroacetates of Y, Ba, and Cu formed by drying the above mixture is redispersed in a fixed amount of methanol to give the starting solution of a given concentration. This solution is then deposited on the substrate surface by spin coating and heat treated in two stages. X-Ray characterization shows that the transformation to 123 is complete in less than 1 hour. Pole figure analysis of the films deposited on (100) strontium titanate and lanthanum aluminate single crystal substrates show that these films have a very high degree of alignment with the substrate both out-of-plane (<0.5 degrees) and in-plane (<1.5 degrees). These chemically derived films were also found to have critical current densities well above 0.5 MA/sq.cm at 77K for 0.5 micron thick films. The applicability of this technique to the production of thick films of YBCO for the development of coated conductors will be discussed. * The work was partially supported by the superconductivity program at the Oak Ridge National Laboratory (ORNL).

3:40 PM Coffee Break in Exhibit Hall

3:50 PM INVITED

EFFECTS OF Mg-Ce ADDITIONS ON THE MAGNETIC PROPERTIES OF MELT TEXTURED $YBa_2Cu_3O_{7-D}$: *Prof. Paul J. McGinn*[1]; Sharon Yeung[1]; [1]Univ. of Notre Dame, Chemical Engr., Notre Dame, Indiana 46556 USA

The effects of Ce-based additions (CeO2 and BaCeO3) in combination with MgO additions on the magnetic properties of melt textured $YBa_2Cu_3O_{7-d}$ have been investigated. The additions lead to improvements in the magnetic properties of $YBa_2Cu_3O_{7-d}$ compared to samples with either addition alone or with no additions. The Ce-Mg addition combination produces a "peak effect" in the magnetic hysteresis loop. This is postulated to be due to the formation of a new type of pinning center. Both Ce and Mg ions are thought to substitute on the Y site in the $YBa_2Cu_3O_{7-d}$ lattice, creating defects that produce a "peak effect" in the magnetic hysteresis loop. Mg additions alone lead to a reduced Tc, while Ce additions restore the Tc and enhance the magnitude of the peak.

4:10 PM INVITED

CATION SUBSTITUTION AND SUPERCONDUCTIVITY IN $Lr1+XBa_{2-X}Cu_3O_7$: *R. W. McCallum*[1]; H. Wu[1]; M. J. Kramer[1]; K. W. Dennis[1]; Y. Xu[1]; [1]Ames Laboratory, Ames, IA 50014 USA; [1]Mankato State University, Department of Physics, Mankato, MN 56002

The Light Rare Earth Elements (LR = La to Gd) form extensive solid solutions when incorporated in the $YBa_2Cu_3O_7$ structure. Substitution of LR^{3+} on the Ba^{2+} site results in a uniform depression of T_c which has been attributed to oxygen forced onto the anti-chain sites in order to compensate for the 3+ charge on the Ba site. It has previously been demonstrated that oxygen partial pressure has an important effect on the processing of $LR_1+xBa_{2-x}Cu_3O_7$ superconductors with a pronounced narrowing of the range of solid solution when the oxygen partial pressure is 0.01 atm and below. This effect may be used to cause the precipitation of second phases during the oxygen annealing process. The dispersed precipitates act as strong flux pinning centers, giving a high critical current density. As the degree of narrowing is a function of LR, the size and density of pinning centers may be varied by the appropriate choice of LR.

4:30 PM INVITED

CHEMICAL CONTROL OF UNDERDOPED AND OVERDOPED STATES IN $Y(Ba_2\text{-}YSRY)Cu_3O_{6+\delta+}$: *Dr. R. S. Liu*[1]; Dr. C. Y. Chang[1]; Dr. J. M. Chen[2]; Dr. R. G. Liu[2]; [1]; [1]National Taiwan University, Dept. of Chemistry, Taipei, Taiwan ROC.

The chemical control of underdoped and overdoped states in the $Y(Ba_{2-y}Sr_y)Cu_3O_{6+\delta}$ ($\delta \sim 0.1$ and 0.9) compounds has been observed by high-resolution O K-edge and Cu L_{23}-edge x-ray-absorption near-edge-structure spectra. Increasing the Sr content in the oxygen-deficient $Y(Ba_{2-y}Sr_y)Cu_3O_{6+\delta}$ ($\delta \sim 0.1$) compounds induces weak superconductivity at temperatures below 10 K. The oxygen-deficient compounds exhibit underdoped state due to the low hole concentration. In contrast, the chemical substitution of Sr for Ba in the fully-oxygenated $Y(Ba_{2-y}Sr_y)Cu_3O_{6+\delta}$ ($\delta \sim 0.9$) compounds gives rise to high hole concentrations within both the CuO_2 planes and the out-of-plane sites, leading to the overdoped state and the decrease of the superconducting transition temperature from 92 K for y = 0 to 84 K for y = 0.8.

4:50 PM

PREPARATION AND CHARACTERIZATION OF HIGH ENERGY BALL MILLED YBCO NANOSIZED POWDERS: *Mr. Hui Fang*[1]; Professor Krishnaswamy Ravi-Chandar[1]; [1]University of Houston, Department of Mechanical Engineering, Houston, TX 77204-4972 USA

Melt textured processing is the more common method for fabrication of bulk YBCO superconductors; this process involves a peritectic reaction in which 211 solids react with an yttrium-deficient liquid phase to form the 123 composition. However, the sluggish nature of this processing impedes the practical application of this process to bulk YBCO. It has been known that for a given volume fraction of 211 in the melt, a decrease in the 211 particle size will result in a reduction in the particle spacing and a more homogeneous distribution of 211, and will result in an increase in the allowable growth rate. Consequently, the peritectic reaction between nanosized 211 with more uniform distribution and yttrium-deficient liquid should lead to a remarkable increase in the growth rate. In this study, we report the preparation and characterization of high-energy ball milled YBCO nanocrystalline materials. XRD, TEM and DTA were employed to characterize the nanosized powders. It is found that after a certain time of milling, the initial orthorhombic 123 phase converts to a nanocrystalline, disordered cubic phase with uniform particle distribution which when heated to the melting temperature decomposes into nanosized 211 particles with a homogenous distribution and an yttrium-deficient liquid phase. The peritectic reaction temperature of this nanocrystalline material is also reported.

5:10 PM

MELT-PROCESSING OF Yb-123 TAPES COATED ON SILVER SUBSTRATE: *Mr. Srinath P. Athur*[1]; Dr. Uthamalingam Balachandran[2]; Mr. Philip Putman[1]; Dr. Kamel Salama[1]; [1]University of Houston, Mechanical Engineering and the Texas Center for Superconductivity, Houston, TX 77204-4792 USA; [2]Argonne National Laboratory, Energy Technology Division, Argonne, IL 60349 USA

In order to demonstrate the feasibility of practical applications of R-123 system superconductors processed by the melt-texturing route, it is necessary to fabricate them in the form of long-length wires or tapes on suitable metallic substrates such as silver. We report on the melt-texturing of Yb-123 coated on silver substrate. Quench experiments were performed to study the growth kinetics in this system. Thin wires of approximately 0.8mm diameter were run through a zone-melting furnace with a gradient of 150 C/cm for a distance of 20mm and quenched. The samples were polished and examined under an optical microscope and under SEM. The maximum growth rate for a stable interface was determined to be 7.2mm/h. These results reveal that by optimizing the growth conditions, it is possible to texture Yb-123 on silver by a melt-processing route. Preliminary results on Yb-123 coated on silver give promise to the manufacturing of Yb-123 on a flexible tape.

HUME ROTHERY AWARD FOR PROF. RYOICHI KIKUCHI: Hume-Rothery Symposium IV

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

Program Organizers: Juan M. Sanchez, The University of Texas, Center for Materials Science, Austin, TX 78712; Lawrence Anthony, The University of Toledo, Dept. of Physics and Astronomy, Toledo, OH 43606

Tuesday PM Room: 104
February 17, 1998 Location: Convention Center

Session Chair: Gerbrand Ceder, MIT, Materials Science and Engineering, Boston, MA 02139

2:00 PM INVITED

FIRST-PRINCIPLES STUDIES OF THE THERMODYNAMIC PROPERTIES OF INTERPHASE AND ANTIPHASE BOUNDARIES IN SUBSTITUTIONAL ALLOYS: *Dr. Mark David Asta*[1]; Dr. Andrew A. Quong[1]; Dr. Stephen M. Foiles[1]; [1]Sandia National Laboratories, Computational Materials Science, 8717, Livermore, CA 94551-0969 USA

The structural and thermodynamic properties of coherent interphase boundaries (IPBs) and antiphase boundaries (APBs) in substitutional alloys have been studied from first principles. In the computational approach a first-principles-based cluster-expansion for the energetics is combined with statistical mechanics simulations which yield thermodynamic properties at finite temperature. In this talk we present results for the temperature dependences of excess free energies and compositional profiles associated with IPBs and APBs in a variety of Al-transition-metal alloy systems. We will include in this talk a comparison of results obtained using a variety of statistical-mechanics approaches including the cluster-variation method, low-temperature expansion and Monte-Carlo simulation. This research was supported by the Office of Basic Energy Sciences, Division of Materials Sciences, U. S. Department of Energy.

2:30 PM INVITED

CVM STUDIES OF ANTIPHASE BOUNDARIES AND INTERPHASE BOUNDARIES: *Dr. Marcel H.F. Sluiter*[1]; Dr. Yoshiyuki Kawazoe[1]; [1]Tohoku University, IMR, 980-77 Sendai, Japan

Some 20 years ago Kikuchi and Cahn used the Cluster Variation Method to study the configurational thermodynamics of antiphase boundaries (APB) and interphase boundaries (IPB) in a binary alloy. In the present work, we will focus on aspects that were not computationally feasible at the time, such as the role of impurities. In particular, a classification of impurities based on features of the ternary phase diagram and the effect of impurities on interfacial segregation and the thermodynamic properties will be discussed.

3:00 PM INVITED

MAGNETIC SURFACE ENHANCEMENT IN FERROMAGNETIC USING CLUSTERS AND THIN FILMS: *Dr. S. Meza-Aguilar*[1]; Dr. F. Aguilera-Granja[2]; Dr. J. L. Moran-Lopez[2]; [1]Institut de Physique el Chimie des Materiaux de Strasburg, Strasburg, 67037 Strasburg Cedex, France; [2]Universidad Autonoma de San Luis Potosi, Instituto de Fisica, San Luis Potosi, 7800 S.L.P., Mexico

We study the magnetic surface enhancement and the Curie temperature in ferromagnetic Ising clusters and thin films as a function of the different geometrical properties of the clusters and surface orientations for thin films. We use a simplify version of the Cluster Variational Method (CVM) that only consider as inequivalent those sites on the surface, while all the internal sites are considered as equivalents regardless of the position. We calculate the critical value of the surface interaction Jsc, that is the value beyond which the surface dominate the

ferromagnetic properties of the system. Comparison with the results of our work and others approximation is done, particularly our results are in agree with those of the reaction field approximation for the case of simple cubic lattice and the FCC(100) of the CVM tetrahedron approximation.

3:30 PM Coffee Break in Exhibit Hall

3:40 PM INVITED
CONTINUOUS DISPLACEMENT CLUSTER VARIATION METHOD STUDIES OF LATTICE DISTORTIONS AND MATERIALS STABILITY: *Dr. Lawrence Anthony*[1]; [1]University of Toledo, Dept. of Physics and Astronomy, Toledo, OH 43606-3390 USA

Recent experimental results have shown the importance of atomic/metallic size effects in determining the stability of ordered intermetallics and bulk metallic glasses. It has also become possible to perform accurate measurements of the deviations of atoms from their equilibrium lattice positions using neutron and x-ray scattering. In this talk, I shall present the results of computational studies of such phenomena using a continuous displacement (CD) implementation of the cluster variation method (CVM).

4:10 PM INVITED
COMPUTATION OF ALLOY PHASE DIAGRAMS BY CONTINUOUS CLUSTER VARIATION METHOD: *Dr. Kinichi Masuda-Jindo*[1]; Dr. R. Kikuchi[2]; [1]Tokyo Institute of Technology, Department of Materials Science and Engineering, Nagatsuta, Yokohama 226, Japan; [2]University of California, Materials Science and Mineral Engineering, Berkeley, CA 94720

A new formulation of the CVM which allows atomic displacement from lattice points is used to calculate the phase stability and phase diagrams of binary alloys. We formulate continuous atomic displacements around lattice points using the point (quasi-chemical scheme), pair and tetrahedron cluster approximations of the CVM [1], and device the grand potential of the alloy systems. In order to obtain the self-consistent solutions of the cluster probability functions, we use the natural iteration method (NIM). We focus our attention on the model 2D alloys as well as fcc binary alloys, like Cu/Au systems. It is shown that the continuous displacement lowers the ordering temperature. In the phase stability calculations of 2D alloys. The local lattice-distortion effects in a disordered Cu-25 at. pct Au solid solution have also been investigated using the cluster probability functions determined by the continuous CVM. For phase separating binary alloys, the change of lattice constant with the composition and the reduction of the transition temperature are shown.

4:40 PM INVITED
CORRECTING THE TETRAHEDRON CVM ENTROPY WITH MONTE CARLO: APPLICATIONS: *Dr. Luiz Guimaraes Ferreira*[1]; [1]Universidade Estadual de Campinas, Instituto de Fisica, 13083-970 Campinas, Sao Paulo, Brazil

The tetrahedron Cluster Variation Method (CVM) (R. Kikuchi, Phys. Rev 81, 988 (1951)) for the face-centered-cubic antiferromagnetic Ising model of nearest neighbor pair interaction is revisited . First, the CVM entropy function of the cluster probabilities Scvm(p) is thought as a state variable defined for any configuration of spins (microstate) instead of a proper entropy. This is allowed because, for any microstate, one can define the probabilities p and calculate the function Scvm(p). Then, using a Monte Carlo algorithm, one determines the entropy function of Scvm. The method is that of the "entropic ensemble" (J. Lee, Phys. Rev. Letters 71, 211 (1993).) and leads to the function S(Scvm). This function is not the expected straight line passing through the origin because, while Scvm may be negative for very symmetric configurations of spins, the entropy itself is always positive. The function S(Scvm) is then used. The first application is to the nearest-neighbor pair interaction model. In a series of Monte Carlo runs we find the cluster probabilities p(T) as functions of temperature, from which we calculate Scvm(p) and the entropy S(Scvm). This entropy is compared with that obtained by temperature integration of the Monte Carlo heat capacity. The agreement between the two results is outstanding, even for temperatures below the antiferromagnetic transition. Thus it looks like that the function S(Scvm) may be an important entropy predictor in Monte Carlo work. The second application was to the phase diagram T(H) of the antiferromagnetic nearest neighbor pair interaction Ising model. As it is well known, pure tetrahedron CVM gives good predictions at H=0 (formation of $L1_0$) and H=8 (formation of $L1_2$), but fails at the triple point (H=3-4) because the transition temperature there is too high. It is also very well known that to predict a good triple point (compatible with the T=1 of MC calculations) one has to go to very large clusters (T. Mori, J. M. Sanchez and D. de Fontaine, Acta Met. 3 3, 1171 (1985); A. Finel and F. Ducastelle, Europhys. Lett. 1, 135 (1986).). In using S(Scvm) instead of Scvm in a CVM program for the model we restored the triple point temperature and field H to their proper values. We are thus being led to the conclusion that the cluster probabilities and correlation functions within the tetrahedron describe fully the statistics of the nearest neighbor interacting fcc Ising model. What shortcomings the tetrahedron CVM approximation may have is related to its not incorporating the highly symmetric configurations (negative Scvm) into the statistics.

TUESDAY PM

INTERNATIONAL SYMPOSIUM ON IRON ALUMINIDES: ALLOY DESIGN, PROCESSING, PROPERTIES & APPLICATIONS: Precipitates and Mechanical Properties

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture Committee, Jt. Electronic, Magnetic & Photonic Materials Division/Structural Materials Division, Alloy Phases Committee, Materials Design & Manufacturing Division, Powder Materials Committee

Program Organizers: S.C. Deevi, Philip Morris USA, Research & Development Center, Richmond, VA 23234; David G. Morris, University of Neuchatel, Inst of Structural Metallurgy, Neuchatel Switzerland; J.H. Schneibel, Oak Ridge National Laboratory, Metals & Ceramics Division, Oak Ridge, TN 37831; Vinod K. Sikka, Oak Ridge National Laboratory, Metals & Ceramics Division, Oak Ridge, TN 37831

Tuesday PM Room: 108
February 17, 1998 Location: Convention Center

Session Chairs: Richard N. Wright, INEEL, Idaho Falls, ID 83451-2218; V. K. Sikka, Oak Ridge National Laboratory, Oak Ridge, TN 37381

2:00 PM INVITED
MICROSTRUCTURAL EVOLUTION AND MECHANICAL BEHAVIOR OF Fe-40Al-0.6C: *Prof. K. Sharvan Kumar*[1]; Mr. Lixin Pang[1]; [1]Brown University, Division of Engineering, Providence, RI 02912 USA

An Fe-40 at.%Al alloy containing 0.6 at.% C was cast, homogenized and hot-extruded. Microstructural characterization of the extruded material was accomplished using optical, scanning and transmission electron microscopy techniques as well as x-ray diffraction and differential thermal analysis. In the as-extruded condition, a fully recrystallized microstructure is recognized with the conspicuous presence of a second phase (perovskite-structure-based $Fe_3AlC_{0.5}$) fairly uniformly distributed within the grains and at grain boundaries. In addition, frequently at grain boundary triple points, but occasionally in the grain interior, dark globules confirmed to be free graphite, were observed. The perovskite carbides were shown to dissolve in the matrix completely in the solid state and this provides an opportunity to quench and age-harden such materials. A variety of heat treatments were undertaken and the resulting microstructures were characterized to enable a more complete understanding of phase transformations in this ternary

alloy system. Precipitate-matrix orientation relationships were determined using TEM techniques. Tensile specimens were machined from both, extruded and extruded and heat-treated alloys and were tested as a function of temperature and strain rate. Fracture surfaces were examined. The mechanical properties will be rationalized on the basis of microstructural observations.

2:30 PM

PRECIPITATION AND DEFORMATION BEHAVIOUR IN IRON ALUMINIDES CONTAINING CARBON AND TITANIUM, ZIRCONIUM OR NIOBIUM: *C. Schlesier*[1]; W. Chen[2]; J. H. Schneibel[3]; R. P. Wahi[1]; [1]Hahn-Meitner-Institut, D-14109 Berlin Germany; [2]Technische Universität Berlin; [3]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

The microstructure and the deformation behaviour of three iron aluminides with the compositions Fe-40Al-0.5X-0.2C-0.05B, at.% (X=Ti, Zr, Nb), have been investigated by means of optical, scanning and transmission electron microscopy and in compression tests. All the three alloys were extruded and exhibited small grain sizes ranging from 12 to 40 µm. Independent of different heat treatments given to these alloys the Ti- and Nb containing alloys formed, as expected, TiC and NbC precipitates, and the Zr-containing alloy contained a ternary intermetallic phase (Fe_6Al_6Zr). In addition, an unidentified Fe-Al-Zr phase with a Zr concentration of approximately 23 at.% as well as ZrC were found. Before mechanical testing, all the alloys were given a heat treatment (1573K / 24 hours) to increase the grain size up to 300 µm. Further, they were aged at 673 K for 120 hours to reduce the vacancy concentration. Compression tests were performed at different temperatures with a constant strain rate of $10^{-4}s^{-1}$. All the three alloys with the large grain size showed the well known stress anomaly of alloys with B2 structure. The stress maximum was reached at 873 K. The dislocation substructure was analyzed for the different deformation temperatures and alloys. This research was sponsored in part by the Division of Materials Sciences, U.S. Department of Energy under contract number DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

2:50 PM

MICROSTRUCTURE OF A QUATERNARY Fe-40Al-0.7C-0.5B ALLOY: *Lixing Pang*[1]; K. S. Kumar[1]; [1]Brown University, Division of Engineering, Providence, RI 02912

The microstructure of an Fe-40Al-0.7C-0.5B alloy was examined; in addition to the matrix B2 phase, four other phases were present. These were $Fe_3AlC_{0.5}$ with the perovskite structure, Fe_2B, a tetragonal boride with the $CuAl_2$ structure, graphite (free carbon), and a new tetragonal phase that is likely coherent with the matrix. An orientation relationship exists between this tetragonal phase and the matrix of the form $[100]_p$ parallel $[100]_m$ and $(001)_p$ parallel $(001)_m$. From selected area diffraction patterns, it is noted that this new tetragonal phase has lattice parameters that can be related to the matrix parameter (a_0) as $a_p=4a_0$ and $c_p=a_0$. The precipitates themselves are rod-shaped with a square cross section. The long axis of the rods coincides with the <100> directions of the matrix and [001] directions in the precipitates. The diagonals of the square cross sections are parallel to the <100> directions in the matrix and [100] or [010] directions in the precipitates. Planar fault-like features are additionally present in the as-extruded material and these also lie on the cube planes of the matrix. These features showed stacking fault-like contrast with the fundamental reflections and APB-like contrast using superlattice reflections. The new tetragonal phase precipitates on the ends of these planar faults, likely on the 1/2[100]-type bounding partial dislocations. High resolution microscopy is currently being performed to identify the nature of the faults and these observations will be discussed.

3:10 PM

TENSILE AND CREEP PROPERTIES OF Fe_3Al-BASED ALLOYS CONTAINING TUNGSTEN: *Yangshan Sun*[1]; Zhonghua Zhang[1]; Feng Xue[1]; [1]Southeast University, Department of Materials Science and Engineering, Nanjing 210096 P.R. China

Tungsten addition to the Fe_3Al-based alloys results in significant influence on tensile properties and creep resistance. For the binary Fe-28Al and the ternary Fe-28Al-5Cr alloys, the yield strength increases notably, while the ductility decreases slightly with the increase of tungsten addition at ambient and high temperatures up to 650°C. The creep resistance is also greatly improved with tungsten addition. The creep rupture life of Fe-28Al-1.5W alloy at 600°C and 200 MPa has increased to 280 h compared to 4 h for the binary Fe-28Al alloy. The main change in microstructure caused by tungsten-rich precipitates. Electron diffraction analyses have revealed that the precipitates were consistent with the structure of M_6C-type carbide. The effect of combined addition of tungsten with niobium and molybdenum to the Fe-28Al-5Cr alloy has also been studied, and the highest yield strength and the longest creep rupture life have been observed on the Fe-28Al-5Cr-0.5W-0.5Nb alloy.

3:30 PM Coffee Break in Exhibit Hall

3:50 PM INVITED

SOME ASPECTS OF RAPID SOLIDIFICATION PROCESSING OF FeE-Al-X ALLOYS: *R. A. Buckley*[1]; [1]University of Sheffield, Department of Engineering Materials, Sheffield S1 3JD United Kingdom

Alloys containing between 20 and 50 at. % Al and up to 15 at. % Ti, V, Cr, Mo, or Co were prepared in ribbon form by chill-block melt spinning. The effects of these alloying elements on the A2/B2/$D0_3$ transition temperatures were determined by DTA. Microstructures of both as-spun and heat-treated ribbons were characterized using optical, TEM, SEM, and XRD. Antiphase domain sizes and morphologies are reported, and correlations between such ordering phenomena, vacancy concentrations, ternary phase precipitations, and mechanical properties (microhardnesses and bend ductilities) are discussed.

4:20 PM INVITED

HIGH-TEMPERATURE STRENGTHENING AND ROOM-TEMPERATURE DUCTILIZATION OF IRON-ALUMINIDES THROUGH ALLOY DESIGN: *Aruna Bahadur*[1]; [1]National Metallurgical Laboratory, Jamshedpur India

Ordered intermetallics possess a strong tendency for chemical ordering resulting in reduced atomic mobility and, therefore, increased resistance to plastic deformation at elevated temperatures. This intrinsic source of high-temperature strength leads to the inherent brittleness of polycrystalline-ordered intermetallics at room temperature. Both strength and ductility are important for structural applications. However, the requirements for optimum strength at elevated temperatures during operation and optimum ductility at ambient temperature during fabrication and handling are often incompatible, and a compromise has to be arrived at. The paper is an overview of the present status of iron aluminides and discusses the improvement in room-temperature ductility obtainable through alloy design and changes in stoichiometry.

4:50 PM

STRENGTHENING OF IRON ALUMINIDES BY VACANCIES AND/OR NICKEL: *Dr. Joachim H. Schneibel*[1]; [1]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

At room temperature, iron aluminides may be strengthened by quenched-in thermal vacancies as well as by solid solution alloying additions. Using nickel additions as an example, this work shows that these two strengthening contributions are not independent of each other. First, strength and density measurements show that nickel slows down the removal of vacancies, making it more difficult to obtain iron aluminides with low vacancy concentrations. Second, for low vacancy concentrations (e.g., 0.1 at. %) additions of nickel result in substantial strengthening. Third, for high vacancy concentrations (e.g., 0.3 at. %), nickel additions cause softening. These results are interpreted in terms of the interaction between the vacancies and the nickel atoms. 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.

5:10 PM INVITED

CONTRIBUTION OF DEFORMATION PROCESSED TEXTURES TOWARDS THE MECHANICAL RESPONSE OF Fe_3Al ALLOYS: *Bimal Kad*[1]; Joe Horton[2]; Vinod Sikka[2]; [1]University of California-San Diego, La Jolla, CA 92093 USA; [2]Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6115 USA

This talk will elucidate i) the dominant deformation mechanisms responsible for texture incorporation in Fe_3Al-based alloys, that are thermo mechanically processed (rolled, forged or extruded) in the working temperature range of 925-1325K, and ii) the contribution of such processing induced textures towards mechanical response within the framework of ductility and fracture. Experimental texture measurements, for samples deformed by rolling, forging and extrusion, in the 925-1325K temperature range, at deformation rates of 10-3-101 sec-1, agree well with the predicted textures for the <111>{110}+ <111>{112} slip system activation, for each of these deformation modes. Additionally, the numerical procedures are quite successful in tracking texture evolution for a combination of deformation modes over the entire 925K-1325K temperature range, provided the recrystallization effects are adequately accounted. These results appear to be in disagreement with prior transmission electron microscopy (TEM) observations, that support the activation of <100>{011} and <100>{001} slip systems. Possible sources of discrepancies are elucidated. Finally, the loss of ductility of Fe_3Al-based alloys, in fully recrystallized microstructures, is explained by textural reorganizations that are likely to impose larger normal stresses on the dominant {100} fracture planes. Sponsored by the Div. of Materials Sciences, U.S. DOE contract DE-AC05-96OR22464 with LMER and the SHaRE program under contract DE-AC05-76OR00033 with ORAU.

5:30 PM

SHEAR LIGAMENT TOUGHENING IN Fe3Al: *Scott X. Mao*[1]; [1]University of Calgary, Department of Mechanical Engineering, Calgary Canada

The environment-assisted cracking behavior of a Fe_3Al intermetallics in air moisture was studied. At room temperature, round tensile specimens were tested at different strain rates. By carefully examining the lateral surface of the tensile specimens, ligament like structure that connects between microcracks was found. SEM pictures show that this structure, which can be called shear ligament, undergoes ductile fracture by shear. This type of fracture dissipates more energy and was believed to enhance the fracture toughness of the material. By use of a micromechanical model of shear ligament toughening, fractute toughness contributed by the shear ligament was estimated at different strain rates. J integral was found to be decreased towards slower strain rate. With the knowledge of hydrogen effect on toughness of matrix material, the total fracture toughness K was predicted to be increased with increasing strain rate.

INTERNATIONAL SYMPOSIUM ON SULFIDE SMELTING '98: CURRENT AND FUTURE PRACTICES: Session IV - Copper Smelting - Impurities and Slags

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

Program Organizers: Tony Eltringham, BMP Copper, 550 California St., San Francisco, CA 94104-1020; Jussi Asteljoki, Outokumpu Oy, Lansituulentie 7, Espoo Fin-02101 Finland; Pete W.J. Chen, Phelps-Dodge Mining Co., P.O. Box 2860, Silver City, NM 88062; Osamu Ishikawa, Bechtel Corp, Mining & Metals, San Francisco, CA 94119-3965; Robert L. Stephens, Ausmelt Technology Corp., 1331 17th St., Denver, CO 80202; Courtney Young, Montana Tech, Metallurgical Engineering, Butte, MT 59701

Tuesday PM Room: 207
February 17, 1998 Location: Convention Center

Session Chair: Karina Powell, Cyprus Miami Mining Corporation, Clayton, AZ 85832

2:00 PM

CURRENT OPERATION OF KOSAKA SMELTER: Yoshihiko Maeda[1]; *Hiroshi Inoue*[1]; Yoshihiko Hoshikawa[1]; Tsuneo Shirasawa[1]; [1]Kosaka Smelter & Refinery, Kosaka Smelting & Refining Co., Ltd., Akita-ken 017-02 Japan

Kosaka Smelter is a copper smelter located in the north of Japan and has been treating complex sulfide concentrates with Flash Smelting Technology for a long time. The Flash Smelting Technology has an advantage in the point of controllability both of matte grade and the heat balance in the reaction shaft. A disadvantage of this technology, however, is that the dust generation ratio is high compared with other copper smelting technologies such as injection smelting. The reason for which the dust generation ratio is higher is that the flash smelting process is of oxidation of some of the sulfur and iron of fine sulfide concentrates with flash reaction in the oxygen-enriched air stream. In addition to the above disadvantage, the complex sulfide concentrate, which contains significant levels of volatile impurities, results in a higher dust generation ratio. This paper outlines recent improvements to the flash reactions in the furnace and the behavior of impurities at the Kosaka Smelter.

2:30 PM

PROBLEMS WITH ELIMINATION OF MAIN IMPURITIES OF THE KGHM POLSKA MIEDZ S.A. COPPER CONCENTRATES FROM THE COPPER PRODUCTION CYCLE (SHAFT FURNACE PROCESS, DIRECT BLISTER SMELTING IN A FLASH FURNACE): Józef Czernecki[1]; *Zbigniew Smieszek*[1]; Stefan Gizicki[1]; Jerzy Dobrzanski[2]; Marian Warmuz[2]; [1]Institute of Non-Ferrous Metals, GLIWICE 44-101 Poland; [2]KGHM Polska Miedz S.A. , Lubin Poland

Polish copper concentrates are characterized by high impurity contents (Pb and As). For concentrate smelting, two processes are being applied at KGHM Polska Miedz S.A., namely the shaft furnace process (Legnica Copper Smelting, Glogow I Copper Smelter) and a single-stage process for blister copper production directly from concentrate in a flash furnace (Glogow II Copper Smelter). Both technologies are used at different physical and chemical conditions which results in different behaviour of the impurities. This paper presents a technological flowsheet of the copper production in a plant using the shaft furnace and another plant in which a single-stage flash process is applied. The metals distribution in particular semi-products is given as well as a method for the impurities elimination from the copper production

cycle. Methods are also discussed for alloy and matte converting and copper refining to obtain low Pb and As concentrations in an anode copper (0.3 mass % of Pb and 1500 ppm of As Max.), which are being used in the KGHM Polska Meidz plants. Useful cooperation between both plants using different technologies, aimed at the limitations of an effect of undesired impurities on the quality of the copper produced has been described.

3:00 PM

IMPURITY DISTRIBUTION AND REMOVAL PRACTICE IN COPPER SMELTING PROCESS OF GUIXI SMELTER: *Yuan Zeping*[1]; [1]Jiangxi Copper Company, Guixi Smelter, Guixi, Jiangxi 335424 China

Guixi Smelter was commissioned in December 1985, with a design capacity of 75,000 tonnes per year of copper, based on Outokumpu-type flash furnace smelting and the oxygen enrichment converting with Peirce-Smith converters. The final capacity of the plant after the second phase expansion project will reach 200,000 tonnes per year of copper. Since commencement of operation, modifications of the plant have resulted in increasing the handling capacity of the furnace and improving the quality of products. There are, however, high contents of detrimental impurities such as arsenic, antimony and bismuth, etc. in the copper concentrates, which especially have an influence on the refining operation. In order to improve the quality of products, obtain comprehensive utilization efficiently, and also improve the operation condition and process, research work on distribution and influence of main impurities has been made in the process of pyrometallurgy. This paper describes distribution and removal practice of main impurities in the copper smelting at the Guixi Smelter.

3:30 PM Coffee Break in Exhibit Hall

3:40 PM

INTEGRATED RECYCLING OF NON-FERROUS METALS AT BOLIDEN RÖNNSKÄR SMELTER: *Theo Lehner*[1]; Vikdahl Anders[1]; [1]Boliden Mineral AB, Rönnskär Smelter, Skelleftehamn S 932 81 Sweden

The Rönnskär Smelter has wide experience recycling non-ferrous metals. Today, smelting and refining from primary and secondary raw materials are integrated in a complex flow sheet. Boliden Ltd. operates a lead flash smelter, a lead refinery, a copper smelter and refinery, a sulfuric acid and sulfur dioxide plant, a slag fuming plant and precious metal plant at its Rönnskär smelter in the north of Sweden. Raw materials include concentrates, ashes, slags, metallic scrap, electronic scrap and steelmaking dust. The paper briefly describes current operations and illustrates current measures to improve productivity. Limits to recycling as experienced by a non-ferrous smelter are illustrated. The behaviour of critical elements contained in raw material are discussed.

4:10 PM

VISCOSITY OF IRON-SILICATE SLAGS AT COPPER SMELTING CONDITIONS: *A. Vartiainen* [1]; [1]Outokumpu Research Oy, Pori FIN-28101 Finland

The viscosity of liquid Fe-O-SiO2 slag at copper smelting conditions was investigated using a rotational viscometer. The parameters studied were oxygen partial pressure (10-10-10-6 bar), composition of slag (Fe/SiO2-ratio) and temperature (1250-1400ºC). The most important factors effecting the viscosity of Fe-O-SiO2 slag were the temperature and compositions near silica and magnetite saturation.

4:40 PM

NUMERICAL HEAT AND FLUID FLOW ANALYSIS OF CONTINUOUS SLAG/MATTE SEPARATION: *Susumu Okabe*[1]; Akira Kaneda[1]; [1]Mitsubishi Materials Corporation, Central Research Institute, Saitama 330 Japan

The Mitsubishi Continuous Copper Smelting/Converting Process employs an electric furnace for settling the slag/matte mixture formed in the smelting furnace. In order to improve the separation of slag and matte and to minimize power consumption, the design and the operating conditions of the electric furnace should be optimized. As the first step, numerical heat and fluid flow analysis of the melts was carried out based on the distribution of current density and Joule's heat obtained by the electric field calculation. For these calculations, 3-D body fitted coordinate system and a finite volume method were applied. Using the developed numerical model, the flow pattern analysis by pulse input of a tracer element was simulated. It was found that a strong thermal convection of the molten slag exists around the electrodes due to the concentrated Joule's heat generation in a small area just under the electrodes, and this strongly affects the flow pattern on the furnace. The calculation results were compared with the data obtained from a plant test.

5:10 PM

EXPERIMENTAL STUDY ON THE REACTION AND DISSOLUTION BEHAVIOUR OF SILICA FLUX IN COPPER SMELTING: *K. Fagerlund*[1]; L. Palmu[1]; H. Jalkanen[1]; [1]Helsinki University of Technology, Department of Materials Science and Rock Engineering , Espoo FIN-02150 Finland

In the work reported in this paper, the rate of dissolution, melting behaviour and phase separation of silica flux with molten slag and matte were investigated. The rate of silica dissolution was measured from the decrease in the diameter of a sintered SiO2 rod rotated in iron silicate slag. Silica dissolution rate in different types of slags was determined. The reaction and separation phenomena of slightly oxidized chalcopyrite samples and cuprous sulfide-magnetite mixtures with quartz sand were examined at about 1350ºC. The samples were water-quenched and polished specimens were studied and analyzed using light optical microscope and scanning electron microscope with energy dispersive spectrometer. The silica dissolution rate in slag was found to increase with decreasing silica content and increasing ferric iron content. Silica sand reaction rate with matte was found to increase with increasing oxidation degree, increasing copper and magnetite content of matte.

5:40 PM

CONVERTING FURNACE UPGRADES AT THE KIDD CREEK METALLURGICAL COPPER SMELTER: *A. MacRae*[1]; M. Wallgren[1]; J. Lenz[2]; A. Majumdar[2]; P. Zuliani[2]; [1]Hatch Associates, Mississauga, Ontario L5K 2R7 Canada; [2]Falconbridge Limited, Kidd Creek Division, Timmins, Ontario P4N 7K1 Canada

Campaign life of the converting furnace has been limited by breakouts throughout the hearth and walls. Throughput of concentrate has increased 60% since start-up of the copper operations in 1981. The Furnace Integrity Program was initiated at Kidd in 1993 to improve the reliability and safety of the smelting-cleaning-converting furnace line. The converting furnace is undergoing a series of upgrades to overcome sever refractory wear to the roof, walls, and hearth. Improvements to the roof include increased cooling, a novel brick/cooler suspension system, and roof compression for gas tightness. Wall modifications include a new upper shell, wall coolers which can be supported during a shut-down from outside of the vessel, and an increase in the number of cooling blocks. Design is currently underway to deepen the refractory hearth to decrease hearth erosion and increase resistance to floatation.

INTERNATIONAL SYMPOSIUM ON VALUE ADDITION METALLURGY: Session IV - Thin Films and Coatings II

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee

Program Organizers: W.D. Cho, University of Utah, Dept. of Metallurgical Eng., Salt Lake City, UT 84112; H.Y. Sohn, University of Utah, Dept. of Metallurgical Eng., Salt Lake City, UT 84112

Tuesday PM Room: 208
February 17, 1998 Location: Convention Center

Session Chairs: R. Y. Lin, University of Cincinnati, Department of Materials Science & Engineering, Cincinnati, OH 45221 USA; S. Guruswamy, University of Utah, Dept of Met Engrg, Salt Lake City, UT 84112 USA

2:30 PM

REACTIVE SPUTTER DEPOSITION OF CHROMIUM NITRIDE COATINGS: *M. Pakala*[1]; Z. Fan[1]; Ray Y. Lin[1]; [1]University of Cincinnati, Department of Materials Science & Engineering, Cincinnati, OH 45221

Deposition of hard chromium coatings using electrodeposition is accompanied by emission of hexavalent chromium mists which is a carcinogenic. Hence alternative chromium deposition technologies are being investigated. Sputter deposition is a strong candidate to economically deposit hard chromium coatings in an environmentally friendly way. Chromium coatings of 2-3 micron thickness were deposited on plain carbon steel coupons using RF magnetron sputter deposition. Depositions were carried out with 0 to 22 vol% nitrogen in the plasma. Structure and composition of the coatings were studied using x-ray diffraction, microprobe analysis and x-ray photoelectron spectrometry (XPS). With increasing nitrogen content, formation of the two nitride phases is observed. The amount of the nitrogen content in coatings are obtained using microprobe analysis. Chromium phase is observed for up to 7% nitrogen in the sputtering gas. The solid solution of nitrogen in the Cr phase coating can be observed by shifting of x-ray diffract ion peak to higher d-spacings and also by XPS analysis. Measurements of the scratch test wear track widths indicate that the Cr2N coatings have better wear resistance than Cr coatings and CrN coatings. The wear resistance also increases with nitrogen content in single phase Cr coatings.

2:55 PM

EXPERIMENTAL DETERMINATION OF THE METASTABLE (TiI, Al) N PHASE DIAGRAM UP TO 700°C: *R. Cremer*[1]; M. Witthaut[1]; D. Neuschütz[1]; [1]Lehrstuhl für Theoretische Hüttenkunde, Rheinisch-Westfalische Technische Hochschule Aachen, Aachen, D-52056 Germany

Metastable (Ti,Al)N coatings with fcc structure have been shown to exhibit superior performance as compared to TiN under conditions of wear due to their better high temperature oxidation resistance and hardness. The oxidation resistance of these coatings increases significantly with increasing Al-content. It is known that the deposition of (Ti,Al)N films with Ti/Al ratios below about 30/70 leads to a hexagonal structure which is not suitable for tribological coatings. Only little is known about the location of the cubic to hexagonal phase transition and hence about the optimum composition of (Ti,Al)N hard coatings. To determine the position of this transition, $Ti_{1-x}Al_xN$ films with different Ti/Al ratios were deposited on high speed steel at substrate temperatures between 100 and 700°C by means of reactive MSIP. The composition of the films was determined by EPMA and XPS, the crystallographic structure by thin film XRD and the morphology by SEM.

3:20 PM

OXIDE, NITRIDE & COMPOUND WEB COATINGS VIA ACTIVATED REACTIVE EVAPORATION: *L. M. Bianchi*[1]; [1]FEP-Aultimut, Brooksville, FL 34613

Oxide, nitride and other compound coatings often do not have adequate properties for newer applications when produced using very high deposition rates and simple evaporation techniques. Solar control, abrasion resistance, low reflectance and barrier effective engineered coatings require exacting properties. Such properties can be obtained by adapting the activated reactive evaporation (ARE) technology to Web production processes. Adaptions of the WAD and MAD processes will be described along with data on the high deposition rates and high web speeds obtained. These fast production capabilities were developed in order to make the processes economically competitive.

3:45 PM Coffee Break in Exhibit Hall

4:00 PM

ELECTROCONDUCTIVE PIGMENTS FOR ANTISTATIC USE: *Junichi Kashiwagi*[1]; Takao Hayashi[1]; Kunio Nakahara[1]; [1]Mitsui Mining and Smelting Co., Ltd, Chemicals Div., Shimonoseki City, Yamaguchi 750 Japan

Electroconductive BaSO4 (Trademark:PASSTRAN TYPE-IV) is produced by coating ultrafine BaSO4 substrate of controlled particle size with a thin semiconductive layer of a doped microcrystalline SnO2. Electroconductive BaSO4 particles, core material of pigment (BaSO4) have low refractive index (d=1.64), can be obtained high transparency due to low scattering of light and its optical properties are nearly pure BaSO4 because of the thickness of the very thin coating electroconductive layer consisting of SnO2. Generally, by using ultra fine particles for paint, high transparency coating can be got but too small particle size causes heavy agglomeration, finally it is very difficult to dispense well. PASSTRAN is chosen good range of core size for dispersion, and easy to use for functional paint. The production of nearly transparent films, or colored coatings when combined with pigments, is possible by using PASSTRAN.

4:25 PM

SOME WAYS TO SYNTHESIZE NEW TYPES OF COMPOSITE ELECTROCOATINGS WITH ENHANCED PROPERTIES: *O. B. Girin*[1]; Yu O. Proshenko[1]; V. I. Bekarev[1]; [1]State Metallurgical Academy of Ukraine, Dept. Physics, Dnepropetrovsk 320635 Ukraine

Several approaches to electrolytic synthesis of new types of composite coatings with improved properties were devised using the authors' findings on development of texture, substructure and structural state in metal electrocrystallization. Texturally composite coatings having improved resistance to wear and corrosion, substructurally composite coatings having improved hardness and corrosion resistance, constitutionally composite coatings with phase transformations having improved corrosion resistance and reflectance, constitutionally composite coatings with structural changes having improved electric resistance, composite coatings with restricted substructure having improved resistance to abrasive-impact wear, composite coatings with modified substructure having improved hardness and strength are addressed.

MATERIALS ISSUES IN MICROELECTRONICS: INTERFACIAL REACTIONS, SOLID STATE TRANSFORMATIONS & THERMAL MANAGEMENT: INTERFACIAL REACTIONS AND THERMAL MANAGEMENT-II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee
Program Organizers: Michael R. Notis, Lehigh University, Dept of Materials Science, Bethlehem, PA 18015; Gautam Ghosh, Northwestern University, Dept. of Materials Science, Evanston, IL 60208-3108; Sung Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598; K. Linga Murty, North Carolina State University, Box 7909, Raleigh, NC 27695-7909

Tuesday PM Room: Centro D
February 17, 1998 Location: Convention Center

Session Chair: TBA

2:00 PM INVITED
COMMERCIALIZED NICKEL/PALLADIUM FINISH FOR LEADFRAMES: *Dr. Don Abbott*[1]; [1]Texas Instruments, Inc., Attleboro, MA 02703 USA

The structure of the palladium/nickel leadframe finish, which Texas Instruments introduced in 1989, and the reasons for its development, will be described. The advantages and features of this lead finish will be reviewed. There are very unique implications for the lead frame manufacturer, the assembly test site and for the end user, some of which were not anticipated at the inception of this system. There are now more than 20 billion devices in the field with palladium/nickel plated leads. A brief update will be given on current work investigating the performance of this system under various conditions.

2:30 PM INVITED
DIFFUSION AND PHASE TRANSFORMATION DURING INTERFACIAL REACTION BETWEEN LEAD-TIN SOLDER AND PALLADIUM: *Dr. G. Ghosh*[1]; [1]Northwestern University, Dept. of Mat. Sci. and Eng., Evanston, IL 60208 USA

Palladium is widely used for metallization of electronic packaging. During soldering palladium dissolves very rapidly in the liquid solder and forms various intermetallics at the solder/substrate interface. Diffusion of atomic species leads to the formation of intermetallics in the solid-state as well. In order to improve the reliability of electronic packaging, it is necessary to understand and control the diffusion and solid-state reactions between the solder and the metallization layer(s). We will discuss the diffusion and interfacial reactions between lead-tin solders and palladium. The product(s) of interfacial reaction are characterized by SEM, TEM and AEM. The microstructural evolution at the interface and the diffusion path will be discussed in terms of the calculated isothermal sections of the lead-tin-palladium ternary system at the experimental temperatures of interest.

3:00 PM
MODELING OF DIFFUSIONAL KINETICS OF THE INTERFACIAL REACTION BETWEEN LEAD-TiN SOLDER AND PALLADIUM: *Dr. Zi-Kui Liu*[1]; Dr. G. Ghosh[2]; [1]University of Wisconsin, Dept. of Mat. Sci. and Eng., Madison, WI 53706 USA; [2]Northwestern University, Dept. Mater. Sci. Eng., Evanston, IL 60208-3108

We have studied diffusion paths in the binary systems Pd-Sn and Pb-Pd and in the ternary system Pb-Sn-Pd in the temperature range of 343°C to 125°C. The concentration profiles in these diffusion couples are measured by SEM. The thermodynamic properties of the Pb-Sn-Pd system have been assessed by THERMOCALC. The DICTRA software package, which is suitable for the simulation of diffusional reactions in multicomponent alloys, has been applied. Using the assessed thermodynamic data of the Pb-Sn-Pd system, a set of kinetic parameters have been derived to describe the diffusion kinetics in the liquid and solid states. The results of modelling the diffusion kinetics in this system will be presented in detail.

3:20 PM
EFFECT OF PALLADIUM ON MECHANICAL AND FATIGUE PROPERTIES OF Sn-Pb EUTECTIC SOLDER: *Dr. Semyon Vaynman*[1]; Dr. Morris E. Fine[1]; [1]Northwestern University, Dept. Mat. Sci. & Eng., Evanston, IL 60208 USA

The effect of 0.5% of Pd on the stress-strain curve, creep and fatigue properties of eutectic Pb-Sn solder are being investigated. Creep rate of eutectic solder at room temperature is not affected by Pd addition. However, at 80°C solder containing Pd creeps more slowly. Increase in the strain rate dramatically increases the yield and tensile stresses of eutectic Pb-Sn solder with Pd as in the binary solder. Fatigue testing is in progress. Mechanisms of solder deformation and failure will be discussed.

3:40 PM Coffee Break in Exhibit Hall

3:55 PM INVITED
MICROSTRUCTURAL TRANSFORMATIONS DURING ULTRASONIC WIRE BONDING TO THIN FILM SUBSTRATES: *Prof. James E. Krzanowski*[1]; Dr. Eli Razon[1]; Mr. Andrew F. Hmiel[1]; [1]Univ. of New Hampshire, Mechanical Engineering, Durham, NH 03824 USA

The ultrasonic wire bonding process is widely used for making interconnections between IC chips and package lead frames, yet the relationships between the wire/substrate materials properties and the bond formation processes are not yet well understood. While the creation of a metallurgical bond at the interface between the wire and substrate is required, the deformation of the wire and substrate also play an important role in bond formation. Bonding to thin film substrates is of particular interest, since thin films often exhibit mechanical behavior distinctly different from bulk materials. In the present study, atomic force microscopy (AFM), SEM and TEM are used to study the nature of deformation and fracture in thin-film wire bonded substrates. The results show that the effects on the substrate can range from slight surface deformation to microcracking and shear flow. Ultimately, these results can be used to predict the wire bond reliability expected from various types of thin film metallization.

4:25 PM
MECHANICAL FATIGUE CHARACTERISTICS OF Sn-3.5Ag-X (X=Bi, Zn, Cu) SOLDER ALLOYS.: *Mr. Yoshiharu Kariya*[1]; Dr. Masahisa Otsuka[2], [1]Graduate school, Shibaura Institute of technology, Minato-ku, Tokyo 108 Japan; [2]Shibaura Institute of Technology, Department of Materials Science and Engineering, Minato-ku, Tokyo 108 Japan

The melting temperatures of the available binary tin-based lead-free solder are higher or lower than that of conventional Sn-Pb eutectic. Thus, new lead-free solders are likely to have multi-components. Furthermore, they should have an improved mechanical reliability because of recent technological developments in the electronics industry. It is, however, not clear to what extent the additional elements affect on the mechanical reliability of binary tin-based solder alloy. In this study, Sn-3.5mass%Ag eutectic solder is selected as a candidate base alloy for replacing the eutectic Sn-Pb, and the effect of additional elements (Bi, Cu and Zn) on the fatigue life of bulk Sn-3.5mass%Ag eutectic at room temperature have been studied over the total strain range from 0.3 to 3 percent (tension-tension). Fatigue life was defined as the number of cycles at which the load decreases to a half of the initial value. The fatigue life dramatically decreases with increasing contents of bismuth and adding this element over 2% makes fatigue life shorter than that of tin-lead eutectic alloy. Addition of copper up to 2% slightly decreases in the fatigue life of Sn-3.5Ag binary alloy, though the fatigue resistance still remains higher than that of in tin-lead eutectic alloy.

4:45 PM

STRUCTURE AND PROPERTIES OF ULTRAFINE-GRAINED SOLDERS: *Dr. Hareesh Mavoori*[1]; Dr. Sungho Jin[1], [1]Bell Laboratories, Lucent Technologies, Murray Hill, NJ 07974

A fine grain size is desirable in solders due to the improved fatigue resistance, strength and possible superplastic behavior. In this work, ultrafine-grained solders have been obtained through the use of nano-sized, non-reacting, non-coarsening refractory dispersions. The processing involves powder metallurgy techniques followed by plastic deformation. Ultrafine grain sizes of the order of 2000 Angstroms have been achieved. The well-known thermal instability problem with ultrafine-grained structure appears to have been overcome in these solder alloys and the microstructure was seen to be quite stable upon high temperature exposure (e.g. 120°C). The mechanical properties and microstructures of solders with different dispersed particles such as Al_2O_3 and TiO_2 are discussed and contrasted with a dispersion free solder subjected to the same processing and testing conditions. The dispersions are seen to have a significant effect on the mechanical deformation characteristics of the solders with respect to creep (2-3 orders of magnitude lower creep rate), improved ductility under high strain rate deformation, improved strength (4-5 times higher tensile strength) at low strain rates, and possibly improved fatigue properties. Possible mechanisms for deformation and creep behavior of these ultrafine-grained solders will also be discussed.

5:05 PM

WETTING INTERACTION OF Pb-FREE Sn-Zn-Al SOLDER ON PLATED SUBSTRATES: *Dr. Kwang-Lung Lin*[1]; Dr. Yu-Chien Wang[1]; [1]National Cheng Kung University, Dept. Mat. Sci. & Eng., Taiwan 701 Taiwan, ROC.

A newly developed Pb-free Sn-9(Zn-5Al) solder was investigated for its wetting behavior on metal plated Cu substrates. The Cu substrates were plated with electroless nickel (EN) or with EN/Cu plating. The wetting behavior was investigated by the wetting balance method. The solder was unable to wet the EN plated Cu substrate without the assistance of flux until at a temperature as high as 460°C. The materials interaction between the solder and the substrate at this high temperature was investigated with SEM elemental mapping. Fluxes including L-glutamic acid and dimethylammonium chloride were applied to enhance wetting behavior. The wetting temperature was lowered to 310°C with the application of these fluxes. The incorporation of a further layer of Cu plating with the EN layer further lowered the wetting temperature to 250°C. The wetting curves of these wetting interactions will be presented to discuss the different wetting behaviors that wre encountered.

MATERIALS PROCESSING FUNDAMENTALS: Session IV

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee

Program Organizers: Patrick R. Taylor, University of Idaho, Dept. of Met & Mining Eng., Moscow, ID 83843-3024; Srinath Viswanathan, Oak Ridge National Lab, PO Box 2008 Bldg. 4508, Oak Ridge, TN 37831-6083

Tuesday PM Room: 206
February 17, 1998 Location: Convention Center

Session Chair: Srinath Viswanathan, Oak Ridge National Lab, PO Box 2008 Bldg. 4508, Oak Ridge, TN

2:30 PM

REACTIVE PHASE FORMATION IN COLD ROLLED ALUMINUM-TANTALUM MULTILAYERS: *Dr. Heino Sieber*[1]; Prof. John H. Perepezko[1]; [1]University of Wisconsin - Madison, Material Science and Engineering, Madison, WI 53706 USA

Multilayer samples of Tantalum and Aluminum with the composition of Al-25Ta were prepared by cold rolling of elemental foils. The microstructure and phases were characterized by XRD, SEM and TEM/SAED and the reactive phase formation sequence was examined by DSC and DTA measurements in multilayer foils rolled for different deformation levels. The rolling procedure results only in a decrease of the layer thickness of the elemental foils and in the decrease of the individual grains size of both elements. While XRD and DSC measurements show no phase formation up to the melting of aluminum for samples rolled up to 40 passes, in samples rolled for 50 or more passes a first phase formation reaction (TaAl3) starts at around 340°C. The DSC scans show the phase formation as a double exothermic peak, related to a 2-dimensional and a 3-dimensional phase growth, that is known for the formation of the first phase in different multilayer systems and also in cold rolled Ni-Al samples. The multilayer microstructure and reaction product morphology are characterized by detailed SEM and TEM investigations in plan view and cross section geometry. The support of ONR (N00014-92-J-1554) is gratefully acknowledged.

2:55 PM

HYDROGEN MICROPRINT TECHNIQUE APPLIED TO OBSERVE IMPURITY HYDROGEN IN ALUMINUM DURING DEFORMATION: *Mr. Akihide Nagao*[1]; Dr. Shigeru Kuramoto[2]; Dr. Motohiro Kanno[2]; [1]Graduate School, The University of Tokyo, School of Engineering, Dept of Materials Science, Bunkyo-ku, Tokyo 113 Japan; [2]The University of Tokyo, School of Engineering, Dept of Materials Science, Bunkyo-ku, Tokyo 113 Japan

The behavior of impurity hydrogen that is contained in the starting material or picked up during processing has been little investigated up to now. Hydrogen microprint technique is applied to investigate microscopic location of impurity hydrogen during deformation in aluminum without hydrogen charging. Three kinds of pure aluminum of 99.99 percent are prepared, in which the content of hydrogen is varied by changing the atmospheres when they are melted and heat-treated. After rolling, tensile specimens with a gage size of 4x4mm and 0.5mm in thickness are stamped out from these three kinds of aluminum sheets. These specimens are covered with liquid nuclear emulsion of silver bromide crystals, stretched by 5,10,15 percent plastic strain or fractured, developed and fixed. The SEM micrograph reveals a number of silver particles on the slip lines and the grain boundaries. Furthermore, it is revealed that the more hydrogen exists in the specimen, the more silver grains appear on the slip lines and the grain boundaries. Consequently, it is concluded that impurity hydrogen is transported to the surface with gliding dislocations during deformation.

3:20 PM

THERMODYNAMICS OF PHOSPHORUS IN MOLTEN Si AND Si BASED ALLOYS.: *Mr. Takahiro Miki*[1]; Mr. Shigeru Ueda[1]; Dr. Kazuki Morita[1]; Dr. Nobuo Sano[1]; [1]University of Tokyo, Department of Metallurgy, Tokyo 113 Japan

Silicon, ferrosilicon and silicomanganese are widely used as deoxidizers in steel refining process. The phosphorus introduced during a deoxidation process can be hardly removed and apt to be brought into final products, giving unfavorable effects on steel properties. On the other hand, silicon is used for solar cell which convert solar energy into electricity. Phosphorus is a typical impurity in silicon which must be removed because phosphorus is a n-type dopant. Therefore, thermodynamics of phosphorus in such metals is important for the evaluation of dephosphorization processes. In the present study, thermodynamic properties of phosphorus in molten silicon at 1723-1848K and those of Si-Fe and Si-Mn alloys at 1723K have been was determined by equilibrating respective metals in a controlled phosphorus partial pressure. The results are expressed as follows: $1/2P_2(g)=P$(mass pct, in Si) $\Delta G^0 = -139000+43.4T$(J/mol) $\varepsilon_P^{Fe}=7.43$, $\rho_P^{Fe}=-16.4$ ($X_{Fe}<0.65$) $\varepsilon_P^{Mn}=12.0$, $\rho_P^{Mn}=-22.2$ ($X_{Mn}<0.5$)

3:45 PM

EFFECT OF ZINC ADDITION AND MELTING ATMOSPHERE ON THE INTERMEDIATE TEMPERATURE EMBRITTLEMENT OF PURE COPPER: *Dr. Shigeru Kuramoto*[1]; Mr. Yoshiyuki Nakamura[2]; Dr. Motohiro Kanno[1]; [1]The University of Tokyo, School of Engineering, Dept of Materials Science, Bunkyo-ku, Tokyo 113 Japan; [2]Graduate School, The University of Tokyo, School of Engineering, Dept of Materials Science, Bunkyo-ku, Tokyo 113 Japan

It has been known that pure copper exhibits intermediate temperature embrittlement when it is deformed at slow strain rate, and that small addition of zinc suppresses this embrittlement. In the present study, the relation between zinc addition and hydrogen behavior has been taken into account since the impurity hydrogen is considered to deteriorate the ductility at intermediate temperatures. A pure copper and Cu-Zn alloys containing 1-5mass% zinc are prepared by melting in argon and in vacuum, so that a comparison can be made between the specimens with normal hydrogen content and the ones with less hydrogen content. Tensile tests are performed at temperatures ranging from 293 to 773 K, and hydrogen behavior is analyzed by use of a newly developed testing machine that can detect gas species evolved from the specimens during deformation and fracture in ultra high vacuum.

4:10 PM

EVALUATION OF NON-EQUILIBRIUM MINOR COMPONENTS IN PYROMETALLURGY: *Dr. Akira Yazawa*[1]; Shigeatsu Nakazawa[2]; [1]Tohoku University, Dept of Met, Graduate Sch of Engrg, Aoba-ku, Sendai 981 Japan

In the field of pyrometallurgy, the equilibrium evaluation has recently become easy by use of thermodynamic package, but it is strongly desirable to estimate the amounts of non-equilibrium minor components to simulate practical processes. To fulfill this request, the activity coefficient is modified and introduced as the simulation parameter under the name of "adjustment coefficient", because any simple physicochemical technique can not be applied to evaluate the non-equilibrium components which are formed by many reasons. By use of this modified thermodynamic calculations, various processes of sulfide smelting are simulated. The loss of copper in slag is caused by equilibrium and non-equilibrium reasons, that is, chemical dissolution and entrainment in slag. The total copper content of slag is simulated by use of the adjustment coefficient. The simulation of the roasting process is more difficult than that of smelting process because so many solid phases are participating and the system is not necessarily in equilibrium. Successful simulation of roasting is realized by assignment of suitable adjustment coefficients for minor constituents which form a pseudo solid solution in the calcine. Some examples from sulfation roasting and dcarsenizing roasting are discussed.

4:15 PM

PHENOMENON OF SUBSTRANCE SELF-ORGANIZING IN A DIFFUSION LAYER DURING ELECTROSPARK ALLOYING: *N. E. Ablesimov*[1]; A. D. Varkhoturiv[1]; S. B. Pyachin[1]; [1]Institute of Materials, Khabarovsk 680042 Russia

Electron probe x-ray microanalyser of a coating deposited with +Elitron-22A+electrospark alloying (ESA) set (anode-Cr, base-catode-chrome vanadium steel) has shown that the zone of phase forming can be divided into three parts: the coating itself, diffusion zone and a thermal affection zone in the base material. Waves of iron and chrome concentrations have been found in the diffusion zone. The authors interpret them as a phenomenon of substance self-organizing in the 30 mm deep diffusion zone, characterized by a change of compositions during the transfer of material from the coating to the base: coating - 20 mm (FeCr2), diffusion zone 30 mm (Fe3Cr, FeCr, Fe4Cr, Fe2Cr), base steel (Fe-87%, Cr-12%, V-1%). This ESA phenomen has been noted for the first time. It may be relative to the phenomenon of periodic formation of secondary products during the mutual diffusion of reacting substances in the case of a one-way reaction. Similar regular structures may appear in the diffusion zone during ESA of vanadium steels with vanadium, manganese steels with manganese and so on. It may be relative to the phenomenon of periodic formation of secondary products during the mutual diffusion of reacting substances in the case of a one-way reaction. Similar regular structures may appear in the diffusion zone during ESA of vanadium steels with vanadium, manganese steels with manganese and so on.

4:40 PM

HEAT EFFECTS IN BIOLEACH SYSTEM: *Ignacy A. Lipiec*[1]; Renato G. Bautista[2]; [1]B.Y.G. Natural Resources, Inc., Whitehorse, Yukon Territory Y1A 2T9 Canada

The advantage of micro-organisms in bioleach systems over the conventional chemical reaction is that they apply the available energy more precisely where it is needed either directly onto the mineral of interest or by efficiently catalyzing the production of a reactant. The literature cover the range from empirical models to describing the macro heat balance in heaps to detailed studies relating the micro heat effects of bacteria adhesion energy to the surface of minerals. In this paper, the heat effects is reviewed in a general way describing bioleaching in the two main macro systems, heap leaching and slurry bioreactors. The micro scale heat effects is the reactions and conditions at the micro-organism scale, including the mechanism of leaching and bacterial behavior.

MICROSTRUCTOLOGY CONNECTING PHASE DIAGRAMS KINETICS & STEREOLOGY TO MICROSTRUCTURAL EVOLUTIONS: F.N. RHINES: Microstructural Evolution

Sponsored by: ASM International: Materials Science Critical Technology Sector, Atomic Transport Committee
Program Organizers: Robert T. DeHoff, University of Florida, Dept. of Materials Sci & Eng., Gainesville, FL 32611-6400; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Tuesday PM Room: 103
February 17, 1998 Location: Convention Center

Session Chair: Mark E. Schlesinger, Rolla, MO 65409-0340

2:00 PM INVITED

MICROSTRUCTURAL EVOLUTION IN MULTICOMPONENT SYSTEMS: *Dr. Gary Purdy*[1]; [1]McMaster University, Department of Materials Science and Engineering, Hamilton, Ontario Canada

Following the pioneering work of Professor Rhines, the materials community has witnessed the development of new methods of treating diffusion and phase transformation in multicomponent systems. This presentation will deal with the ways in which the thermodynamics of these systems influences their diffusion and transformation behaviour. Starting with simple two-component models for phase transformations, it is noted that the introduction of third component brings with it a new dimensionality and consequently, a rich set of new phenomena. The examples considered include coherent diffusion, alloy solidification, particle coarsening and the growth of a new phase from solid solution.

2:30 PM

MODELING MICROSTRUCTURAL EVOLUTIONS IN ELECTRICAL INTERCONNECTIONS: *Dr. Jorma Kivilahti*[1]; [1]Helsinki University of Technology, The Microelectronics Centre, Otakaari 5A, 02015 Espoo Finland

Improving the reliability of electronic and optical components and devices being packaged and assembled either by soldering or with some other interconnection technique a better understanding of microstructural evolutions in ever-smaller interconnections is of great practical interest. For this purpose a special concept of the "local nominal composition" (LNC) in the effective joint region has been introduced.

Essentially, it combines the thermodynamic and diffusion kinetic approaches and can be used for predicting the microstructural evolutions in microjoints and in thin material layers. This approach together with careful microstructural studies is shown to provide an viable tool for developing new interconnection materials and processes. As specific examples the evolution of microstructures in Pb-free solder systems and in the Cu/SnPb and Ni/SnPb interconnection systems will be examined.

2:50 PM

METALLOGRAPHY, METEORITES, AND THE Fe-Ni PHASE DIAGRAM: *Dr. Phyllis Z. Budka*[1]; Dr. J.R.M. Viertl[2]; [1]General Electric, Senior Engineer, Schenectady, NY 12309 USA; [2], Schenectady, NY 12309 USA

The nickel-iron meteorite Widmanstätten structure, a metallographic feature visible to the naked eye, was used to build the first iron-nickel phase diagram in 1904. Today, this Widmanstätten structure is used for calculations of "metallographic cooling rates," currently on the scale of a few centigrade degrees per million years, that is the foundation for models of small asteroidal body formation. This approach relies on the 1904 assumption that the Widmänstatten structure is the product of an equilibrium solid state phase transformation: gamma austenite transforms to alpha ferrite. Modern researchers have demonstrated that the thermal history of body-centered cubic low nickel-iron (alpha or delta) cannot be determined by metallographic techniques. Therefore, a re-examination of the initmate connection between the Fe-Ni phase diagram, meteoritic Widmanstätten structure and metallographic cooling rates is warranted.

3:10 PM

WIDMANSTATTEN FORMATION IN IRON METEORITES: *Dr. Joseph I. Goldstein*[1]; [1]University of Massachusetts, College of Engineering, Amherst, MA 01003 USA

The Widmanstatten pattern was first observed in an iron meteorite by von Widmanstatten of the Natural History Museum of Vienna more than a century ago. The Widmanstatten pattern has been seen in all types of materials and is commonly used to describe any structure showing a well defined crystallographic relationship between two phases. The Widmanstatten pattern in iron meteorites formed by the nucleation of bcc Fe-Ni on the close packed planes of the parent fcc Fe-Ni phase. The growth of the bcc phase is controlled by the diffusion of Ni in the Fe-Ni-P ternary phase diagram. The same bcc Fe-Ni phase is also observed to grow in the metal of stony-iron and stony meteorites. This paper will show experimental results on the solid state transformation for the formation of the Widmanstatten pattern. In addition the growth of the Widmanstatten will be described using a model involving ternary diffusion in the Fe-Ni-P system as a function of cooling rate. It will also be demonstrated that the formation of the Widmanstatten pattern cannot be formed directly by solidification of bcc Fe-Ni from the melt.

3:30 PM Coffee Break in Exhibit Hall

3:40 PM INVITED

COMPUTER SIMULATION OF MICROSTRUCTURAL EVOLUTION AND COARSENING KINETICS OF ORDERED INTERMETALLIC PRECIPITATES IN CUBIC ALLOYS: *Dr. Long-Qing Chen*[1]; Dr. D. Li[1]; Dr. R. Poduri[1]; [1]Pennsylvania State University, Department of Materials Science and Engineering, University Park, PA 16802 USA

Many important alloy systems such as Ni-based superalloys and Al-Li alloys contain ordered intermetallic precipitates. The precipitate morphology and coarsening kinetics depend on many factors such as volume fraction, temperature and lattice mismatch between precipitates and matrix. Despite decades of extensive research, existing theories still fail to predict the particle size distributions and the volume fraction dependence of the kinetic rate constants in the cubic-growth law of the average precipitate size. This presentation will discuss our initial attempts to numerically simulate the microstructural evolution and coarsening kinetics of ordered intermetallic precipitates using microscopic diffusion equations and the continuum diffuse-interface phase-field model. In these models, the coalescence or encounters among precipitates which belong to one of the several degenerate antiphase domains are automatically taken into account. The dependencies of two-phase morphology, the particle size distribution, the structural function, and the corresponding coarsening kinetics on the volume fraction and the lattice mismatch were systematically studied. The results will be compared with existing theories and experimental measurements. The effect of applied stress on the two-phase microstructures will be discussed. This work is supported by the Office of Naval Research.

4:10 PM

MICROSTRUCTURAL DEVELOPMENT DURING PROCESSING OF ZIRCONIUM SILICIDE COATINGS: *Dr. Charles A. Odegard*[1]; Mr. Feng-Lei Liu[1]; Dr. Arturo Bronson[1]; [1]University of Texas at El Paso, Matl. Cntr. Synth. & Proc., El Paso, TX 79968 USA

Silicides have been studied as potential oxidation barrier coatings for composites for use at ultrahigh temperatures (>1600°C). In this study, the microstructural development was investigated for samples processed isothermally and in a thermal gradient. For the isothermal experiments, oxidation of the silicide coating is controlled by a silica layer, through which oxygen diffuses, and a three-phase field, which fixes the oxygen potential. The coexisting three phases consisted of either SiO_2 -ZrSi-ZrO_2 or $ZrSi_2$-ZrO_2-$(ZrSi)_L$, for which the latter consisted of a liquid silicide $(ZrSi)_L$. The zirconium within the liquid silicide preferentially oxidizes because ZrO_2 is the most stable oxide of the Zr-Si-O system. At temperatures from 1600 to 2000°C, $(Zr)_L$ oxidation depletes the silicide liquid of zirconium and causes formation of the Si-$ZrSi_2$ eutectic structure upon solidification. At 2000°C, the solid ZrSi phase was found adjacent to the ZrO_2 and SiO_2 phases. For samples processed in a thermal gradient, phase segregation of Si was observed. High temperature mechanisms that can contribute to phase formation and segregation in a thermal gradient will be discussed, including oxidation, thermotransport, and solidification.

4:30 PM

NONEQUILIBRIUM PHASE TRANSFORMATIONS IN Ti-(30-60)Al: *Dr. Young-Won Kim*[1]; [1]Wright Laboratory Materials Directorate, WPAFB, OH 45433 USA

Solid-state phase transformations taking place in the Ti-(25-55) at% Al system during cooling from the high temperature phase fields (beta- or alpha-Ti), were investigated by conducting controlled heating/cooling experiments, and by analyzing thermal response (DTA), phase relation (X-Ray and BSEI), orientation relationship (X-ray/TEM), and microstructural evolution (TEM/BSEI/OM). Solution treatments of the alloys in the a-Ti phase field, followed by cooling at rates below 30C/sec, resulted in three types of transformations, depending upon composition and/or cooling rate, that is, Type I (aFa2); Type II (aFa2Fa'2+g); and Type III (aFa'+gFa'2+g). For a given cooling rate, the transition from Type I through III took place as Al content increases. Both II and III reactions resulted in the lamellar structure consisting of crystallographically aligned a2-Ti3Al and g-TiAl laths. The mechanisms, nucleation processes and growth kinetics involved in these reactions are analyzed using the enthalpy changes and TEM analysis. The ordering enthalpy in Type I and II reactions was composition-independent. The ordering in Type III, a'Fa'2, occurred at increasingly higher temperatures with increasing Al content, and this is explained in terms of compositional changes and dynamic constraint effect.

4:50 PM

THE FCC/HCP PHASE EQUILIBRIA AND PHASE TRANSFORMATION IN COBALT BASED BINARY SYSTEMS: *Dr. Ji-Cheng Zhao*[1]; [1]General Electric Corporate Research and Development, Schenectady, NY 12301 USA

The FCC * HCP phase transformation in cobalt based binary systems has been studied extensively by monitoring the cooling and heating transformation-start temperatures. The experimental data on the related phase boundaries are widely scattered. This wide scattering is due to the fact that the FCC * HCP transformation is very sluggish and that there are more than one kind of transformations taking place. Most existing versions of the related Co-X phase diagrams do not have a good assessment of these phase boundaries. This work will present a

way to assess the related phase equilibria. The results on several Co-X (X = Al, C, Cr, Fe, Ge, Mn, Mo, Ni, Pt, Si, Sn, Ta, Ti, V, W) binary systems will be presented.

5:10 PM

MORPHOLOGICAL DEVELOPMENT OF THE MICROSTRUCTURE IN THREE PHASE W-Ni-Fe ALLOYS: *C. G. Mukira*[1]; T. H. Courtney[2]; [1]GE Research and Development Center, Schenectady, NY 12301; [2]Michigan Tech University, Department of Metallurgy, Houghton, MI 49931

We have studied the microstructural development of a wide range of W-Ni-Fe compositions, generated through mechanical alloying of elemental powders. A three-phase (W, NiFeW intermetallic, and fcc Ni-rich phase) fine structure, precipitates during the high temperature consolidation. The initially randomly distributed microstructure, characteristic of the as-consolidated alloys, rearranges into microconstituents (that have distinct phase volume fractions in them) upon extended isothermal treatment. At earlier heat treatment times, these microconstituents undergo a rapid increase in size, followed by a period when the structures undergo minor changes. We show that this morphological development is a manifestation of surface energy reduction phenomena, and describe the factors that controls the growth of these microconstituents. Experimental results evidencing microconstituent development are presented. This work was supported by the Army Research Office.

MICROSTRUCTURE AND ITS EFFECTS ON AMORPHOUS NANOPHASE & NANOCRYSTALLINE MATERIALS: Session IV - Coating and Layered Structures

Sponsored by: ASM International: Materials Science Critical Technology Sector, Flow & Fracture, Jt. Electronic, Magnetic & Photonic Materials Division/Structural Materials Division, Alloy Phases Committee, Chemistry & Physics of Materials Committee, Structural Materials Division, Physical Metallurgy Committee

Program Organizers: Ram B. Bhagat, Pennsylvania State University, 227 Hammond, University Park, PA 16802; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899; Stephen Spooner, Oak Ridge National Lab, Solid State Division, Oak Ridge, TN 37831

Tuesday PM Room: 205
February 17, 1998 Location: Convention Center

Session Chairs: Bernard H. Kear, Rutgers University, Materials Science and Technology Department, Piscataway, NJ 08854; Marie-Isabelle Baraton, LMCTS-CNRS ESA 6015, Faculty of Sciences, Limoges F-87060 France

2:00 PM Opening Remarks

2:10 PM INVITED

THE MECHANICAL AND TRIBOLOGICAL PROPERTIES OF NANOCRYSTALLINE AND NANOLAMINATED SURFACE COATINGS: *Derek O. Northwood*[1]; Ahmet T. Alpas[1]; [1]University of Windsor, Mechanical and Materials Engineering, Windsor, Ontario N9B 3P4 Canada; [1]Ryerson Polytechnic University, Faculty of Engineering and Applied Science, Toronto, Ontario M5B 2K3 Canada

The mechanical and tribological (friction/wear) properties were determined for nanocrystalline aluminum and nanolaminated Al/Al2O3,Ti/TiN and Ti/Cu composite films. The aluminum grain size varied between 15 and 10^6nm. The Al and Ti layer thicknesses in 2O3 and Ti/TiN composites ranged from 70 to 500 nm and from 150 to 450 nm, respectively. Within the grain size range of 15-100 nm, the hardness of the aluminum follows a Hall-Fetch type relationship. The hardness of the Ti/TiN and Al/Al2O3 films also follows a Hall-Petch relationship with the Ti and Al layer thickness. The Ti/Cu composites show a softening effect with decreasing Ti layer thickness. The coefficient of friction and wear rate for nanolaminated Ti/TiN and Al/Al2O3 compositives are consistently reduced as the metal layer thickness is reduced. The micromechanisms responsible for the differences in mechanical and tribological properties are examined and the results compared with published literature.

2:50 PM

METHODOLOGIES FOR CONSOLIDATION OF NANOSTRUCTURED CERAMICS: *Ganesh Skandan*[1]; Yijia Chen[2]; N. Glumac[2]; F. Cosandey[2]; S. C. Liao[2]; William E. Mayo[2]; Bernard H. Kear[2]; [1]Nanopowder Enterprises Inc., an SMT Company, Piscataway, NJ 08854-3908; [2]Rutgers - The State University of New Jersey, Piscataway, NJ 08855-0909

Significant advances have been made recently in the synthesis of oxide ceramic nanopowders. Non-agglomerated nanoparticles such as SiO2, TiO2, SnO2, and Al203 in the 3 - 50 nm size range, and having a narrow particle size distribution, are now routinely produced by Combustion Flame - Chemical Vapor Condensation (CVC). Presently, consolidating the nanoparticles into large samples that are of commercial importance, and processing the nanoparticles into useful coatings, are two of the major unresolved issues. We have made significant progress in sintering nanoparticles in a proprietary high pressure apparatus at relatively low temperatures. For example, starting from g-Al203 nanoparticles, a density in excess of 93% was achieved by sintering at 600°C with an applied pressure of 5.5 GPa. The conversion to the a-phase was complete. We have also used the Combustion Flame to directly deposit nanoparticles to form adheres coatings on a variety of substrates at low temperatures. For example, an adherent coating of Sn O2, 3 mm in thickness, was deposited on an oxidized silicon substrate at < 400°C at a deposition rate of 0.5 mm/mint The high pressure consolidation technique and the in situ high rate deposition process will be described along with the characterization data on the processed materials.

3:10 PM

SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE Ni, INCONEL 718, AND STAINLESS STEEL COATINGS: *Ms. M. L. Lau*[1]; Dr. H. G. Jiang[1]; Dr. E. J. Lavernia[1]; [1]University of California, Irvine, Department of Chemical Engineering and Materials Science, Irvine, CA 92697-2575 USA

The present paper describes the synthesis and characterization of nanocrystalline Ni, Inconel 718, and stainless steel coatings. The feedstock powders were prepared by mechanical milling which micron-sized powders were milled in either methanol or liquid nitrogen environment to produce flake-shaped agglomerates with the average grain size of less than 100 nm. The powders were introduced into the HVOF process to produce nanocrystalline coatings. The fuel gas to oxygen ratio was varied to study how oxidation mechanism affects the microstructure and properties of the coatings. X-ray diffraction analysis and transmission electron microscopy were used to determine the average grain size of the milled powders. Scanning electron microscopy was used to analyze the particle morphology as well as the microstructure of the coatings. In addition, coating properties of various materials were characterized by hardness measurements. The hardness of the nanocrystalline coatings is compared to those of the micron-sized coatings.

3:30 PM

INTERFACIAL STABILIZATION OF FCC Ti IN Ti/Al MULTILAYERS: *R. Banerjee*[1]; X. D. Zhang[1]; M. J. Mills[1]; S. A. Dregia[1]; H. L. Fraser[1]; [1]The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210

Sputter deposited Ti/Al multilayers with varying bilayer periodicities (1) and volume fractions of Ti (fTi) have been studied by high resolution electron microscopy (HREM). These multilayers exhibit a number of interesting polymorphic transitions. Ti changes from its bulk stable

hcp structure to metastable fcc and Al transforms from fcc to hcp at small layer thicknesses (3/4 5 nm). The effect of 1 and fTi on the structural transitions will be discussed. The formation of fcc Ti and hcp Al in Ti/Al multilayers can be explained on the basis of a classical thermodynamic model of phase transformations involving the competition between bulk and interfacial energies. Based on the experimental results the energy of interfacial stabilization has been calculated for hcp}Efcc transformation in Ti. There is a mismatch of~ 8 % between the lattice parameters of fcc Ti and fcc Al. The structure of the fcc Ti / fcc Al interface has been investigated by HREM. It is a semi-coherent interface with a periodic array of misfit 1/2 <110> type dislocations which have split into partials. The details of the interfacial structure and their implications on the stabilization of fcc Ti will be discussed in this paper.

3:50 PM

SYNTHESIS, PROCESSING AND MICROSTRUCTURE OF ALUMINUM BORATE-M (M=Co, Ni, AND Cu) NANOCOMPOSITES: *Jonathan D. Stolk*[1]; Arumugam Manthiram[1]; [1]The University of Texas at Austin, Center for Materials Science and Engineering, Austin, TX 78712 USA

Metal matrix composites reinforced with aluminum borate whiskers have drawn considerable interest recently. The aluminum borate whiskers exhibit mechanical properties comparable to silicon carbide whiskers, but at a much lower cost than SiC. This study involves the synthesis of nanocomposites consisting of aluminum borate and transition metals such as Co, Ni and Cu by a simultaneous hydrolysis of aluminum butoxide and reduction of metal chloride solutions with sodium borohydride. The as-prepared samples are heat treated in a reducing atmosphere at temperatures from 800 to 1200ºC to investigate the temperature- and time-dependence of the growth of metal particles and aluminum borate whiskers. Formation of a needle-like aluminum borate phase occurs at temperatures around 900ºC, and the length and aspect ratio of the $Al_{18}B_4O_{33}$ whiskers increase with temperature and time. The nanocomposite powders are characterized by X-ray powder diffraction, scanning and transmission electron microscopies, and microhardness measurements.

4:10 PM

CORRELATION BETWEEN PARTICLE VELOCITY AND TEMPERATURE PROFILE OF NANOCRYSTALLINE COATINGS BY HIGH VELOCITY OXY-FUEL SPRAYING: *Ms. Maggy L. Lau*[1]; Dr. Hong G. Jiang[1]; Dr. Enrique J. Lavernia[1]; [1]University of California, Irvine, Dept. of Chemical Engineering and Materials Science, Irvine, CA 92697-2575 USA

The present paper describes a mathematical model predicting the particle The present paper describes a mathematical model predicting the particle behavior of nanocrystalline coating during high velocity oxy-fuel (HVOF) thermal spray. The feedstock powders were synthesized by methanol and cryogenic milling to produce flake-shaped agglomerates with an average grain size of less than 100 nm. Different milling times were used to produce agglomerates with various aspect ratios. The powders were then introduced into the HVOF thermal spray to produce nanocrystalline coatings. Scanning electron microscopy and transmission electron microscopy were used to study the morphology of the nanometric particles and the sprayed coatings. The equations describing the momentum and energy transfer coupling with a modified aspect ratio were used to account for the size and morphological effects of the nanometric agglomerates. Changes in particle characteristics due to the morphology of the agglomerates will be discussed.

4:30 PM

PHONON LOCALIZATION AND RAMAN SCATTERING IN NANOCRYSTALLINE SILICON: A CRITICAL EXPERIMENTAL REVIEW OF VALIDITY OF THEORETICAL MODELS: *Stan Veprek*[1]; Ch. Ossadnik[1]; I. Gregora[2]; [1]Technical University Munich, Institute for Chemistry of Inorganic Materials, Garching/Munich S-85747 Germany; [2]Czech Academy of Sciences, Institute of Physics, Czech Republic CZ-18040

Already in 1980 our group reported on the frequency shift of Raman spectra in nc-Si [1] and attributed it to the phonon localization within the quasi-isolated crystallites [1,2]. This work was followed by a number of experimental and theoretical studies, and Raman scattering is nowadays being used as a standard technique for the determination of crystallite size, its distribution and the fraction of the crystalline Si in films consisting of a mixture of nc-Si and a-Si. However, already in our early [2] and later [3] work we have shown and emphasized that the Raman shift and the normalized scattering intensity from nc-Si depends not only on the crystallite size but also on mechanical stress in the material which may even dominate the observed phenomena. In the present paper we shall briefly summarize these results and present new, systematic study of the effect of the crystallite size and mechanical stress on the phenomena observed in Raman scattering. These results will clearly show that Raman scattering alone cannot provide any unambiguous information regarding the crystallite size, its distribution and crystalline fraction unless additional information regarding the structure of the films are obtained by other techniques. These results will also show the limits of the accuracy of such measurements. [1] Z. Iqbal et al., Appl. Phys. Lett. 36(1980)163; J. Phys. C 14(1981)295; Solid St. Commun. 37(1981)993. [2] Z. Iqbal and S. Veprek, J. Phys. C 15(1982)377; Sarott et al. Solid St. Commun. 42(1982) 465[3] S. Veprek et al., Phys. Rev. B 36(1987)3344

4:40 PM General Discussion Moderator: Ram B. Bhagat

TUESDAY PM

MODELING THE MECHANICAL RESPONSE OF STRUCTURAL MATERIALS: Session II: Constitutive Modeling

Sponsored by: Structural Materials Division, Structural Materials Committee

Program Organizers: Eric M. Taleff, The University of Texas, ASE/EM CO600, Austin, TX 78712; Rao Mahidhara, Cypress Semiconductor Corp, San Jose, CA 95134

Tuesday PM Room: 202
February 17, 1998 Location: Convention Center

Session Chairs: U. F. Kocks, Univ of Texas, Los Alamos, NM 87545; C. Persad, The University of Texas at Austin, Institute for Advanced Technology, Austin TX 20744

2:00 PM INVITED

MECHANICAL BEHAVIOR OF Ta AND Ta-W ALLOYS: C. L. Briant[1]; C. Bull[1]; *D. Lassila*[2]; [1]Brown University , Division of Engineering, Providence, RI 02912 USA; [2]Lawrence Livermore National Labs, Livermore, California 94551

This paper reports a study of the microstructural evolution that occurs in polycrystalline Ta and Ta-W alloys during mechanical testing. The materials were first annealed and then tested in compression at strain rates between 10-4 and 104 s-1 at temperatures between 77K and 573K. One set of materials was also shock loaded. The results show that in pure Ta increasing the strain rate or lowering the temperature causes cell diameters to decrease, the area of material that contains cells relative to the area that contains general tangles of dislocations to decrease, and the dislocation density to increase. Increasing the tungsten content of the alloy had a similar effect. Shock loading introduced new features into the microstructure, some of which were identified as twins. These results will be discussed in terms of models that incorporate these experimental variables.

2:30 PM

MICROPLASTIC CONNECTIVITY AND MACROPLASTICITY: *P. Matic*[1]; A. Kee[2]; [1]Naval Research Laboratory, Fracture Mechanics Section - Code 6382, Washington, DC 20375 USA; [2]Geo-Centers, Inc., Fort Washington, MD 20744 USA

Stress and strain path dependence of macroscopic bulk metal plasticity is well known. The origins of macroscopic plasticity, however, lie in the microplastic deformation fields generated by actual microstructures. An understanding of the transition from isolated microplasticity to macroplasticity, however, requires an assessment of how microplastic zone connectivity (or percolation) develops across complex microstructural features. In principle, for an individual set of experiment or simulation data, this is very straightforward to do visually from full field deformation fields. In practice, when many cases may be of interest and many features are present in each case, automation of the plastic connectivity assessment by software allows for a more complete and rapid analysis. In this investigation, the evolution of microplasticity across microstructures are evaluated. Finite element simulations were conducted to establish databases which could be analytically assessed for plastic connectivity. These simulations were based on images of actual microstructural morphologies such as those of porous metals. These databases were analyzed using software employing graph theory methods to automate the connectivity evaluation. The results were used to track the onset of microyield in the microstructure and the resulting microplastic patterns. The microplastic connectivity was used to generate estimates of the bulk yield surfaces for anisotropic microstructures.

2:50 PM

ATOMIC AND GRAIN LEVEL MODELING OF POLYCRYSTAL DEFORMATION: P. Dang[1]; *N. Chandra*[1]; [1]Florida A&M University, Florida State University, Department of Mechanical Engineering, FAMU-FSU College of Engineering, Tallahassee, FL 32310 USA

Modeling of superplastic deformation mechanisms is carried out in two different scales of atoms and grains. Computational methods at each level are introduced. In the atomistic simulation, interatomic potentials using Embedded Atom Method (EAM) are used in conjunction with molecular statics and dynamic calculations. Atomistic simulations are performed on a series of grain boundary structures in aluminum, and the energies associated with each of their equilibrium configurations are computed. The equilibrium grain boundary structures are obtained using energy minimization technique. The temperature and applied stress effects on grain boundary sliding (GBS) and migration are analyzed in details by using molecular dynamics calculations. In the grain level modeling, a micromechanical model is developed from the constituent grain level to the level of polycrystalline bulk materials, to study the stress-strain rate relations. Using this approach, the influence of temperature and grain size on the high-temperature deformation behavior of superplastic materials are predicted over a wide range of strain rates

3:10 PM

FINITE ELEMENT SIMULATIONS OF HIGH RATE ELECTROMECHANICAL TESTS OF Al 6061-T6 TENSILE COUPONS : A. Yeoh[1]; *C. Persad*[1]; [1]The University of Texas at Austin, Institute for Advanced Technology, Austin, TX 78759 USA

This study describes the results of a new technique for subjecting tensile test coupons to pulsed electromagnetic loads. An experimental arrangement that is symmetrical about the center plane of an electromagnetic launcher was used. A pair of identical test coupons was subjected to multiple current pulses until tensile failure of the coupons occurred. This experimental arrangement permitted tension specimens to be subjected to pulsed electromagnetic forces and high strain rates. The purpose of the initial tests was to study failure mechanisms in Al 6061-T6 when stressed under more realistic loading conditions. In parallel with the experiments, finite element simulations using Hsieh's EMAP3D code under identical current excitation were performed. The goal was to visualize temperature gradients and "hot spots" on the specimen. Thermocouples attached to the surface of the specimen provided a basis to compare the simulation and the experimental results. Post-test metallurgical analyses of the failed specimens were performed to characterize the mode of failure. These results show that finite element simulations can be employed as a predictive tool to map thermal contours and predict failure locations in conductors of arbitrary shape.

3:30 PM Coffee Break in Exhibit Hall

3:40 PM

THE ELECTROPLASTIC EFFECT IN CERMETS: R. M. Gee[1]; C. Persad[1]; V. Sarin[2]; [1]The University of Texas at Austin, Institute for Advanced Technology, Austin, Texas 78759 USA; [2]Boston University, Boston, MA 02215

Abstract not available.

4:00 PM

MODELING HYPERVELOCITY IMPACT CRATERING IN COPPER AND ALUMINUM: S. C. Quinones[1]; *L. E. Murr*[1]; [1]The University of Texas at El Paso, Department of Metallurgical and Materials Engineering, El Paso, Texas 79968 USA

A 2-D computer code (Autodyn 3.0) has been utilized in modeling the residual hardness contours in crater half sections produced by impacting 3.2 mm diameter 1100 aluminum and soda-lime glass spheres into 1.3 cm or greater thickness targets of polycrystalline copper and 1100 aluminum; over a projectile velocity range from 1 to 6 km/s. Utilizing both Lagrangian and Eulerian processors with a Johnson-Cook constitutive relationship, and appropriate failure criteria, experimental hardness and yield strength measurements have been accurately modeled along with residual crater geometries, rim jetting and fracture, and target spallation. The validated computer calculations in the 1 to 6 km/s range have allowed predictive calculations of cratering in the hypervelocity impact range above 8 km/s where laboratory gun experiments are untenable. Target softening and especially the development of a wide zone of dynamic recrystallization at the crater wall are observed at hypervelocities in both copper and aluminum targets. Supported by NASA-JSC Grant NAG-9-481 and a Murchison Chair Endowment (L.E.M.). Software support provided by Century Dynamics, Inc.

4:20 PM

STRESSES IN SiO2 THIN FILMS: *Terry J. Delph*[1]; [1]Lehigh University, Dept. of Mechanical Engineering & Mechanics, Bethlehem, PA 18105-3085 USA

The oxidation of silicon to form SiO2 is widely used as a method to isolate active devices in integrated circuits. Because a molecule of SiO2 is approximately 2.4 times as large as an atom of silicon, this reaction is accompanied by a large volume expansion. In most practical situations, this expansion is constrained, leading to the generation of stresses on the order of 500 MPa or greater. There is a well-founded belief that these high stress levels have a substantial effect on the oxidation behavior. The standard model which has been developed to describe these effects postulates that both the oxidant diffusion coefficient and the reaction rate constant are exponential functions of stress. One of the basic postulates of the standard oxidation model is that planar oxidation is a stress-free process, with all of the volume expansion taking place normal to the plane of oxidation. However a careful analysis of available experimental data, taking into account time-dependent viscoplastic effects, indicates that this is far from the case. In fact, there exists a relatively small component of expansion in the plane of the oxidation front can generate large in-plane stresses which relax slowly with time. In the case of nonplanar oxidation, this leads to the existence of a boundary layer near the oxidation interface, in which elastic effects play a major role. These results have fairly considerable implications for the correctness of some of aspects of the standard oxidation model.

4:40 PM

HIGH STRAIN SHEAR OF LEAD: M. D. Monti[1]; I. R. Shaw[2]; *W. G. Ferguson*[2]; W. H. Robinson[1]; [1]Penguin Engineering Limited, Penrose, New Zealand USA; [2]University of Auckland, Department of Chemical and Materials, Auckland, New Zealand

M. D. Monti (Penguin Engineering Limited, P. O. Box 33093, Penrose, New Zealand), I. R. Shaw, W. G. Ferguson (Department of Chemical and Materials Engineering, University of Auckland, Private Bag 92019, Auckland, New Zealand), and W. H. Robinson (Penguin

Engineering Limited, Auckland, New Zealand) Initially the high strain deformation characteristics of pure (>99.97%) lead were assessed for the application of lead as a damping media for earthquake technologies. Many tests were performed to explore the mechanical and metallurgical properties including damping, fatigue, and recrystallization of lead during high strain cyclic deformation. All tests were performed on unconstrained lead specimens. Further work was performed to consider the effects of pressure on these characteristics of lead. Tests were performed at pressures from one atmosphere to 70 MPa. The role of micro-voids, pumping, and geometry breakdown were studied, and the contribution of these to the reduction of the shear force and finally to failure was assessed. The knowledge accumulated in this continuing investigation has provided a comprehensive theory of the deformation characteristics of lead. Lead has been shown to be a high damping capacity material suitable for cyclic applications, such as lead-rubber bearings which are used for base-isolation of buildings. Such devices gave excellent performance in the North-Ridge and Kobe Earthquakes.

5:00 PM
EFFECT OF TEXTURE ON THE PSEUDOELASTIC RESPONSE OF A SHAPE MEMORY ALLOY: *Ms. Michelle Somerday*[1]; Dr. John A. Wert[1]; [1]University of Virginia, Materials Science and Engineering, Charlottesville, VA 22903-2442 USA

The dependence of the pseudoelastic elongation of a shape memory alloy (SMA) on single crystal orientation with respect to the applied stress or strain state suggests that texture may have an impact on the pseudoelastic behavior of SMA polycrystals. Prior investigations have recognized this orientation effect and also the impact of grain constraint on the pseudoelastic elongation of a polycrystal. The present article systematically analyzes the effect of texture on pseudoelastic behavior of SMAs, demonstrating that the three main considerations are the specific texture component examined, the constraint experienced by sample grains, and the SMA. Model results show that a <110> fiber texture component has little effect on the pseudoelastic behavior of unconstrained and constrained NiTi polycrystals and unconstrained Cu-Zn-Al polycrystals, but has a modest impact on constrained Cu-Zn-Al polycrystals. A <111> fiber texture affects the pseudoelastic behavior of unconstrained NiTi polycrystals slightly but affects unconstrained NiTi and constrained Cu-Zn-Al polycrystals moderately, and unconstrained Cu-Zn-Al polycrystals greatly. The role of texture in determining the pseudoelastic behavior of SMA can be understood by considering the variation of inverse Schmid or Taylor factor with orientation of the uniaxial tensile stress or extension axis. This work is sponsored by the National Science Foundation under grant no. DMR-9924487.

NON-AEROSPACE APPLICATIONS OF TITANIUM & ITS ALLOYS: Session IV - Armor and Ballistic Applications

Sponsored by: Structural Materials Division, Titanium Committee
Program Organizers: F.H. (Sam) Froes, University of Idaho, IMAP-Mines Bldg. #321, Moscow, ID 83844-3026; P.G. Allen, Timet, P.O. Box 2128, Henderson, NV 89009; M. Niiomi, Toyohashi Univ of Technology, Dept. of Production Systems Eng., Toyohashi 441 Japan

Tuesday PM Room: 101
February 17, 1998 Location: Convention Center

Session Chair: M. G. Wells, US Army Research Center, Weapons and Mat. Res. Directorate, Aberdeen Proving Grounds, MD 21005-5069

2:10 PM
EFFECT OF THERMOMECHANICAL PROCESSING ON THE BALLISTIC PERFORMANCE OF TITANIUM: *Mr. Matthew Burkins*[1]; Mr. Jeffrey Hansen[2]; Mr. Jack Paige[2]; Mr. Paul Turner[2]; [1]U.S. Army Research Laboratory, Attn: AMSRL-WM-TA, Aberdeen Proving Ground, MD 21005-5066; [2]U.S. Department of Energy, Albany Research Center, Albany, OR 97321-2198

Although titanium alloys have been well characterized for aerospace uses, they are not well characterized for use in armor systems. In an effort to provide increased information to armored vehicle designers, the U.S. Army Research Laboratory (ARL) and the U.S. Department of Energy, Albany Research Center (ALRC) performed a joint research program to evaluate the effect of thermomechanical processing on the ballistic limit velocity for an Extra-Low Interstitial (ELI) grade of Ti-6Al-4V. ALRC obtained MIL-T-9046J, AB-2 plates from RMI, rolled these plates to final thickness, performed the annealing, and collected mechanical and microstructural information. ARL then tested the plates with 20mm Fragment Simulating Projectiles (FSPs) and .50 caliber AP M2 bullets in order to determine the ballistic limit velocity of each plate. Titanium processing and annealing did have an effect on the ballistic limit velocity, but the magnitude of the effect depended on which penetrator was used.

2:30 PM
THE EFFECT OF WELDED MICROSTRUCTURE ON BALLISTIC PROPERTIES OF Ti-6Al-4V ALLOY PLATES : *Y. Kosaka*[1]; M. Daggett[1]; B. Bristow[1]; G. Johnson[1]; S. Reichman[1]; M. Burkins[2]; [1]Oregon Metallurgical Corporation, Albany, OR 97321; [2]U.S.Army Research Laboratory, Aberdeen Proving Ground, MD 21005-5066

An attempt was made to understand the effect of welded microstructure on ballistic properties of Ti-6Al-4V alloy plates. A Ti-6Al-4V filler metal was welded by Gas Tungsten Arc Welding (GTAW) on concave dips that were artificially ground on wrought Ti-6Al-4V alloy plates prior to welding. The depth of concave dips was changed up to 20% of the thickness of mother plates. Ballistic test was performed by 20mm FSP which was shot against weld metals or the other side of weld metals. V_{50} was used for the evaluation of a ballistic resistance. Welded plates exhibited lower V_{50} values compared with that of mother plates by up to 12%. The degree of deterioration depended on the depth and the position of weld metals with respect to the projectile. Tested plates were subjected to metallographic observations of the cross-section of fractured areas. The results of the ballistic tests will be discussed in conjunction with metallurgical factors and their fracture behavior.

2:50 PM
TITANIUM APPLICATIONS AND R & D FOR ARMY GROUND SYSTEMS: *Martin G.H. Wells*[1]; Jonathan S. Montgomery[1]; [1]U.S. Army Research Laboratory, Weapons and Materials Research Directorate, Aberdeen Proving Ground, MD 21005-5069

There is a continuing critical need to reduce the weight of military ground vehicles and gun systems. Titanium alloys are ideal candidates for integral armor/vehicle structure applications because of their high strength-to-weight ratio and excellent ballistic performance compared with steel and aluminum alloys. Existing and projected applications of titanium on the M1 Abrams main battle tank, the M2 Bradley Fighting Vehicle, the new light weight 155mm Towed Howitzer and future systems will be reviewed. On-going research and development work to reduce cost both of the initial material and component fabrication will also be discussed. These latter activities include the relaxation of aerospace material specification requirements, the evaluation of a single melt Ti-6Al-4V electron beam hearth melted ingot, the effect of oxygen content in Ti-6Al-4V on mechanical properties and ballistic performance to permit the use of lower cost raw materials, and plate cutting and welding studies.

3:10 PM
EFFECTIVENESS OF TIMETAL(R) 15-3 AS BODY ARMOR AGAINST SMALL ARMS (RIFLES): *Mr. J. C. Fanning*[1]; [1]TIMET Henderson Technical Laboratory, Henderson, NV 89009

TIMETAL(R) 15-3 (Ti-15V-3Al-3Cr-3Sn) is a metastable beta titanium alloy capable of a wide range of mechanical properties depending on heat treatment and processing. The alloy has been used prima-

rily in aerospace applications, particularly fabricated sheet metal structures on aircraft. Recently, interest in TIMETAL(R) 15-3 for non-aerospace applications has been increasing, particularly for body armor (including vests, shields and helmets). The overall effectiveness of conventional body armor materials, such as aramid fabric, can potentially be enhanced by the addition of a titanium alloy sheet or plate. This paper provides the results of ballistic trials on TIMETAL(R) 15-3 tested against several common small arms, including 7.62 x 39mm (AK-47 family), 5.56 x 45mm (M16 family), and 7.62 x 51mm NATO. The purpose of the testing was to identify trends in performance that would be applicable to the use of the alloy in various types of armor systems. In this paper, ballistic protection levels are correlated with mechanical properties, failure modes and microstructures are described, and potential applications are discussed.

3:30 PM

EVALUATION OF A RUSSIAN TITANIUM TANK DOOR: *Martin G.H. Wells*[1]; B. Roopchand[2]; O. N. Senkov[3]; [1]Army Research Lab, Aberdeen Proving Ground, MD USA; [2]TACOM, Warren, MI USA; [3]IMAP, University of Idaho, Moscow, ID USA

A number of Russian tank titanium doors have found their way into the scrap inventory in the United States. In the current study, a tank door which was located in the Oremet scrap yard was evaluated. It was found that the doors consisted either of the Ti-6Al-rV or Ti-4Al-2V alloys. The doors consisted of a beta worked microstructure which would have good ballistic properties. However, it is unclear whether the doors were constructed with ballistic properties in mind, or whether titanium was used rather than steel to reduce weight to allow the doors to be opened manually. The results of the study will be presented in this paper.

PROCESSING-STRUCTURE-PROPERTY RELATIONSHIPS OF COMPOSITE INTERFACES: SESSION III: MECHANICS AND MODELING OF INTERFACES

Sponsored by: Jt. ASM International: Materials Science Critical Technology Sector/Structural Materials Division, Composite Materials Committee, Materials Design & Manufacturing Division, Powder Materials Committee

Program Organizers: Sunil G. Warrier, UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894; Mary Lee Gambone, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433; Ray Y. Lin, University of Cincinnati, Dept. of Materials Sci. & Eng., Cincinnati, OH 45221-0012; Benji Maruyama, WL/MLLM, Wright Lab Materials Directorate, WPAFB, OH 45433

Tuesday PM Room: Centro A
February 17, 1998 Location: Convention Center

Session Chairs: Bhaskar S. Majumdar, UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432; Kevin Kendig, Wright Laboratory Materials Directorate, Wright-Patterson AFB, OH 45433

2:30 PM INVITED

RELATIONSHIPS BETWEEN INTERFACE PROPERTIES AND COMPOSITE PROPERTIES IN SiC/Ti MMC's: *D. B. Miracle*[1]; B. S. Majumdar[2]; S. G. Warrier[2]; D. B. Gundel[3]; [1]Wright Laboratory Materials Directorate, Wright-Patterson AFB, OH 45433 USA; [2]UES, Inc., Dayton, OH 45432; [3]3M Austin Center, Austin, TX

A recent understanding of the influence of interface properties on the deformation and properties of continuously-reinforced metal matrix composites will be presented. SiC/Ti-6Al-4V composites with three different commercial interface coatings which have weak, intermediate, and strong interfacial bonding will be discussed. Techniques for quantifying interface properties under shear and normal loading will first be described, and then the response of these composites under longitudinal loading, transverse creep and tension, and fatigue crack growth will be presented. Future directions for providing an improved balance of longitudinal and transverse composite properties through modification of the interfacial properties will be highlighted.

3:15 PM

THE COMPARISON OF BETA AND ALPHA TITANIUM MATRICES TO THE EVOLUTION OF RESIDUAL STRESSES IN TITANIUM MATRIX COMPOSITES: *P. Rangaswamy*[1]; M. Daymond[1]; M.A.M. Bourke[1]; N. Jayaraman[2]; [1]LANSCE, Los Alamos National Laboratory, Los Alamos, NM 87545; [2]University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221

A study was conducted using X-ray, Neutrons and FEM modeling of residual stresses in two different classes of Titanium Alloys. All of the composites contained Textron's SiC fiber, designated SCS-6, and were based on beta and alpha-beta titanium alloy matrices: Ti-15-3, Ti-6-4 respectively. Residual Stresses were determined using conventional X-ray (d vs. Sin2y) techniques and Neutron diffraction using Time of Flight technique. Continuum Micro-mechanics based Finite Element Modeling was used to predict the average stresses and strain in the surface and the bulk after completion of the fabrication cycle, using multi-ply models taking into account the fiber stacking sequence (both rectangular & hexagonal). Since X-rays measures stresses at the surface and Neutron measure over the entire cross-section of the specimen (bulk), the primary objective of this study was to take into consideration a modeling approach where the surface stresses and the bulk average stresses are predicted to facilitate a realistic comparison with the results obtained through the experimental techniques. Based on the available mechanical properties, the stresses predicted and experimentally determined were highest in the Ti-6-4 matrix composite and lowest in the Ti-15-3 matrix composite. Ti-64 showing higher Young's modulus, Yield Strength and thermal coefficient expansion, and Ti-15-3 showing the lower Young's modulus, Yield Strength and thermal coefficient expansion, these trends support the measured and predicted stresses. Excellent agreement in the stresses determined experimentally and the predictions in the longitudinal direction both qualitatively and quantitatively were achieved. However, only qualitative agreements was achieved in the transverse direction. These results are discussed and presented in the context of the modeling approach taken, experimental issues related to diffraction techniques, possible implications of not taking into consideration the creep effects and the role of the interface.

3:40 PM

INTERFACE RESIDUAL STRESS MEASUREMENT USING NEUTRON DIFFRACTION AND IMPACT ON BOND STRENGTH DETERMINATION OF SiC/Ti-6Al-4V INTERFACES: *S. G. Warrier*[1]; P. Rangaswamy[2]; M.A.M. Bourke[2]; S. Krishnamurthy[1]; D. B. Miracle[3]; J. Roberts[2]; [1]UES, Inc., Dayton, OH 45432; [2]LANSCE, Los Alamos National Laboratory, Los Alamos, NM 87545; [3]Wright Laboratory Materials Directorate, Wright-Patterson AFB, OH 45433

Fiber/matrix interface debonding has been identified with the initiation of non-linearity in the stress-strain curve during transverse loading. The interface bond strength is calculated from the far-field applied debond stress, using finite element analysis. Clearly, the calculated value of the bond strength is controlled by the magnitude of computed radial compressive stress present at the interface. Up to now there has been no systematic experimental study of the magnitude of this stress component. In this study, the radial compressive stress at the interface for three different SiC/Ti-6Al-4V composite interfaces with significantly different characteristics has been measured using the time of flight neutron diffraction technique. The measured residual stress values, finite element predictions and their effect on bond strength measurement will be addressed. This research has been performed at the Wright Laboratory Materials Directorate with support from AF Contract F33615-96-C-5258 and at LANSCE, Los Alamos National Laboratory.

4:05 PM Coffee Break in Exhibit Hall

4:20 PM
VALIDATION OF MULTI-CONCENTRIC CYLINDER MODEL FOR SAPPHIRE/MO/NiAl COMPOSITES: *D. E. Boss*[1]; J. W. Richardson, Jr.[2]; [1]Northwestern University, Evanston, IL 60201; [2]Argonne National Laboratory, Argonne, IL 60439

The large coefficient of thermal expansion mismatch between many of the compounds and existing fiber reinforcements creates significant thermal residual stresses, which may lower the mechanical properties of the composite. One approach to mitigating the thermal residual stresses is the use of fiber coatings. Many studies have been performed to model the effect of fiber coatings on the residual stress distribution in composites, but few studies have been down to validate the accuracy of the models. In this study, the residual stress calculations of a concentric cylinder model, developed by NASA, for the sapphire/NiAl system with a molybdenum interface coating are compared to residual stress measurements made using neutron diffraction. Four different thicknesses of Mo interface coatings are evaluated.

4:45 PM
A STUDY OF THE SIGNIFICANCE OF FEM ANALYSIS OF SINGLE FIBER PUSH-IN WITH INTERFACE FRICTION: *K. V. Pochiraju*[1]; G. P. Tandon[2]; N. Pagano[3]; [1]Stevens Institute of Technology, Department of Mechanical Engineering, Hoboken, NJ 07030; [2]Adtech Research Systems, Inc., Dayton, OH; [3]Wright Laboratory, WL/MLBM, Wright-Patterson AFB, OH 45433

Accurate analysis methods enhance the understanding of the fiber-matrix interfacial damage evolution and may also provide improved methods for obtaining material property values from experimental data. In this paper, the detailed stress distributions and load-displacement solutions obtained by using two different techniques capable of considering several interfacial conditions are compared. The models considered are the Axisymmetric Damage Model (ADM) which produces an approximate elasticity solution and the Finite Element Method (FEM) with the fiber-matrix interface modeled as frictional contact. ADM computes the load-displacement relationship and stress fields in the model for a pre-specified state of interface damage without using incremental loading. Several simulations are performed for polyester fiber/epoxy matrix system for which experimental data is available. The ADM and FEM show good correlation in force-displacement relationships and the stress fields for the bonded and frictional sliding damage zones. Some differences between the solutions are noted and explained.

5:10 PM
EFFECT OF INTERFACIAL DEFORMATION ON THE CREEP BEHAVIOR OF CONTINUOUS FIBER REINFORCED METAL-MATRIX COMPOSITES: *I. Dutta*[1]; R. Nagarajan[1]; J. E. Funn[1]; M. Esmele[1]; [1]Center for Materials Science and Engineering, Department of Mechanical Engineering, Monterey, CA 93943

Results of experimental creep studies of interfaces in metal-matrix composites, based on fiber-pushout tests, will be reported. Based on the experimental results on model systems (Pb matrix with Ni, W and quartz fibers), a law for interfacial strain accommodation will be proposed. Analytical results of a model for composite creep, utilizing this law for interfacial sliding, will be presented, and the impact of interfacial sliding and changing matrix creep mechanisms at different times during composite creep will be evaluated. Results of separate experiments to isolate and evaluate the contributions of the interface and matrix on the overall composite creep strain will also be reported. Finally, creep data on commercially available Al-SiC and Al-Al2O3 fiber composites will be presented and analyzed in light of the results obtained from the model systems. Supported by the National Science Foundation under contract # DMR-9423668.

5:35 PM
MODELING OF MICROSTRUCTURE-MECHANICAL PROPERTIES RELATIONSHIPS IN SiC-COATED CARBON FIBERS: *Horacio Nassini*[1]; [1]Comisión Nacional de Energía Atómica (CNEA), Centro Atómico Bariloche, (8400) Bariloche-Río Negro República Argentina

SiC thin films deposited by CVD on carbon fibers are frequently proposed as a diffusion barrier to prevent the chemical reactions at high temperatures between reinforcing fibers and matrix in aluminum-based composites. Single-filament tensile tests have shown that mechanical properties of SiC-coated carbon fibers are strongly degraded respect to uncoated fibers, and this phenomenon is mainly attributed to the significant thermal stresses generated during coating process, due to large differences in thermal expansion coefficients between substrate and coating. In this paper, a model to theoretically predict the tensile mechanical properties of SiC-coated carbon fibers as a function of coating microstructure and thickness, is presented. The model is based on a detailed stress analysis with a microcomposite coaxial cylinder model, complemented by concepts of linear-elastic fracture mechanics.

REACTIVE METALS—GENERAL SESSIONS: Reactive Metals — General Session II

Sponsored by: Light Metals Division, Reactive Metals Committee
Program Organizer: John N. Hryn, Argonne National Laboratory, 9700 S. Cass Ave. Bldg. 32, Argonne, IL 60439

Tuesday PM Room: 209
February 17, 1998 Location: Convention Center

Session Chair: Keith Axler, Los Alamos National Labs., Los Alamos, New Mexico

---

2:00 PM
THE DEVELOPMENT OF STABLE CERAMIC MATERIALS FOR THE CONTAINMENT OF MOLTEN ZIRCONIUM AND URANIUM ALLOYS: *Sean M. McDeavitt*[1]; Garth W. Billings[2]; [1]Argonne National Laboratory, Chemical Technology Division, Argonne, IL 60439 USA; [2]Integrated Thermal Sciences, Inc.

The effective containment of molten reactive metals is a critical issue in the electrometallurgical treatment of spent nuclear fuel as well as in the reactive metals industry. Electrometallurgical treatment includes melt-consolidation steps for stainless steel-zirconium (SS-Zr) alloys and for uranium metal. Although cold-hearth melting may be an option for future processing equipment, existing process equipment at ANLW est mandates the hot containment of these molten reactive metals. Candidate ceramic materials consisting of oxides, carbides, nitrides, borides and sulfides were selected based on thermodynamic stability and a series of elevated temperature interaction experiments have been performed using zirconium, SS-Zr alloys and uranium under inert gas at temperatures up to ~2100°C. The experiments were recorded in situ using an external camera. The best-performing materials were fabricated into prototype crucibles (~10 cm dia by ~15 cm tall) for bench-scale melting experiments. The results of the interaction experiments, materials selection and refinement process, and prototype experiments will be discussed.

2:30 PM
PLASMA ARC MELTING OF URANIUM-6 WT. % NIOBIUM ALLOYS: *Paul S. Dunn*[1]; Deniece R. Korzekwa[1]; Philip K. Tubesing[1]; [1]Los Alamos National Laboratory, Materials Science Technology Division, Los Alamos, NM

Ingot fabrication of uranium-6 wt.% niobium(U-6Nb) alloys historically have been fabricated using a three step melting process with vacuum arc remelting (VAR) being the final step. The ingots from this processing scheme have extensive macrosegregation in the form of banding with alternating niobium contents ranging from 3.5 to 11.0 wt. % niobium. The segregation problem is due to the large variation in melting point and density between uranium and niobium as well as

inherent segregation problems in the VAR process. Plasma arc melting (PAM), was investigated as a melting technique for the U-6Nb alloy in an attempt to improve the macrosegregation problem and minimize the number of melting steps to make acceptable ingots. The paper presents the results of variations in melt parameters; plasma gas composition, power input and melt stirring, on ingot quality. All ingots were characterized by wet chemistry, X-ray florescence and optical metallography. The results indicate that hydrogen additions to the plasma gas reduce oxide slag that forms during the melting process and improves ingot quality. Additionally, the macrosegregation is reduced using the PAM process because of the smaller molten pool and lower local solidification times. Based on these results, a two step PAM process has been selected for further development of the alloying and processing sequence.

3:00 PM
CARBON REDUCTION IN URANIUM BY PLASMA ARC MELTING: *Philip K. Tubesing*[1]; Deniece R. Korzekwa[1]; Paul S. Dunn[1]; [1]Los Alamos National Laboratory, Los Alamos, NM

Traditional vacuum induction melting (VIM) of uranium increases carbon impurity levels in uranium due to the use of graphite hardware. Any higher-carbon uranium destined for remelting is diluted with chemically purified lower-carbon material to control the carbon level in the final casting. We undertook a study to determine if plasma arc melting (PAM) technology could be utilized to reduce the quantity of carbon impurity in uranium. By using PAM and varying the material processing rates and plasma gases, we successfully achieved reduced levels of carbon impurity. This paper will discuss the PAM technique, introduce the concept of gas additions into the plasma stream, and present the results of our gas, chemical composition, and metallographic second phase analyses.

3:30 PM Coffee Break in Exhibit Hall

4:00 PM
CASTING OF URANIUM AND BISMUTH THROUGH FILTERS: EXPERIMENTS AND SIMULATIONS : *Deniece R. Korzekwa*[1]; Paul S. Dunn[1]; Philip K. Tubesing[1]; [1]Los Alamos National Lab, Materials Science and Technology Division, Los Alamos, NM 87545

Filters are used in the casting industry to physically remove inclusions in the material, as well as improve the fluid flow within the mold assembly. This study looks at the effect of filters in a uranium casting process. The filters were placed in the mold assembly to lower the carbon content in the uranium. Al_2O_3, FSZ, ZTA and YAG filters with both 20 pores per inch (ppi) and 65 ppi were tested. Castings were poured at varying superheats and pour rates. The carbon content was measured in the resulting castings to determine the effectiveness of the filter and the filters were analyzed both macro and microscopically. Computer simulations were done using the code FLOW-3D. The main goal of the modeling was to explore fluid flow through the filters and to predict the residence time of material in various filters. To validate the code predictions, real time radiography was made of bismuth flowing through filters. Good correlation was observed between the simulations and the experiments.

4:30 PM
PHASE DEVELOPMENT IN AS-CAST, ZIRCONIUM-BASED ALLOYS CONTAINING TYPE 304 STAINLESS STEEL AND Pu: *Dennis D. Keiser, Jr.*[1]; Sean M. McDeavitt[2]; [1]Argonne National Laboratory - West, Idaho Falls, ID 83403-2528; [2]Argonne National Laboratory-East, Argonne, IL USA

Argonne National Laboratory has developed a stainless steel-zirconium alloy to contain the metallic waste components that are residual from its electrometallurgical treatment for spent nuclear fuels. This process extracts uranium from irradiated fuel elements. In cases where the treated fuel elements have Zircaloy cladding, the resulting metal waste form will be zirconium-rich. Other minor constituents found in the alloy include components found in stainless steel (Fe, Ni, Cr, Mo, etc.), actinides (U, Pu, and Np), and what have been termed for the electrometallurgical treatment process "noble metal" fission products (Tc, Pd, Ru, Re, etc.). To study the microstructural and phase development in as-cast, zirconium-rich alloy ingots that contain stainless steel constituents and actinides, different zirconium-based alloys with added Type 304 stainless steel and Pu have been cast in induction and resistance furnaces and have been analyzed using SEM/EDS and XRD. The observed microstructures, the types of developed phases, and the partitioning behavior of the various components between the alloy phases are discussed. Microstructural development in these alloys is compared with that reported for Zircaloy-2 and 4. Adding Pu to a Zr-8 wt.% 304SS alloy was observed to produce little change in the microstructure or the types of phases that develop upon solidification of the alloy. Increasing the amount of added Pu, up to a maximum of 10 wt.% Pu, resulted in a higher Pu solubility in the observed alpha-Zr phase and a higher Pu concentration at alpha-Zr lath boundaries. The highest Pu concentrations were observed at the alpha-Zr lath boundaries.

SOLIDIFICATION AND DEPOSITION OF MOLTEN METAL DROPLETS: Session I

Sponsored by: Materials Design and Manufacturing Division, Solidification Committee

Program Organizers: Men G. Chu, Alcoa Technical Center, Molten Metal Processing Center, Alcoa Center, PA 15069; Enrique J. Lavernia, Univ. of California-Irvine, Dept. of Chem Eng & Matls Sci, Irvine CA 92217-2575; Jung-Hoon Chun, M.I.T., Dept. of Mechanical Engineering, Cambridge, MA 02139

Tuesday PM Room: Plaza Room D
February 17, 1998 Location: Convention Center

Session Chair: Men G. Chu, Alcoa Technical Center, Molten Metal Processing Center, Alcoa Center, PA 15069

2:00 PM INVITED
SOLIDIFICATION OF SPRAY FORMED ALUMINUM ALLOY DROPLETS: Dr. Douglas M. Matson[1]; Mr. Matthieu Rolland[1]; *Dr. Merton C. Flemings*[1]; [1]Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139 USA

Bench-scale laboratory equipment was built to study process parameters in spray casting of aluminum alloys. Mean droplet size ranged from about 30 - 80 microns depending on spray pressure. Droplet speeds were up to about 6 meters per second as measured by high speed video imaging. Alloys studied were Al-4.5% Cu(wt.) and Al-4.3% Fe(wt.). Undercooling was estimated from metallographic sections by measuring the volume fraction solid which formed in supercooled liquid. Cooling rates were estimated from dendrite arm spacing measurements. A heat flow and solidification model based on the Ranz-Marshall correlation showed good agreement with experiment.

2:25 PM INVITED
HETEROGENEOUS NUCLEATION DURING SOLIDIFICATION OF DROPLETS: Mr. Peter G. Höckel; Dr. Heino Sieber; *Prof. John H. Perepezko*; University of Wisconsin - Madison, Materials Science and Engineering, Madison, WI 53706 USA

During the central processing step of spray deposition and other atomization methods where a bulk liquid is dispersed into a droplet stream, nucleonic isolation takes place, promoting undercooling of the droplets. The crystallization of undercooled droplets offers an effective method to examine nucleation kinetics due to catalysis by primary phases and also by incorporated particles. Both single droplet and droplet population samples provide complimentary information on nucleation behavior which has a strong impact on solidification process modeling. Often, heterogeneous nucleation occurs at the interface between the primary phase and the undercooled liquid. In order to relate nucleation to specific sites, controlled thermal cycles have been performed in eutectic and peritectic systems. The results from recent

studies suggest some deficiencies in the conventional spherical cap model of catalysis and identify new directions for kinetics analysis and modeling. The support of the NSF (DMR-9308148) and NASA (NAG8-1278) is gratefully acknowledged.

2:50 PM

ATOMIZATION PROCESSING EFFECTS ON MICROSTRUCTURE SELECTION IN ALUMINUM ALLOY POWDERS: Mr. James P. Foley[1]; Dr. Iver E. Anderson[1]; Matthew G. Osborne[1]; Dr. Rohit K. Trivedi[1]; [1]Ames Laboratory, Ames, IA 50011 USA

The range of processing parameters available through the use of different atomization techniques have allowed a variety of solidification microstructures to be investigated in Al-Si and Al-Cu alloy powders. Two types of atomization, a high pressure gas atomization process and a rotating disk atomization process with an optional liquid spray quench, permitted access to several regimes of rapid solidification because of the wide differences in particle size and external cooling environment that could be imposed. Alloy compositions were varied from eutectic to near-eutectic to study variations in solidification microstructure of the particles in both coupled growth and primary phase nucleation controlled growth morphologies. Microanalytical techniques in the SEM and quantitative metallographic analysis permitted phase identification, phase volume fractions, and interphase spacing measurements. The results have been analyzed by using rapid solidification models to gain understanding about the particle solidification path and the probable nucleation event for each type of sample. Support from the DOE-BES-DMS under contract no.W-7405-Eng-82 is gratefully acknowledged.

3:10 PM

N-FLIGHT SOLIDIFICATION BEHAVIOR OF ALUMINUM ALLOY DROPLETS PRODUCED BY THE UNIFORM DROPLET SPRAY PROCESS: *Ms. Jean-Pei Jeanie Cherng*[1]; Professor Jung-Hoon Chun[1]; [1]Massachusetts Institute of Technology, Department of Mechanical Engineering, Cambridge, MA 02139 USA

The uniform-droplet spray (UDS) process produces uniformly-sized, molten metal droplets by imposing sinusoidal perturbations on a laminar jet. The thermal and dynamic states of the uniform droplets are precisely controlled, as opposed to those in conventional spraying processes which inherently produce ranges of spraying conditions and hence non-uniform microstructures and porosity when used in spray forming. In this research, the UDS process was adapted to spray aluminum alloys. A droplet thermal model was developed by assuming Newtonian cooling and the Scheil behavior during solidification. To verify the thermal model, droplets of aluminum alloys were quenched at different flight distances and examined metallographically. Due to the large difference in cooling rates, it is possible to distinguish the solid phase formed during flight from the liquid phase prior to quenching. The fraction solid was measured with image analysis tools and the results are discussed in detail.

3:30 PM

SOLIDIFICATION BEHAVIOR OF MOLTEN METAL DROPLETS: *Mr. Hidekazu Todoroki*[1]; R. Lertarom[1]; A. W. Cramb[1]; T. Suzuki[2]; [1]Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA; [2]University of Tokyo, Department of Metallurgy, Tokyo 113 Japan

In order to understand the solidification behavior of pure iron, nickel, 304 stainless and IF steels, experiments were undertaken to measure the heat transfer rate and solidification structure of droplets solidified against a water cooled copper mold. Temperature measurement of the mold-side cast surface was carried out by a photo-diode sensor while in the copper mold the temperature profile was determined by thermocouples. Significant undercooling, up to 150 K below the equilibrium melting point or liquidus, was observed in most cases before the initiation of solidification and was found to be more significant in the case of alloy solidification. The heat flux and the heat transfer coefficient was evaluated between the cast metal droplet and the mold during the first 0.5 seconds of solidification. Both quantities were found to vary as a function of time, superheat and liquid metal composition. For 304 stainless steels, microstructural observation allowed relationships to be developed between undercooling, the cooling rate of a droplet, the heat flux and the solidification structure of the solidified droplet.

3:50 PM Coffee Break in Exhibit Hall

4:00 PM

DROPLET SOLIDIFICATION USING THE IMPULSE ATOMIZATION TECHNIQUE: Louis C. Morin[1]; S. Hoskins[1]; A. Merle[1]; H. Henein[1]; [1]University of Alberta, Advanced Materials and Processing Laboratory, Edmonton, Alberta T6G 2G6 Canada

Impulse Atomization is a novel single fluid atomization technique which generates droplets in a narrow size range. A wide range of lead, aluminum and copper alloys have been atomized using this technique under a range of operating conditions. In this paper we shall examine the morphology and the microstructure of some of these alloys. The first series of experiments to be examined deal with the atomization copper. It will be shown that the droplets form by the growth of surface instabilities on a segment of fluid. These fluid segments emanate from the atomizing nozzle with each frequency applied to the melt. Examination of IA powders of 90Pb/10Sn and 89Cu/11Sn bronze revealed surface porosity. In addition, microstructural examination of the powder cross-section indicates that droplet solidification nucleated inside the droplet, with crystal growth occurring to the droplet surface. Finally, for a number of alloys and at the same powder size, powder microstructure will be shown to be considerably finer by Impulse Atomization than by gas atomization. These findings reveal that Impulse Atomization is a rapid solidification technique with great flexibility in engineering alloy microstructure.

4:20 PM

FACTORS INFLUENCING PENETRATION OF PARTICLE INTO DROPLET IN SPRAY FORMING AND CO-INJECTION TECHNIQUES: *Mr. Bing Li*[1]; Dr. Enrique J. Lavernia[1]; [1]University of California, Irvine, Department of Chemical & Biochemical Engineering and Materials Science, Irvine, CA 92697-2575 USA

Spray forming and co-injection technique has evolved into one of the few most promising methods for synthesizing metal/intermetallic matrix composites over the last decade. One of the key steps governing the formation and the characteristics/performance of the composite in spray forming and co-injection technique is the penetration of ceramic particles into droplets. Effective penetration of particles into the droplet and their homogeneous distribution in the droplet may lead to uniform incorporation of reinforcements in the final product bulk material. Segregation of particles in the droplets because of limited penetration capability, on the other hand, would generally give rise to segregation of reinforcements in the deposit, such as layered characteristics. In this study, the factors affecting the penetration behavior, including droplet temperature, droplet status (solid/semi-solid/liquid), droplet velocity, droplet size, droplet flight time, particle velocity, and particle size were synergetically investigated. For all of these mentioned factors except droplet flight time, their individual effects on the penetration behavior were investigated previously. The present study focused on the combined effect of these factors, especially droplet temperature and droplet status.

4:40 PM

THE NUCLEATION OF ZIRCONIUM MELTS: *Craig W. Morton*[1]; William H. Hofmeister[1]; Robert J. Bayuzick[1]; Aaron J. Rulison[2]; John L. Watkins[2]; [1]Vanderbilt University, Applied and Engineering Sciences, Nashville, TN 37240 USA; [2]Space Systems/Loral, Palo Alto, CA 94303-4604 USA

Using classical nucleation theory, nucleation temperature data from zirconium undercooling experiments has been evaluated. The undercooling experiments were conducted on millimeter size samples of high purity zirconium in an electrostatic levitator at Space Systems/Loral. Several sets of nucleation temperature data were obtained in a containerless, high vacuum environment using various processing conditions. The effects of initial material purity level, melt overheat, and sample size on undercooling and nucleation have been investigated. For each data set, the preexponential factor and work of cluster formation in the classical nucleation rate equation were determined using a statistical analysis technique. The values for the mean undercooling

ranged from 326 K (0.15 T_m) to 349 K (0.16 T_m) and scaled with the material purity. The values for the preexponential factors and the work terms also scaled with the purity levels but were higher than the theoretical estimates. Melt overheat and sample size differences were observed to have no detectable effect on the nucleation kinetics. The results will be discussed in terms of the limitations of the classical nucleation rate equation.

5:00 PM

FORMATION AND UNDERCOOLING BEHAVIOR OF LIQUID DROPLETS ENTRAINED IN ITS PRIMARY PHASE: *Dr. O. P. Pandey*[1]; [1]Thapar Institute of Engineering & Technology, School of Basic & Applied Sciences, Patiala 147001 India

The formation and undercooling behavior of various size liquid droplets entrained in its primary matrix was studied for Ag-Ge and Ag-Cu alloys. A wide size range of droplets were generated in the matrix of Ag for Ag-Ge alloys. A maximum undercooling of 81°C below the eutectic temperature was recorded for Ag-Ge alloys. Similarly for Ag-Cu alloys an undercooling of liquid droplets were extended up to line temperature undergoes diffusionless solidification. On further cooling, the solidified liquid phase transforms through the spinodal route. Examination of solidification structure of the undercooled alloys by TEM, X-ray diffraction, polarized light microscopy and EPMA techniques indicated presence of metastable phase. The mechanism of formation of droplets and their non-equilibrium solidification structure analyzed through different techniques will be discussed.

TUESDAY PM

STRENGTHENING IN HIGH TEMPERATURE INTERMETALLICS: STRENGTHENING IN HIGH TEMPERATURE INTERMETALLICS IV: Gamma Titanium Aluminides 1

Sponsored by: Structural Materials Division, Mechanical Metallurgy Committee

Program Organizers: Young-Won Kim, UES, Inc., Materials & Proc. Division, Dayton, OH 45432-1805; Michael J. Kaufman, University of Florida, Dept. of Materials Sci & Eng., Gainesville, FL 32611-2066; Chain T. Liu, Oak Ridge National Lab, PO Box 2008 Bldg 4500S, Oak Ridge, TN 37831-6115

Tuesday PM Room: 107
February 17, 1998 Location: Convention Center

Session Chairs: Dennis M. Dimiduk, Wright Laboratory, Materials Directorate, Wright-Patterson AFB, OH 45433; Michael V. Nathal, NASA-Lewis Research Center, Cleveland, OH 44135

2:00 PM

ALLOY MODIFICATIONS TO OPTIMIZE STRENGTH, DUCTILITY AND MICROSTRUCTURAL STABILITY IN ULTRAFINE LAMELLAR Ti-47Al-2Cr-2Nb ALLOYS: *Dr. Philip J. Maziasz*[1]; Dr. C. T. Liu[1]; [1]Oak Ridge National Laboratory, Metals Ceramic Division, Oak Ridge, TN 37831-6115 USA

Hot-extrusion and/or heat-treatments above the α-transus temperature (Tα) produce unique refined colony-ultrafine lamellar structures in a Ti-47Al-2Cr-2Nb base alloy. The combination of colony size <70 μm, average interlamellar spacing (λL) of 100-200 nm, and α_2- α_2 ($l_{/\alpha}$) of 200-500 nm produces 3-5% ductility and up to 800 MPa yield-strength (YS) at room temperature, and 600-750 MPa at 800°C. Additions of B and W refine the lamellar structure, and lowering the Al content of the alloy from 47 to 46 at.% further refines both λ_L and λ_α. Additions of W and B enhance the coarsening resistance of the ultrafine lamellar structure during aging at 800°C. Microcompositional analysis of individual lamellar reveals that the α_2 forms with a metastable composition in the as-processed materials, and that diffusional changes toward the equilibrium α2 phase composition precede continuous lamellar coarsening during aging. Alloy design strategies for improving microstructural refinement and stability in wrought γ-TiAl alloys will be discussed. Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials (AIM) Program under contract DE-AC05-96OR22464 with Lockheed-Martin Energy Research Corp.

2:30 PM

STRENGTHENING IN TiAl ALLOYS: *Dr. Young-Won Kim*[1]; [1]UES, Inc., Dayton, OH 45432 USA

Significant increases in tensile strength in TiAl alloys have recently been possible by refining lamellar grains and structure through advanced wrought processing routes and heat-treatments. Lamellar arrangements in these designed microstructures sometimes can be textured colonies which, though not quantified, offer increased ductility. These controlled microstructures can also enhance secondary creep resistance, fatigue strength and damage tolerance below BDTT. However, their benefits for crack initiation/threshold (K1c/Kth), impact resistance, and higher temperature deformation/fracture are either uncertain or known only qualitatively. Furthermore, such beneficial design has not been demonstrated in thick-section specimens. This talk will first assess the methods and mechanisms for designing the refined microstructures and then discuss the limitations in producing balanced, or application-specific, properties. The ongoing investigations at Wright Laboratory indicate that the shortcomings can be significantly alleviated by alloy modification and microalloying. These results will also be evaluated. Finally, the importance of combining microstructure control and chemistry modification for strengthening gamma alloys in a balanced way will be discussed with highlights from specific examples. * UES, Inc., Dayton, OH 45432, USA

3:00 PM

PRECIPITATION OF CARBIDES AND NITRIDES IN TiAl: *Prof. Minoru Nemoto*[1]; K. Oh-ishi[1]; W. H. Tian[1]; S. Komatsu[1]; [1]Kyushu University, Department of Materials Science and Engineering, Fukuoka 812-81 Japan

Transmission electron microscope observations have revealed that fine needle shaped precipitates of perovskite type appear parallel to only the [001] ax is of carbon or nitrogen doped TiAl during aging after quenching from higher temperatures. The cross section of needle is rounded square for carbide and rounded rectangular for nitride at lower aging temperature. At higher aging temperatures both the needles have a rounded square cross section. Selected area electron diffraction analyses have revealed that long range ordering of carbon-vacancy and nitrogen-vacancy pairs exists in the perovskite needles at lower aging temperatures. Two variants of ordered domains coexist in a carbide needle but only one domain in a nitride needle. Long range ordering becomes less dominant or disappears at higher aging temperatures. The room temperature strength as well as high temperature strength of TiAl increase appreciably by fine precipitation of carbide and nitride needles.

3:20 PM

EFFECT OF HOT-EXTRUSION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF A TWO-PHASE TiAl ALLOY: *M. Oehring*[1]; F. Appel[1]; R. Niefanger[1]; U. Lorenz[1]; S. Eggert[1]; R.Wagner[1]; H. Clemens[2]; N. Eberhardt[2]; [1]GKSS Research Center, Institute for Materials Research, Geesthacht D-21502 Germany; [2]Plansee Aktiengesellschaft, Technology Centre, A-6600 Reutte, Austria

Cast ingots of a Ti-47at.% Al-(Nb, Cr, Mn, Si, B) alloy were hot-extruded in order to investigate the influence of processing conditions on microstructural development and the resulting mechanical properties. Hot-extrusion was performed over the temperature range 1250 - 1380°C on differently heat treated billets. Depending on extrusion temperature either fine equiaxed or refined lamellar microstructures were obtained. The microstructural homogeneity was found to depend on the heat treatment prior to extrusion. After hot-extrusion, compression and chevron tests were conducted with specimens parallel and perpendicular to the extrusion direction, so that the effect of the

texture developed during extrusion on properties could be examined. The results indicate an anisotropy in strength and fracture toughness. TEM was used to characterize the microprocesses of plastic deformation in these specimens with respect to the observed mechanical properties.

3:40 PM
GRAIN GROWTH IN TIAL-BASED INTERMETALLICS WITH A NANOCRYSTALLINE STRUCTURE AND ITS EFFECT ON PROPERTIES: *Dr. Oleg N. Senkov*[1]; N. Srisukhumbowornchai[1]; Prof. Froes H. Froes[1]; [1]University of Idaho, Institute for Materials and Advanced Processes (IMAP), Moscow 83844-3026 ID

Grain growth in nanocrystalline TiAl-based compacts, Ti-47.5Al-3Cr and Ti-48Al-2Nb-2Cr, was studied during annealing at 725°C to 1200°C for up to 800 hours. These compacts were synthesized by hot isostatic pressing from mechanically alloyed amorphous powders. Normal grain growth occurred which can be described by a single thermally activated rate process limited by a permanent pinning force. The activation energy of the grain growth was close to the activation energy for lattice diffusion of Ti in TiAl. The microhardness of the nanocrystalline samples was reciprocal to a square root of the grain size, following the Hall-Petch relationship.

4:00 PM
THE YIELD STRENGTH OF SEMI-COHERENT LAMELLAR INTERMETALLICS: *Dr. Peter M. Hazzledine*[1]; Dr. Satish I. Rao[1]; [1]U.E.S. Inc., Dayton, OH 45432 USA

The yield strength of a lamellar intermetallic such as polysynthetically twinned Ti-Al in the hard mode is controlled by the lamellar thickness through the Hall-Petch effect. In this material the lamellae are thick enough to permit the development of regular pile ups of dislocations across the lamellae. In the high-strength fully lamellar Ti-Al alloys, however, the lamellae are thin enough that several new effects appear: Sizeable coherency stresses exist in the lamellae and consequently the density of mismatch dislocations decreases, the lamellar thickness is comparable with dislocation spacings and consequently the pile ups break into queues of pile ups. All of these effects change the value of the Hall-Petch stress. This paper reports a calculation of the yield strength expected in very fine lamellar materials.

4:30 PM
MICROSTRUCTURAL EFFECT ON FATIGUE CRACK PROPAGATION IN FULLY LAMELLAR TiAl ALLOYS AT ROOM TEMPERATURE AND 800°C: Mr. C. Choi[1]; *Professor Chong So Lee*[1]; [1]Pohang University of Science and Technology, Department of Materials Science and Engineering, Pohang 790-784 Korea

Fatigue crack propagation (FCP) behavior of gamma TiAl alloys is largely affected by microstructural variations, and it is known that fully lamellar microstructures result in the highest fatigue crack growth resistance at both room and high temperatures. In the fully-lamellar material, the FCP resistance is expected to additionally depend on microstructural features such as grain size and lamellar spacing, but this critically important aspect has not been investigated systematically. In this study, this aspect has been investigated at RT and 800°C for two gamma TiAl alloys: Ti-46.5Al-2Cr—3Nb-0.2W and Ti-47Al-1.5Cr-0.5Mn-2.5Nb-0.17B. The results from the tests of CT specimens and detailed microscopic analysis have shown that the FCP is a function of the two microstructural features and that the relative effect varies with temperature. The influence of lamellar spacing in FCP is much greater than grain size at RT; at 800°C, however, it is reduced to result in similar da/dN curves for both fine (0.4 μm) and coarse (5.5 μm) lamellar spacings. The individual and combined effects of the microstructural features, and their temperature dependence, on the FCP resistance will be analyzed by incorporating the observed fracture modes.

4:50 PM
THE EFFECT OF PRIOR LOW CYCLE FATIGUE ON THE HIGH CYCLE FATIGUE BEHAVIOR OF Ti-46 Al-2Nb-2Cr-1Mo-0.2B: *David C. Maxwell*[1]; Dr. A. H. Rosenberger[2]; Dr. J. M. Larsen[2]; [1]University of Dayton Research Institute, Dayton, OH 45469-0128 USA; [2]Air Force Research Laboratory, Materials Directorate (WL/MLLN), OH 45433-7818 USA

A Goodman stress-life diagram has been developed for a gamma titanium-aluminide alloy, composition Ti-46Al-2Nb-2Cr-1Mo-0.2B (at%), at stress ratios from -1 to +0.9 at room temperature, 540°C, and 760°C. The low cycle fatigue life (10^5 cycles) has been determined at three stress ratios and specimens were fatigued to 10, 25 and 50 percent of their low cycle fatigue life and then tested to failure in high-cycle fatigue. The goal of this study was to determine the effect of prior low-cycle fatigue damage on the high-cycle fatigue life and access the validity of Miner's Law for this titanium-aluminide alloy.

5:10 PM
THE INFLUENCE OF THE TEXTURE ON MECHANICAL PROPERTIES IN SHEETS OF TiAl ALLOYS: *Dr. Arno Bartels*[1]; Dr. Christian Hartig[1]; Dr. Helmut Clemens[2]; [1]Technical University Hamburg-Harburg, Physics and Technology of Materials, Hamburg D-21071 Germany; [2]Metallwerk Plansee GmbH, Technology Center for New Materials, Reutte/Tirol A-6600 Austria

Sheets of TiAl alloys rolled at Plansee AG in Austria exhibit a near-gamma or duplex microstructure with a modified cube texture. The c-axes of the tetragonal unit cells in the grains are aligned with the transverse direction in the sheets. This cube texture causes anisotropies of the yield stress, ductility and work hardening which depend on the temperature. At 700°C, the yield stress in the transverse direction is up to 25% higher than in rolling direction and even higher than at room temperature. Also the creep resistance at 700°C is improved in transverse direction. During heat treatments up to 1400°C the modified cube texture becomes weaker and consequently the anisotropy of the mechanical properties decreases.

SUPERPLASTICITY AND SUPERPLASTIC FORMING: Session IV - High Strain Rate Superplasticity

Sponsored by: Materials Design and Manufacturing Division, Shaping and Forming Committee
Program Organizers: Amit K. Ghosh, Univ of Michigan, Dept. of Matls Sci & Eng., Ann Arbor, MI 48109-2136, Thomas R. Bieler, Michigan State Univ, Dept of Matls, Sci & Mechanics, E Lansing, MI 48824-1226

Tuesday PM — Room: 106
February 17, 1998 — Location: Convention Center

Session Chair: Terrence G. Langdon, University of Southern California, Departments of Materials Science and Mechanical Engineering, Los Angeles, CA 90089-1453

2:00 PM INVITED
A CRITICAL EVALUATION OF HIGH STRAIN RATE SUPERPLASTICITY: *R. S. Mishra*[1]; A. K. Mukherjee[1]; [1]University of California, Department of Chemical Engineering and Materials Science, Davis, CA 95616

High strain rate superplasticity (HSRS) has been observed in a number of alloys and composites. The reported data base is extensive enough to perform a critical evaluation of the mechanism of HSRS. The prior thermomechanical processing has a major influence on the optimum HSRS parameters. Our analysis shows a very clear relationship between microstructural refinement and origin of HSRS. In-situ transmission electron microscopy results are also presented. These results completely rule out the possibility of any role of liquid phase on the onset of HSRS. Based on these results, a microstructural mechanism for HSRS is presented. The present approach also leads to processing strategies for developing microstructure for optimum HSRS.

2:25 PM INVITED

THE SIGNIFICANCE OF LOAD TRANSFER IN HIGH STRAIN RATE SUPERPLASTICITY OF METAL MATRIX COMPOSITES: *Y. Li*[1]; T. G. Langdon[1]; [1]University of Southern California, Departments of Materials Science and Mechanical Engineering, Los Angeles, CA 90089-1453

Superplasticity has been observed in a number of metal matrix composites deformed at strain rates ranging from 10-2 to 102s-1. By considering the possibility of the presence of a threshold stress in the deformation process, a true stress exponent close to 2 is often obtained, but the true activation energy is generally much higher than the value for self-diffusion energy in the matrix. With incorporation of load transfer into the analysis, it is demonstrated that the high value of the activation energy is reduced to a value close to the energy for grain boundary diffusion. It is concluded that the dominant mechanism for high strain rate superplasticity in metal matrix composites is grain boundary sliding accommodated by a process controlled by grain boundary diffusion.

2:50 PM INVITED

THE NATURE OF HIGH-STRAIN-RATE SUPERPLASTICITY IN ALUMINUM MATRIX COMPOSITES: *R. Kaibyshev*[1]; [1]Institute for Metals Superplasticity Problems RAS, Ufa 450001 Russia

The phenomenon of high-strain-rate superplasticity is considered in the PM2014-20%Al2O3p metal matrix composites and is compared with superplastic behavior of its monolithic aluminum alloy PM2014 in detail. The optimum strain rates for superplasticity in the MMC tend to be considerably higher than in its matrix alloy. In addition, there is a difference between microstructural evolution during superplastic deformation in the composite and its matrix alloy. It was found that structural evolution of the aluminum matrix composite is unusual during superplastic deformation. At the initial stage of superplastic flow, the number density of lattice dislocations increases but subsequently decreases with further strain in the stable flow stage. Both at low strain rates in region I and at high strain rates in region III the density of lattice dislocations is higher than that in the optimum strain rate range of superplasticity. In the aluminum alloy the increase of strain rate leads to monotone increase of lattice dislocation density.

3:15 PM

TEXTURE CHANGES DUE TO HIGH STRAIN RATE DEFORMATION IN MECHANICALLY ALLOYED ALUMINUM: *Z. Jin*[1]; T. R. Bieler[2]; [1]Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545; [2]Michigan State University, Department of Materials Science and Mechanics, East Lansing, MI 48824

Texture analysis is a powerful technique for investigation of superplastic deformation. High strain rate superplastic (HSRS) deformation and texture evolution in mechanically alloyed aluminum In90211 were investigated. Texture evolution was compared in specimens deformed at 475°C and 0.0001 s-1 (region I), 2.5 and 75 s-1 (region II) and 330 s-1 (region III). Initial (undeformed) textures consisted of two deformation texture skeletons similar to normal FCC rolling textures. These orientations were roughly maintained throughout deformation, but the changes in sharpness and rotations from the initial orientation differed significantly in the three specimens. The differences indicate that in region III, the dominant contribution to deformation is from dislocation slip; in region II, the superplastic deformation occurs by grain boundary sliding (GBS), dislocation slip, and recrystallization (at larger strains); and in region I, GBS is the dominant deformation mechanism, and the rotations occur in the opposite direction as slip in region III. These results suggest that an optimal balance between dislocation slip and GBS is a requirement.

3:35 PM Coffee Break in Exhibit Hall

3:50 PM INVITED

FRACTURE MECHANISMS IN HIGH STRAIN RATE SUPERPLASTIC ALUMINUM MATRIX COMPOSITES: *H. Iwasaki*[1]; T. Mori[1]; M. Mabuchi[2]; K. Higashi[3]; [1]Himeji Institute of Technology, College of Engineering, Department of Materials Science and Engineering, Hyogo 671-22 Japan; [2]National Industrial Research Institute of Nagoya, Nagoya 462 Japan; [3]Osaka Prefecture University, College of Engineering, Department of Metallurgy and Materials Science, Osaka 593 Japan

Recently it was pointed out that the deformation mechanisms of high strain rate superplasticity are drastically changed by the presence of liquid phase for aluminum matrix composites with discontinuous reinforcement materials. In the present paper, effects of liquid phase on the fracture mechanisms are investigated for high strain rate superplastic Si3N4p/Al-Cu-Mg (2124) composite. In a solid state including no liquid, cracks developed perpendicular to the tensile axis by plastic tearing because of the stress concentrations at the interfaces. However, no cracks were formed in a state including a small volume of liquid, indicating that the stress concentrations are relaxed by a liquid phase. On the other hand, cracks were again formed in a state including too much liquid. A thin liquid phase is required to limit decohesion at liquid boundaries.

4:15 PM

HIGH STRAIN RATE SUPERPLASTICITY OF 1n90 ALUMINUM, TiC AND A1N PARTICULATE REINFORCED ALUMINUM COMPOSITES: *T. Imai*[1]; S. Kojima[2]; D. Jiang[3]; M. Jianfu[3]; G. L'Esperance[4]; B. Hong[4]; [1]National Industrial Research Institute of Nagoya, Nagoya 462 Japan; [2]Nagoya Municipal Industrial Research Institute, Nagoya Japan; [3]Harbin Institute of Technology, Harbin 150001 P.R. China; [4]Ecole Polytechnique de Montreal, Montreal, Quebec H3C 3A7 Canada

The purpose of this work is to study superplastic characteristics and the microstructure variation during high strain rate superplastic (HSRS) deformation for 1N90, pure aluminum, TiC/1N90 and TiC/2014 Al composites and also to reveal the deformation mechanism of the HSRS. 1N90 pure aluminum alone exhibits the m value of 0.30~0.47 and the total elongation of 300-500% at strain rate of 0.01s-1 and at 893~913K. 1N90 pure aluminum consists of grains of about 10~20 mm and subgrain of 0.6~1.0 mm and TiC/2014 Al composites indicate an m value of about 0.3 and the total elongation of 200~300% at strain rate of 0.8s-1 and at 833K. The composite includes the grain of 2 mm and also subgrain of 0.6~1.0 mm. Therefore, it is thought that subgrain sliding might contribute to HSRS in addition to fine grain boundary sliding and interfacial sliding at semi-solid phase.

4:35 PM

CAVITATION BEHAVIOR OF A 2124/Al/SiCp COMPOSITE DURING HIGH STRAIN RATE SUPERPLASTICITY: *G. H. Zahid*[1]; R. I. Todd[1]; P. B. Prangnell[1]; [1]Materials Science Centre, UMIST, Manchester M1 7HS UK

A powder processed 2124Al/SiCp composite has been produced which exhibited high strain rate superplasticity after warm rolling. The superplastic properties achieved were comparable to those obtained by other workers in materials which were given special thermomechanical treatments or hot extruded. A total elongation of more than 400% was achieved at a strain rate of 8.3 °° 10-2 s-1 before the specimen fractured due to cavitation. Constant strain rate tests were carried out to study the cavitation behavior of the composite. Metallographic examination revealed that cavities started nucleating before a strain of 0.5 was reached. Most of the cavities were observed in areas with a lesser number of SiC particles and larger grains which is contrary to the observations of cavities forming in clustered regions during fracture of MMCs. This remained the case for the whole range of strain up to fracture. A considerable amount of strain hardening also occurred during constant strain rate tests. These observations suggest that premature fracture occurred due to the presence of coarser grains, resulting from an uneven distribution of reinforcement. A detailed quantitative study of cavitation behavior has been carried out.

ZINC-BASED STEEL COATING SYSTEMS: PRODUCTION & PERFORMANCE: Session IV - New Coatings and General Galvanizing

Sponsored by: Structural Materials Division, Ferrous Metallurgy Committee

Program Organizers: Frank E. Goodwin, Int'l Lead Zinc Research Org., PO Box 12036, Research Triangle Park, NC 27709-2036; Michelle DuBois, Cockerill Sambre, Quai Du Halage 10, Flemalle B-4400 Belgium; Jong-Sang Kim, Pohang Iron & Steel Co. Inc., Kwangyang Research Labs, Chonnam 544 090 Korea; Josef Faderl, Voest Alpine Stahl Linz, Turmstrasse 45, PO Box 3,A-4031 Linz, Austria; Eduardo A. Silva, USS Technical Center C-20, Monroeville, PA 15146

Tuesday PM Room: Centro Room B
February 17, 1998 Location: Convention Center

Session Chair: Eduardo A. Silva, USS Technical Center C-20, Monroeville, PA 15146

2:00 PM

TEXTURE CHARACTERIZATION OF GALFAN(R) COATED STEEL SHEETS (PRELIMINARY RESULTS): *J. Wégria* [1]; M. Dubois[2]; M. J. Philippe[3]; C. Esling[3]; [1]UM Research, Olen Belgium; [2]Cockrell-Sambre, Flémalle Belgium; [3]Metz University, LETAM, Metz France

In this preliminary study, we have analyzed the influence of the substrate thickness as well as the coating thickness on the texture of the latter. The work was carried out on industrial samples. The obtained results show that the studied parameters influence the intensity and the spread of the texture as well as the microstructure of the coating. The results are discussed in terms of coated steel formability.

2:20 PM

TENSION BEND STAINING OF PREPAINTED GALVALUME: *T. W. Cape*[1]; D. W. Gomersall[1]; S. G. Denner[1]; [1]National Steel Corporation, Trenton, MI

Galvalume, a hot dip coating on steel with a composition of approximately Zn-55% Al-1.6% Si, has demonstrated remarkable perforation corrosion durability and has gained widespread acceptance for the construction of metal buildings. One drawback to the expanded use of prepainted Galvalume has been the premature cosmetic corrosion experienced at roll formed tension bends. The causes of this straining are related to fractures in the metallic coating which are not 'bridged' with the use of certain classes of paint systems. This paper will review work performed to minimize metallic coating fracture through the use of lower silicon contents and the development and use of paint systems which enable the bridging of these fractures.

2:40 PM

INFLUENCE OF TYPICAL PROCESS PARAMETERS ON THE MICROSTRUCTURE OF GALFAN COATINGS: *F. Hinterberger*[1]; W. Maschek[1]; J. Faderl[1]; [1]Voest-Alpine, Linz Austria

Galfan coated steel sheet is a common product on today's continuous hot dip galvanizing lines. Many features like wetting behavior in the Galfan bath, denting, cell size and formation of proeutectic zinc dentrites in Galfan coatings have been studied in the past but a general idea about the solidification sequence in the Galfan coating is still missing. In numerous laboratory trials samples were produced by changing the precleaning condition of the steel substrate, the bath temperature, the Al content in the bath, the coating thickness and the cooling after dipping. The macroscopic visible cell size but also differences in the formation of proeutectic zinc dentrites were systematically evaluated. Furthermore some observations on the different eutectic structures (rod and lamellea) and on the build up of the Galfan cell were made. By studying the microstructure no relation between the formation of the proeutectic zinc dendrites and the macroscopic visible eutectic cell size was found. The eutectic cell itself is build up of many subcells with different eutectic morphologies. Both, the formation of the proeutectic zinc dendrites and eutectic cell can be controlled to a different degree by varying galvanizing parameters. Also a strong influence of impurities in the Galfan bath was found. Altogether this work should be a further contribution to understand the solidification of Galfan coatings.

3:00 PM

HIGH SPEED ZINC GALVANIZING FROM A METHANESULFONATE SOLUTION: Reinhard Wormuth, Ph.D.[1]; *Nicholas M. Martyak, Ph.D*[2]; Allen Jones, Ph.D[3]; [1]Thyssen Krupp Stahl AG, Dortmund D-44120 Germany; [2]Atochem North America, King of Prussia, PA 19046; [3]Atotech, USA, Rock Hill, SC 29731

A new development in high speed electrogalvanizing based on zinc methanesulfonate (ZnMSA) shows promise in producing smooth zinc deposits at current densities up to 300 A/dm^2. The conductivity of the ZnMSA solution is superior to that of zinc sulfate over wide zinc metal, pH and temperature ranges. Rotating cathode studies investigated the effects of steel strip speed, solution flow rate, pH, zinc metal and additive concentrations on the surface roughness, deposit structure and orientation of the zinc coatings. The deposits were characterized using scanning electron and atomic force microscopy, x-ray diffraction and surface profilometry. Results from the rotating cathode studies comparing a standard zinc sulfate electrolyte to the new zinc methanesulfonate solution will be discussed.

3:20 PM

ELECTRO-PLASMA PROCESSING - A NEW, COST-EFFECTIVE, CLEANING AND COATING TECHNOLOGY: *E. I. Meletis*[1]; [1]Louisiana State University, Mechanical Engineering Department, Materials Science and Engineering Program, Baton Rouge, LA 70803

Electro-plasma processing (EPP) is a novel technology that was originally developed in Russia and recently acquired by MTI that plans to introduce it to the industry. The new technology can overcome many of the drawbacks of existing methods, both for the cleaning and for metal-coating steel. Experiments have demonstrated that EPP can (1)efficiently remove mill-scale and other contaminants from steel surfaces at low energy consumption (1-2.5 kW/m^2) by electrolytic cleaning that can also result in a desired surface topography (smooth or rough) and (ii) produce metal coatings of almost any desired alloy composition. The speed of coating can greatly exceed that of electroplating and can match that of dipping. In this paper, the fundamental processes involved in the cleaning and coating stages during EPP are summarized and the latest results on Zn-Al coatings deposited by this technique are presented. The coatings were characterized in terms of morphology and adhesion to the steel substrate, and cross sectional composition utilizing metallographic, SEM and EDX techniques. Corrosion behavior and resistance were studied by conducting several tests varying from anodic polarization to salt spray testing. Comparative corrosion studies were also performed on galvanized steel specimens to demonstrate the capability of the EPP technique as an emerging new technology.

3:40 PM

A NEW INTERPRETATION OF THE SANDELIN EFFECT: *Y. Leprêtre*[1]; J. M. Mataigne[1]; [1]SOLLAC, BP 30109, Montataire France

This paper provides a consistent an original interpretation of the Sandelin effect, based on the fundamental principles of thermodynamics and diffusion in ternary systems. Its conclusions are based on a bibliographical review, and are justified by the results of a similar analysis performed about the inhibiting effect of aluminium (see an other paper also presented at TMS). According to the most reliable results of the literature, the proposed interpretation assumes that, whatever the silicon content in iron, the coating is initially statified like that obtained on pure iron (incubation period). However, because of the very low solubility of silicon in ζ the balance between the silicon fluxes within the different layers cannot be reached, and silicon conception in δ_1, that can then get an equilibrium with liquid zinc. This leads to the formation of multiphase domains involving the liquid and to a linear

growth law. Satisfactorily, this new interpretation shows that invoking the precipitation of FeSi particles to explain the Sandelin effect is needless, as the hypo- and hypersandelin behaviors, either due to silicon or phosphorus, can be understood according to the shape of the diffusion paths in the ternary diagram, and to the localization of the formation of the ζ precipitates they require.

4:00 PM

INFLUENCE OF THE SUBSTRATE CRYSTALLOGRAPHIC TEXTURE ON THE SANDELIN EFFECT: *J. Wêgria*[1]; R. Sokolowski[2]; M. J. Philippe[3]; C. Esling[3]; [1]UM Research, Olen Belgium; [2]UM Zinc, Auby France; [3]Metz University, LETAM, Metz France

The effects of substrate crystallographic orientation in galvanizing of binary FE-Si were investigated (Si additions ranging from 0.10% to 0.45%). The following substrate orientations were studied: (100),(110) and (211). The results suggest a relationship between galvanizing reactivity and substrate texture. This study was extended to hot rolled industrial steels with similar Si-content. The obtained results confirm a crystallographic orientation effect: the curve of the variations, in function of Si-content, of the (100) diffraction intensity has a shape which is quite similar to the Sandelin curve. This feature is observed only for the (100) planes. The orientation of steel surface grains is responsible for the nucleation and the growth of the Fe-Zn intermetallic compounds. The influence of the interface energy on the intermetallic compounds formation has already been suggested: it is proposed that the variation of this interface energy is related to the substrate texture.