PMP 2000

	Monday-November 6		Tuesday-November 7		Wednesday-November 8	
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Parc Ballroom 1	Plenary Session					
Parc Ballroom 3			Aqueous Processing I	Iron & Steelmaking II	Aqueous Processing II	Iron & Steelmaking III
Dante		Electronic, Magnetic & Photonic Materials I	Electronic, Magnetic & Photonic Materials II	Powder Preparation & Processing I	Powder Preparation & Processing II	Powder Preparation & Processing III
DaVinci 1		Resourses & Its Related Environment	Thin Films & Coatings I	Thin Films & Coatings II	Thin Films & Coatings III	
DaVinci 2 & 3		Composite Materials I	Composite Materials II	Composite Materials III	Composite Materials IV	
Cervantes		Non-ferrous Alloys & Light Metals I	High Temperature Materials	Non-ferrous Alloys & Light Metals II	Non-ferrous Alloys & Light Metals III	
Michaelangelo		Waste Management I	Electrolytic Processing I	Waste Management II	Electrolytic Processing II	Waste Management III
Raphael		Iron & Steelmaking I	Rare Metals	Solidification Processing I	Solidification Processing II	
Rubens		Control & Analysis in Materials Processing I	Copper, Nickel, Zinc, Lead and Tin I	Control & Analysis in Materials Processing II	Copper, Nickel, Zinc, Lead and Tin II	

2000 PMP II: Second International Conference on Processing Materials for Properties Technical Program

Plenary

Monday AM Room: Parc Ballroom I
November 6, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Brajendra Mishra, Colorado School of Mines, Metall. & Matls. Engr., Golden, CO 80401 USA; Chikabumi Yamauchi, Nagoya University, Grad. Sch. of Eng., Dept. of Matls. Sci. and Eng., Nagoya 464-8603 Japan

8:30 AM Welcoming Remarks: Brajendra Mishra

8:45 AM
Nonferrous Metals Production: Advances in Process Technology and Environmental Protection: *Hong Y. Sohn*[1]; [1]University of Utah, Metall. Eng. Dept., 135 S 1460 E, Rm. 412, Salt Lake City, UT 84112-0114 USA

The nonferrous production industry has gone through a remarkable transformation in recent years in terms of developing and adopting new technologies. Technology innovations have largely been driven by the need for greatly improved environmental protection in addition to increased productivity. One of the most notable aspects of the new technologies is their ability to reduce the emission of sulfur and other pollutants to extremely low levels. The current industry trends towards more environmentally friendly technologies are bringing about additional benefits of developing more efficient, technically advanced, and continuous processes in closed reactor vessels. Key aspects of these modern trends include the use of increased oxygen enrichment in the process gas, better process control, reduced manpower requirements, and plant operations with more skilled personnel. Computer simulation and modeling of complex processes have become more reliable, realistic, and thus increasingly useful in metal production. Some of the notable examples are reviewed. Finally, the need and advantages for value-added production in the nonferrous industry are discussed.

9:30 AM
Soft Solution-Processing for Advanced Materials-Concept, Realization, and Perspective: *Masahiro Yoshimura*[1]; [1]Tokyo Institute of Technology, Matls. and Struct. Lab., Ctr. for Matls. Des., 4259 Nagatsuta, Midori, Yokohama, Kanagawa 226-8503 Japan

Based upon the fact that almost all materials and energies in nature have been cycled essentially by water cycle on the earth, we can conclude that fabrication and processing of artificial (industrial) materials must be based upon water (solution) flow to minimize the loads to the environment. According to above consideration, we are proposing Soft Solution-Processing (SSP) which means direct (preferably than indirect) fabrication of inorganic materials with desired composition, structure, properties, shape, size, location from aqueous solution at a mild temperature and pressure condition. The SSP is more environmentally friendly but more difficult than "dry" processing using gas(es), vapor(s), molecule(s), cluster(s), atom(s), ion(s), in vacuum gas and/or in plasma. This is because that species in a solution has been stabilized by solvation (hydration in water) energy, therefore they have smaller driving force (ΔG) for the reaction but require higher activation energy (ΔG^*) than highly energetic species in dry processings. However, SSP becomes possible when we could choose certain systems and conditions. For example, we have succeeded to fabricate thin/thick films of $BaTiO_3$, $SrTiO_3$, (Ba, Sr, Ca)WO_4, $LiCoO_2$, $LiNiO_2$ on various substrates in alkaline solutions at 100-200°C, sometimes even at RT, with/without electrochemical (anodic) oxidation. They are well-crystallized and deposited on the substrates, thus no post-heating has been required in contradistinction to so-called "sol-gel" coated and electrophoretically deposited films. Layered films have also been fabricated using a flow-cell apparatus. Future perspective including direct patterning will be presented.

10:15 AM Break

10:30 AM
The Development of Graded Thin Film/Coating Systems: *John J. Moore*[1]; Brajendra Mishra[1]; D. Zhong[1]; T. Dennin[1]; E. Hixson[1]; S. Challappalli[1]; [1]The Colorado School of Mines, Adv. Coat. and Surf. Eng. Lab., Golden, CO 80401 USA

The paper will discuss the design, processing and the resultant properties of graded thin films and coating systems that are needed to meet specific performance criteria. Three examples of graded films and coatings, i.e., Cr-N, $MoSi_2$-SiC, and Ti-TiN-TiAlN-NiAl, will be used to demonstrate the philosophy adopted in the current research programs in the Advanced Coatings and Surface Engineering Laboratory (ACSEL) at the Colorado School of Mines. The Cr-N graded thin film system is currently being developed for metal forming applications, the $MoSi_2$-SiC graded coating is being developed for high temperature oxidation resistance of refractory metals, while the Ti-TiN-TiAlN-NiAl graded coating is being developed for glass molding dies.

11:15 AM
Future Materials Requirements for High Energy-Intensity Production of Aluminum: *Barry Welch*[1]; [1]University of Auckland, P.O. Box 42-240, Coates Ave., Orakei, Auckland 1130 New Zealand

Abstract text unavailable.

Non-ferrous Alloys and Light Metals - I

Monday PM Room: Cervantes
November 6, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Barry J. Welch, University of Auckland; Takashi Nakamura, Tohoku University, Instit. for Adv. Matls. Process.

2:00 PM Keynote
Use of Refractory Hard Cathodes to Reduce the Energy Consumption in Aluminium Smelting: *Mark P. Taylor*[1]; [1]Comalco Aluminium Limited, GPO Box 153, Brisbane, Queensland 4001 Australia

Over 20 million tonnes of installed aluminium smelting capacity exists world-wide, with smelter energy consumption in the range 14-17 kWh/kg aluminium. In terms of the reduction cell technology itself, the best sustained energy consumption is just below 13DC kWh/kg, or 50% energy efficient. For 15 years Comalco Ltd. in Australia has been engaged in developing and testing reduction cells which use a refractory hard cathode, composed of a TiB_2/carbon composite coating over a graphitised carbon cathode. This technology has the potential to reduce energy consumption significantly while simultaneously increasing the life of the cells, which is currently limited by wear rate of the conventional carbon cathodes used throughout the world. This paper presents the latest results from Comalco's test cells, and analyses the implications of the technology's commercialisation in terms of the impact on greenhouse gas generation through reduced power requirements, and on the life cycle of the metal for automotive applications.

2:30 PM

Influence of Heat Treatment Conditions on the Mechanical Properties of New Rheocasting Aluminium Parts: *Helmut Kaufmann*[1]; Peter J. Uggowitzer[2]; [1]Leichtmetall Kompetenzzentrum Ranshofen, Ranshofen A-5282 Austria; [2]ETH Zurich, Instit. for Metall., Sonneggstrasse 3, Zurich CH-8092 Switzerland

New Rheocasting is a novel route of semi-solid casting, in which the globular semi-solid precursor material is produced by controlled cooling of only slightly superheated melts in conventional steel crucibles. The resulting semi-solid slugs are placed into the sleeve of a vertical Squeeze Casting Machine and then filled into steel dies. Solidification occurs under hydraulic pressures up to 100 MPa. Due to the reduced cost of precursor material, reduced investment cost and easier recycling, the New Rheocasting process offers cost reductions of up to 25% over conventional semi-solid casting routes. The New Rheocast products are pore free, pressure tight, weldable and heat treatable castings. In the present work the New Rheocasting Process is introduced and the influence of heat treatment on mechanical properties of the New Rheocast alloy A356 is discussed. The alloy A356 is cast under ideal New Rheocasting conditions into a step die on an UBE HVSC 350 Squeeze Casting machine. As?cast, T5 and T6 heat treatment conditions with variations in solutionizing and aging time/temperature parameters are applied and the samples are tested by tensile testing. Alternatively, the same heat treatment conditions are applied to conventional Squeeze Casting parts and compared with New Rheocasting. Questions of potential cost savings due to modified heat treatment conditions are also addressed in this paper.

2:55 PM

Synthesis and Thermal Properties of Ti-Based Amorphous Alloys with a Large Supercooled Liquid Region: *Pee-Yew Lee*[1]; Nan-Fong Hsu[1]; C. Y. Ma[2]; [1]National Taiwan Ocean University, Matls. Eng. Dept., 2, Pei-Ning Rd., Keelung, Taiwan 202 Taiwan; [2]Chung-Shan Institute of Science & Technology, Matls. and Electro-Optics Rsch. Div., P.O. Box 90008-8-4, Lung-Tan, Tao-Yuan, 325 Taiwan

Various techniques have been developed to obtain amorphous alloys, but most of the research effort and industrial interest is centered on the different implementations of the rapid solidification and solid state reaction. The techniques to synthesize amorphous alloys via solid state reaction include hydrogenation, multilayer interdiffusion, and mechanical alloying (MA). As previous investigations demonstrated, amorphization by mechanical alloying has been observed for a variety of binary alloy systems. The product material of mechanical alloying is in powdered form and is suitable for compaction and densification in many shapes. In this paper, we report the fabrication of Ti-Ni-Cu-B-Si amorphous powders by mechanical alloying technique. According to the results, after 5~7 hours of milling, the mechanically alloyed powders were amorphous at compositions with (x y) equal to 20~40%. For the compositions with (x y) larger than 45% or smaller than 10%, the structure of ball-milled powders is either a partial amorphous single phase or coexistent partial amorphous and crystalline phases. The thermal stability of the amorphous powders was also investigated by differential thermal analysis. As the results demonstrated, several amorphous powders were found to exhibit a wide supercooled liquid region before crystallization. The glass transition process in the Ti64Ni30Si4B2 is found to be a kinetically modified thermodynamics phase transformation process.

3:20 PM

Titanium Powder Production by Reactive Molten Salt as a Reductant: *Tetsuya Uda*[1]; *Toru H. Okabe*[2]; Yoshio Waseda[1]; [1]Tohoku University, Rsch. Ctr. for Metall. Process Eng., Instit. for Adv. Matls. and Process., 2-1-1 Katahira Aoba-ku, Sendai, Miyagi Pref. 980-8577 Japan; [2]Tohoku University, Instit. of Adv. Matls. and Process., 2-1-1 Katahira Aoba-ku, Sendai, Miyagi Pref. 980-8577 Japan

Halide salt has been tested as a reductant for producing titanium powder directly from TiCl4. From the results of thermodynamic investigation for several rare earth chloride systems, DyCl2 was found to be appropriate for the reduction of TiCl4, and it can be used instead of the conventional metallic reductant. When TiCl4 was fed into molten salts containing DyCl2 at 1073K, titanium powder of several ten micron meters in diameter was obtained. In contrast to the sponge titanium produced by the conventional Kroll process, the present new process using halidothermic reduction appears to be suitable for producing metal powder. From the thermodynamic point of view, the resultant DyCl3 can be reduced to DyCl2 by magnesium. These particular are possible way of the production of titanium powder production directly from TiCl4 as well as the development of a new continuous titanium reduction process by utilizing low valence ions as reductants.

3:45 PM Break

4:00 PM

Modeling of the Reactive Immersion of Ceramic Particles into Molten Aluminum Alloy: *Kevorkijan M. Varuzan*[1]; [1]Independent Researcher, Lackova 139, Limbus 2341 Slovenia

In this paper, the linear time dependence and the exponential temperature dependence of the volume fraction of ceramic reinforcement dispersed in matrix has been addressed theoretically and has been verified experimentally. It was demonstrated that the rate of reactive wetting Ww depends on exergonic nature of an interfacial reaction, the kinetics of the performed interfacial chemistry and the efficiency of system to use the energy released by an interfacial chemical reaction for the formation of new surface in melt. It was found that the rate of the reactive wetting does not necessarily correspond to the rate and the level of an exergonic nature of performed interfacial chemical reaction. However, further understanding of the ability of system to convert the energy released by performed interacial reaction to the energy of new surface created in melt becomes necessary in order to optimize the process of immersion.

4:25 PM

Properties of AlMgSi1 Wrought Alloy Components Produced by Means of the NRC-Process: *Heimo Wabusseg*[1]; Giancarlo

Gullo[1]; Helmut Kaufmann[2]; Peter J. Uggowitzer[1]; [1]Swiss Federal Institute of Technology ETH, Instit. of Metall., ETH Zentrum, Sonneggstrasse 3, Zurich CH-8092 Switzerland; [2]Centre of Competence on Light Metals, LKR, P.O. Box 26 Lambrechtshausenerstrasse, Ranshofen A-5282 Austria

By taking advantage of the thixotropic properties of AlMgSi1 wrought alloy 4-step parts were casted by using the NRC-process. This process produces a semi-solid slurry including globular chrystals directly from molten metal without cost-intensive stirring technique. Optimal casting temperatures for the formation of the semi-solid slug have been determined and correlated to the characteristic microstructural parameters such as grain size, shape factor and contiguity volume. The thixotropic shaping to the 4-step component was performed at different temperatures of the semi-solid slug, i.e. the solid fraction of the slurry was varied within a relatively wide range. By means of tensile tests it was shown, that excellent mechanical behaviour can be achieved by applying optimal NRC-processing conditions.

Composite Materials - I

Monday PM Room: DaVinci 2&3
November 6, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: V. K. Vijayalakshmi, N.M.K.R.V. College for Women; Hideo Nakae, Waseda University, Dept. of Matls. Sci. and Eng.

2:00 PM Keynote
Fracture in Ductile-Brittle Graded Composites: *I. E. Reimanis*[1]; [1]Colorado School of Mines, Metall. and Matls. Eng. Dept., Golden, CO 80401 USA

Theoretical and experimental work on the fracture of compositionally graded composites is first reviewed. While much progress has been made in establishing predictive models for fracture and deformation of graded materials, certain theoretical aspects are lacking, and furthermore, there are only a few examples of experimental verification. In this work, step-wise graded Ni/Al_2O_3 and Cu/W composites have been formed for model fracture tests. The elastic response, fracture, and deformation of these systems is examined using a variety of techniques, including compression, wedge loading, four-point bending, crack path observations, phase shifted optical moire interferometry and the finite element method (FEM). The relative contribution to fracture of several factors, including elastic and plastic mismatch, thermally induced residual stresses, and the inherent fracture energy of each of the constituents and composite layers is examined. Experimental and FEM simulation results of fracture and deformation behavior are discussed in terms of these factors in addition to established fracture models for composites, metals and ceramics.

2:30 PM
Property/Processing Relationships for Continuously Fabricated MMC Fiber-Reinforced Wire: Shannon L. Sampson[1]; Joseph T. Blucher[1]; *Jacqueline A. Isaacs*[1]; [1]Northeastern University, Mech. Ind. and Manu. Eng., 334 Snell Eng., 360 Huntington Ave., Boston, MA 02115 USA

A liquid metal pressure infiltration technique for continuous fabrication of continuous fiber reinforced metal matrix composites has been developed to produce wire with diameter of approximately 1.3 mm. Lengths of 350m have been routinely produced, with production rates of 8 m/min. Until now, it had been impossible to produce continuous long pieces of fiber reinforced MMC's. This novel technique solves prior difficulties by quickly transporting the fibers through a melt at a high pressure, while preventing liquid metal from escaping the infiltration vessel. Continuous production of fiber reinforced MMC's could lead to reduced manufacturing costs, which could increase their marketability. The composites are fabricated with an aluminum metal matrix with approximately 50% continuous aluminum oxide fibers. Results show that the MMC wire is well infiltrated and exhibits excellent tensile properties. In general, increased production rates show improved ultimate tensile strength and smaller grain sizes for all MMC wires investigated.

2:55 PM
Charactarisation of Corrosion Properties of ZA-27/Quartz Metal Matrix Composites: *P. V. Krupakara*[1]; Jayagopal Uchil[2]; Trivikram[3]; [1]RV College of Engineering, Dept. of Chem., Bangalore, Karnataka 560059 India; [2]Mangalore University, Dept. of Matl. Sci., Mangalagangothri, Mangalore, India; [3]RV College of Engineering, Dept. of Mech. Eng., Mysore Rd., Bangalore 560059 India

This paper deals with the corrosion characterization of quartz particulate reinforced ZA-27 alloy metal matrix composites. Quartz particulates of size 50-80 microns are used as reinforcement. Composites containing 2, 4, and 6% by weight of quartz and unreinforced matrix were tested. MMC's are prepared by liquid melt metallurgy technique using vortex method. Specimens are prepared according to ASTM standards. They are tested in both as cast and heat treated condition. The corrodant used in 1N hydrochloric acid. As the quartz content increased the composite was seen to become more resistant to corrosion. Heat treatment at 340°C for hours ranging from 4 to 12 hours also enhanced the corrosion resistance of the composites as well as unreinforced matrix material. Explanation to this phenomenon is attempted.

3:20 PM
Oxidation Behaviour of Aluminium-6061/Hematite Particulate MMC's at Elevated Temperatures: *S. C. Sharma*[1]; N. Shanmukha[1]; M. Krishna[1]; [1]R.V. College of Engineering, Dept. of Mech. Eng., Rsch. and Dev., Bangalore, Karnataka 8600535 India

The oxidation behavior of an aluminium composite reinforced with 0, 2, 4, and 6% by weight of hematite particles at 200 to 500°C has been investigated. With in the experimental temperature range a parabolic weight gain was observed. The weight gain as a function of oxidation time becomes linear after an initial period. On oxidation formation, an oxide scale was found whose morphology depends on temperature, cooling rate and sub-scale formation at the interface between the matrix and reinforcement. The morphology and phase content of the product of oxidation were characterized by EDAX. Detailed analyses by SEM showed that the oxide scales were not homogenous through out, but exhibited several layers, which differ in microstructure and composition with increase in thickness. The presence of hematite particulate reinforcement has very little effect on the oxidation behavior. The interface oxidization rate was found to be higher than the other regions. The oxidation products were either metallic oxides or ceramic oxides. The exposure time of specimens to oxidation was 1000 minutes at various temperatures. In the present work key principles of alloy oxidation were discussed. Also the paper emphasizes on various reactions taking place and the activation energies involved in the oxidation reactions.

3:45 PM Break

4:00 PM

Fabrication and Mechanical Properties of the SiC Fiber Reinforced Matrix Composites: *Makoto Yoshida*[1]; Kenya Nagahisa[1]; Kazuya Kitatani[1]; Naoto Ohta[2]; Toshihiro Ishikawa[3]; Jin Pan[1]; Gen Sasaki[1]; Hideharu Fukunaga[1]; [1]Hiroshima University, Mech. Eng. Dept., 1-4-1 Kagamiyama, Higashi, Hiroshima, Hiroshima Pref. 739-8527 Japan; [2]Toyo Tanso Company Limited, Eng. and Dev. Ctr., 2181-2 Nakahime, Ohnohara-Cho, Mitoyo-Gun, Kagawa Pref. 769-1612 Japan; [3]Ube Industries Limited, Ube Rsch. Lab., 1978-5 Kogushi, Ube City, Yamaguchi Pref. 755-8633 Japan

In order to improve anti-oxidization of the SiC fiber reinforced CMC's the oxide matrices with various CTE, Al2O3-18.5 at% YAG (CTE:5~8ppm), Y2SiO5 (4.7ppm) and MAS glass (2~5ppm) were used to hot press with Tyranno-SA SiC fiber at 1923 K or 1473 K (MAS). The preform for hot press was prepared by infiltrating the slurry into the fiber. The microstructure of the obtained materials and chemical reaction between fiber and matrices were investigated. The bending strength (JIS R1601) and the energy to failure (JCRS201) were also examined. As to the monolithic Al2O3-YAG oxide material, the bending strength and the energy were 680 MPa and 50 J/m2 respectively at room temperature. The energy was improved by using graphite coated SiCf up to 10,000 J/m2. It is necessary in this material to reduce the interfacial bonding strength by an adequate coating on the fiber.

4:25 PM

Study on Oxidation Resistance of High Temperature of Electrodeposited RE-Ni-W-P-SiC Multiple Functional Composite Materials: *Guo Zhongcheng*[1]; Yang Xianwan[1]; Zhai Dacheng[1]; Zhu Xiaoyun[1]; [1]Kunming University of Science and Technology, Dept. of Metall., Kunming, Yunnan 650093 China

Oxidation resistance of high temperature of RE-Ni-W-P-SiC multiple functional composite materials has been studied. Results show that relationship between weight of oxidized films of pure Ni, Ni-W-P, Ni-W-P-SiC and RE-Ni-W-P-SiC coatings and time of oxidation is mixture curves, i.e. composite principle of linear and curve. The principle of growth of oxidized films is approximate linear equation when time of oxidation is below 60 minutes. However, the principle is shown in the form of power function equation when time of oxidation is over 60 minutes. Rates of oxidation of four coatings are pure Ni>Ni-W-P>Ni-W-P-SiC>RE-Ni-W-P-SiC. The change of weight of oxidized films of Ni-W-P, Ni-W-P-SiC and RE-Ni-W-P-SiC coatings is raised in the form of exponential function with increasing temperature of oxidation. Compared with Ni-W-P-SiC composite coating, oxidation resistance of RE-Ni-W-P-SiC coating is increased 2-3 times. Cross sections and X-ray diffractions of the coatings show that RE-Ni-W-P-SiC Composite coating has better oxidation resistance of high temperature.

Resources and Its Related Environment

Monday PM Room: DaVinci 1
November 6, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: D. Javernick, Los Alamos National Laboratory; Yoshiaki Umetsu, Tohoku University; Tetsuya Shoji, The University of Tokyo

2:00 PM Keynote

Steel Making Technology for Environmental Protection: *Kiyoshi Shibata*[1]; Jun-ichi Hayashi[1]; [1]Nippon Steel Corporation, Adv. Tech. Rsch. Labs., 20-1 Shintomi, Futtsu, Chiba 293-8511 Japan

Steel making technology posses strong potentials for environment protection in three directions. One is, of course, pollution control in steel making process itself. Japanese steel industry has cut down various type of enission drastically since 1970's. Secondly, those process technologies can be applied to reduce environmental burden in other industries. One of the successful examples is municipal waste treatment facility. Thirdly, developing advanced steel products, such as high strength steel for automobile body, can contribute to improve the environmental performance during their use, based on life cycle thinking. Some examples of the successful development and future task will be discussed.

2:30 PM

Economic Growth and Relationships between Key Metals and Energy Consumption: *Takashi Nishiyama*[2]; Jie Liu[2]; Toshihide Ito[1]; [1]Kansai University, Dept. of Inform., Osaka 569-1095 Japan; [2]Kyoto University, Grad. Sch. of Energy Sci., Kyoto 606-8501 Japan

Trends in the consumption of energy and key metals and the underlying determinants of these trends vary among human societies, and in their evolution from agricultural to industrial and subsequently, to service societies. The disparity between economic development and relationships among aluminum, copper, lead, zinc crude steel and primary energy consumption were analyzed based on statistical data on both a global and regional basis. According to the growth rates of energy and essential metal demand, economic development can be divided into three stages. In the first stage, the growth rates of both energy and key metals are minimally supported by non-mechanized agriculture and light industry. Most developing countries such as India are included in this stage. The second stage, chiefly based on heavy industry, is characterized by very high growth rates of key metals and by high growth rates of energy. China and Korea are in this second stage. Korea will soon move into the third stage, while China is expected to continue its strong expansion in the near future. In the third stage, manufacturing capabilities shift from heavy to service industries. The service sector is less metal-intensive than the heavy industry sector, whereas the trend of energy consumption continues to increase at high rates. Japan has reached the third stage.

2:55 PM

A Macro Model for Steel Usage Pattern in Japan using a Pollution Balance Model: *Keiji Kakudate*[1]; Yoshihiro Adachi[2]; Toshio Suzuki[2]; [1]The University of Tokyo, Grad. Sch., 7-3-1, Hongo, Bunkyo-ku, Tokyo 113-8656 Japan; [2]The University of Tokyo, Dept. of Metall., 7-3-1, Hongo, Bunkyo-ku, Tokyo 113-8656 Japan

A macro model for evaluating steel usage pattern in Japanese society is proposed. The steels are mainly classified into two usage gropes for machinery and constructions, and it is assumed that steels are discharged from the society in accordance with each lifetime distribution. The amount of recycling steel and the stock are calculated using a population balance model with the input data of the statistics for basic steel production, domestic consumption ratio, machinery scrap ratio and consumption ratio for constructions. The validity of this model is examined by the comparison with the calculated results and statistics of stock and steel production by electric furnaces. The copper concentration in recycling steel and the amount of total CO2 emission are calculated, and the effect of recycle ratio on the total amount of CO2 emission is discussed with the allowable copper concentration.

3:20 PM

Recovery of Pure MnO_2 from Medium-Grade Local Manganese Ores: *M. B. Morsi*[1]; [1]Central Metallurgical Research and Development Institute, Pyro. Div., P.O. Box 87, Helwan, Cairo, Egypt

Local medium-grade manganese ore was blended with different amounts of sodium bisulphate as sulphatizing agent and roasted at temperature up to 800°C for various periods. The roasted products were subjected to water leaching to recover the soluble manganese sulphate. Maximum Recovery of manganese as sulphate achieved under optimum conditions was 98.5 %. The leached liquors of manganese suphate were purified and chemically treated to obtain pure γ-MnO_2 suitable for dry cell batteries. The purified solution was concentrated to obtain crystallized manganese sulphate and other valuable manganese chemicals. The mechanism of the sulphatizing roasting process was suggested.

3:45 PM Break

4:00 PM

Exergy Analysis of Plastic Recycling: *Hitoshi Ohya*[1]; Shigeki Koyanaka[1]; Shigehisa Endoh[1]; Atsushi Inaba[2]; [1]National Institute for Resources and Environment, Matls. Process. Dept., 16-3 Onogawa, Tsukuba, Ibaraki 305-8569 Japan; [2]National Institute for Resources and Environment, Rsch. Plan. Off., 16-3 Onogawa, Tsukuba, Ibaraki 305-8569 Japan

Recycling is very important for the global environment, however, it is very difficult to determine its quantitative impact. Generally speaking, a life cycle assessment is currently the most popular and typical method to solve it. Exergy is important when considering the global environmental impact. All of our activities have been developed to obtain a high exergy material and/or energy and throw away the waste and/or waste heat. We are trying to use exergy to estimate the quantity of energy and matter for resource accounting. The environmental assessment of plastic recycling was possible using this method and the exergy reduction of the entire system was estimated by the yield of matter and the process efficiency.

4:25 PM

International Domination and Conflicts Viewed from Metal Resources: A Historical Review: *Tetsuya Shoji*[1]; [1]The University of Tokyo, Dept. of Environ. Sys., Dept. of Geosys. Eng., Sch. of Eng., Tokyo 113-8656 Japan

Many conflicts have been caused on struggles for mineral and energy resources in the human history. One of the recent conflicts was the Gulf Crisis, at which Iraq claimed that Kuwait belonged to Iraq, and invaded Kuwait. In ancient Egypt, kings got gold from Eastern Desert and copper from Sinai. When kings were strong, they could keep copper mines in Sinai. When they were weak, they could not keep the mines and the route from the mines to the capitals, and hence the economic background also decreased. Every dynasty in the ancient Mesopotamia got copper and silver from the Taurus (Turkey), gold from Egypt, and copper from Cyprus. Hittite firstly used iron. Unfortunately, we have no record of conflicts caused on iron resources. This may imply that iron resources were common such as placer, and that more important resources were wood as fuels. In Ancient Greek, Athens developed the power based on silver produced in the Laureion district. The medieval Muslim World got a lot of silver from Horasan and Transoksania, much gold from Africa. The Habsburg family was the most important in the recent European history. The economics of the family was based on silver mines in Tyrol. They also controlled Hungarian copper, and established the first copper syndicate in the world. The Spanish supremacy increased with increasing amounts of silver imported from the New World (South America) in middle 16th Century, and decreased with the decreasing amounts in late 16th Century. The Industrial Revolution began in England where a remarkable amount of coal was used already, and progressed along iron deposits from the Western to Eastern Europe. Great Britain established the supremacy in 19th Century. In the process to establish the supremacy, Great Britain defected Boor people, and got gold and diamonds in South Africa. Not only Great Britain but also the other European Powers occupied African lands as colonies, and developed various kinds of metal resources such as copper in Zambia and Congo, tin in Nigeria, chrome in Zimbabwe, manganese in Gold Coast. In 20th Century, Germany rapidly developed, and hence became to need a lot of mineral and energy resources. For this reason, Germany clashed against Great Britain and France at many places in Europe, Africa, and West Asia. After the World War I, Germany tried to get the resources lost by the war. United States of America became to control natural resources in South America and other places in the world after the war, especially after the World Crisis. Japan was industrialized following the European Powers and United States of America, and extended the activity in East Asia to get natural resources. The war for mineral and energy resources has not happened among the World Powers after the World War II. However, many countries still use their powers to get natural resources in local areas in the world.

4:50 PM

Challenges Facing Mine Development: *Susumu Nagae*[1]; Hajime Ikeda[1]; [1]Metal Mining Agency of Japan, Tokiwa Bldg. 1-24-14, Toranomon, Minato-Ku, Tokyo 105-0001 Japan

Recently, a number of mine development projects have been setback by social, economic and, environmental circumstances that had not been seen before. Many of these problems do not always originate from direct mine pollution. Typical examples of these are conflicts between the exploration company and the local residents, sudden objections just before the development is to begin, and unexpected tightening of environmental regulations. The common background to these problems is expanding global environmental awareness. This includes complex factors such as the expansion of exploration to remote regions, the brisk activities of environmental NGOs, the increase of international environmental laws and, governmental policy changes. Today, the range of issues concerning mine development is widening significantly. The participation of local residents and NGOs in the development planning, together with the company and government, has become essential for smoothly carrying out mine projects and making them more beneficial for stakeholders.

Waste Management - I

Monday PM Room: Michaelangelo
November 6, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Patrick R. Taylor, University of Idaho, Plasma Process. Lab., Moscow, ID 83844-3024 USA; Masao Suzuki, Aiteku Gijitsu-Kaihatsu Company

2:00 PM Keynote

Utilization of Mining and Smelting Technologies and Facilities to Waste Management in Japan: *Yoshihiko Maeda*[1]; [1]Dowa Mining Company Limited, Waste Mgmt. Busi. Unit, 1-8-2 Marunouchi, Chiyoda-ku, Tokyo 100-8282 Japan

Mining industry once had been a polluter of toxic heavy metals and compounds as SOx gas, and naturally has acquired a variety of technology and facilities to reduce pollutants emission or to stabilize toxic elements, while expanding its production capacity and increasing in yield and productivity. In accordance with the world spread environmental problem, the industry is expected to contribute to reduce various environment burdens to the earth in many fields and favorably to establish so-called recycling society by utilizing mining and smelting technology and facilities. Some of these application examples are presented in this paper.

2:30 PM

Utilization of Wastes in Production of Portland Cement and Associated Environmental Measures-Separation and Decomposition of Chlorine Compounds: *Mitsuhiro Ito*[1]; [1]Taiheiyo Cement Corporation, Eng. and Tech. Compounds Dept., 3-8-1, Nishikanda, Chiyoda-ku, Tokyo 101-8357 Japan

For using various wastes contained much chlorine as alternative raw materials for Portland-cement, two practical methods for removing the chlorine were developed. One is; Ash-dust wastes with a high chlorine content, should first be washed and then dehydrate to a cake before introducing them into a cement manufacturing process. The cake can be used as an alternative to some cement raw materials. The other method is; For chlorine that is introduced into a cement manufacturing process from various wastes that have not been pre-washed, a method of extraction and removal during the manufacturing process is much effective. By combining these method, various wastes that were previously considered unsuitable for recycling due to their chlorine content can be used a lot more in cement manufacturing plants. Such methods will represent a major contribution to environmental protection. And they also have been solved the problems of diffusion of heavy metals and the generation and diffusion of dioxin from the cement plants. On the other hand, a manufacturing technology has been developed for "Eco-cement," a special cement that uses only incinerated ash produced from municipal wastes and sludge from sewage as raw materials.

2:55 PM

Removal of Impurities during Silver Recylcing Processes: *Takeshi Kamata*[1]; Mamoru Takayanagi[1]; Yasushi Akahori[2]; Itaru Jimbo[2]; Shunichi Takemoto[2]; Hiromasa Takeuchi[2]; [1]Matsuda Sangyo, Nishi-Shinjuku 1-26-2, Shinjuku-ku, Tokyo 163-0558 Japan; [2]Tokai University, Dept. of Metallu. Eng., 1117 Kitakaname, Hiratsuka, Kanagawa 259-1292 Japan

A cooperative research work on the silver recycling process is undertaken by Matsuda Sangyo Co., Ltd. and Tokai University in Japan. Silver wastes treated here are mainly from photograph industries where the antimony content in the printing paper is increased to suppress its inflammability in the recent years. The wastes are concentrated and then treated pyrometallurgically, where the impurities are removed by evaporation and oxidation. In the present paper, recent status of silver recycle industries in Japan will be reviewed. The result of the fundamental studies on the removal of related impurities and their behavior during the process will also be discussed.

3:20 PM

Elimination of Nickel from Copper Scrap Melt: *Hiroyuki Sano*[1]; Shin-ya Iwahori[2]; Mitsuru Tanahashi[2]; Toshiharu Fujisawa[1]; Chikabumi Yamauchi[2]; [1]Nagoya University, Rsch. Ctr. for Adv. Waste and Emiss. Mgmt., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Dept. of Matls. Sci. and Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

Pyrometallurgical process using Cu2O-based slag was proposed as a recycling process for copper scrap. However, it is difficult to remove nickel from copper by oxidation refining. Therefore, it is necessary to reduce the concentration of nickel in copper before the oxidation refining. In the present work, copper enrichment stage in this recycling process, where copper scrap melt is separated into Fe-C melt and molten copper, was taken up and the compositions of both melts were measured. Based on the above results, distribution ratios of nickel between Fe-C melt and molten copper were calculated. Furthermore, optimum condition of copper enrichment stage was discussed from the viewpoint of nickel removal and copper recovery. The possibility of recovering 4N-purity copper by using this recycling process was confirmed.

3:45 PM Break

4:00 PM

Precipitation Selectivity of Titanium Compounds from BF-Slags: *Zhitong Sui*[1]; Yuhai Li[1]; Guangqiang Li[1]; Junwei Ma[1]; Taiping Lou[1]; Nianxin Fu[1]; [1]Northeastern University, Schl. of Matls. and Metall., P.O. Box 119, Shenyang, Liaoning 110006 PRC

The precipitating behavior of mineral phase, perovskite (CaTiO3) in BF-slag, is obviously affected by operation factors such as chemical composition, atmosphere, temperature of heat-treatment, cooling rate and additives to the slag. The titania content in the perovskite phase can be artificially modified from 48% TiO2 of as-received BF-slag to 83% TiO2 of the treated slag by the transfer condition based on the thermodynamic and kinetic investigation for the titanium compound in the molten slag. The case study on kinetics of precipitation and growth for perovskite phase during the solidification process shown that the selective growth and coarsening of perovskite crystals can be archived by proper treatment based on the experimental data. A novel technique to recovery titanium compounds from BF-slag has been derived from the study results.

4:25 PM

The Processing of the Solid Household and Industrial Waste in the Slag's Bubbled Smelt with the Usage of the Vanyukov's Furnaces: *Andrey V. Tarasov*[1]; Valery M. Paretsky[1]; [1]State Research Institute of Non-Ferrous Metals, Gintsvetment, 13 Acad. Korolyov St., Moscow 129515 Russia

The design is based on the thermal processing of the waste in the intensively bubbled melt in the Vanyukov's furnace at the relatively high temperature (1350-1450°C). In such a case: the secondary waste are not formed and the especially toxic substances at the outlet from the furnace are absent. Harmful matters content in exhausted gases in the process of HGW treatment in the bubbled melted slag, mg/nm$_3$.

4:50 PM

Disposal of Waste Fluoride and Chloride Gases in the Manufacturing Process of Semiconductors: *Hideki Yamamoto*[1]; Junji Shibata[1]; [1]Kansai University, Dept. of Chem. Eng., Fac. of Eng., 3-3-35 Yamate, Suita, Osaka 564-8680 Japan

A novel technology to make trifluoro-nitrogen(NF_3) and other fluoride and chloride gases harmless has been developed. The NF_3 and other gases are toxic and special type of gases that are used in the process of manufacturing semiconductors such as the dry etching of semiconductors, cleaning of CVD systems etc. Total wasted amount of NF_3 and other fluoride and chloride gases (NF_3, CF_4, SF_6, CHF_3, BF_3, BCl_3, Cl_2) are increasing with an increase in semiconductor manufacturing. Since, NF_3 is highly toxic and stable chemically, they are liable to exert far-reaching adverse influences on global warming in the same manner as carbon dioxide gas and freon gases. The chemical reaction between fluoride and chloride gases and halogenized metals or metal ox-

ide was utilized in our disposal process. We established a new technology to make NF_3 and other toxic gases, contained in the exhaust gas of semiconductor manufacturing plants, convert to a harmless substance. Experimental results showed that the chemical reactions could take place at substantially lower temperatures of 80-400°C compared with the combustion treatment method.

Iron & Steelmaking - I

Monday PM Room: Raphael
November 6, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Brajendra Mishra, Colorado School of Mines, Dept. of Metall. and Matls. Eng., Golden, CO 80401 USA; Masamichi Sano, Nagoya University

2:00 PM Keynote
Kinetic Studies of Tank Degassing: *Gordon A. Irons*[1]; Dian-Cai Guo[1]; Dorel Anghelina[1]; [1]McMaster University, Matls. Sci. and Eng. Depts., 1280 Main St. West, Hamilton, Ontario L8S 4L7 Canada

To produce low-carbon (<30 ppm) steel in the tank degassing process, a ladle of steel with an oxidizing slag is placed in a vacuum tank equipped with bottom bubbling. The kinetics of this process are slow compared to the more prevalent RH degassing process, providing the impetus for the present work. There are several aspects to this work: water models of the fluid flow in the ladle; simulation of the decarburization (CO evolution) with CO2 desorption in water; pilot-plant (60 kg) studies of the decarburization kinetics; mathematical modelling of the fluid flow in the ladle; mathematical models for each of the kinetic sites (plume, top slag and auto-nucleation); and full-scale trials. The results show that during the refining cycle the contribution from each reaction site changes, suggesting avenues for reducing cycling time and final carbon contents.

2:30 PM
Capability of CO-H2-H2S Mixtures to Carbidize Iron Ore Reduced at Elevated Temperatures: *Yoshiaki Iguchi*[1]; Koji Matsubara[1]; Shoji Hayashi[1]; [1]Nagoya Institute of Technology, Matl. Sci. and Tech., Gokiso-cho, Showa-ku, Nagoya, Aichi 466-8555 Japan

The capability of CO-H2-H2S or CO-CO2 mixtures to carbidized reduced iron ores primarily depends on the reduction temperature and the nature of iron ore. We examined the effect of the reduction temperature from 1073 to 1273K, and a hematite iron ore and a limonite iron ore on the carbidization in the gas mixtures at 973K. It was confirmed that the capability decreases with increasing the reduction temperature and the carbidization ceases at a certain fractional carbidization less than 1, fq<1.0, before the completion.

2:55 PM
Reaction Rate of Coke with CO_2 and Degradation of Coke after Reaction: *Masahiro Kawakami*[1]; K. Murayama[1]; T. Takenaka[1]; S. Yokoyama[1]; [1]Toyohashi University of Technology, Dept. Prod. Sys. Eng., Tempakucho-aza-Hibarigaoka 1-1, Toyohashi 441-8580 Japan

Cokes play an important role of spacer in blast furnace. However, they degenerate during Boudouard reaction. Blast furnace cokes are reacted with pure CO_2 at 1072 to 1673 K. Reaction rate was obtained by the weight change of spherical specimen. The specimen after reaction was examined by microscopic observation. The reaction rate increased with temperature and was expressed by two Arrhenius type lines. The activation energy was 126 KJ/mol at higher temperature than 1373K and 202 KJ/mol at lower temperature range. At the higher temperature range, the reaction proceeded from the surface leaving unreacted core, whereas homogeneously whole inside at lower temperature. The polosity increased from the center to surface at higher temperature, whereas it was almost constant at lower temperature.

3:20 PM
Reduction Rate of Chromite Ore by Fe-C-Si Melts: Takamitsu Nakasuga[1]; Haiping Sun[1]; Kunihiko Nakashima[1]; *Katsumi Mori*[1]; [1]Kyushu University, Dept. of Matls. Sci. and Eng., Faculty of Eng., 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan

The kinetic behavior of reduction of chromite ore by carbon and/or silicon dissolved in liquid iron has been investigated under various experimental conditions. The results obtained are as follows: 1) When powders of chromite ore were added on the Fe-C melt surface, the rate of the reduction of the ore was so slowly, but when silicon was coexistent in the iron, it was preferentially oxidized and then the rate of reduction was considerably accelerated. 2) When the fluxes, such as SiO_2, $Na_2B_4O_7$, CaO-SiO_2, were added to the ore, which reacted with the ore to form a liquid slag and the reduction was also promoted.

3:45 PM Break

4:00 PM
Aging Process of Alloyed ADI: *Hamid Bayati*[1]; Roy Elliott[1]; [1]University of Manchester, Manchester Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK

Despite numerous studies regarding ADI, the aging behaviour has not been studied in detail. Such a study provides vital information regarding the changes in mechanical and physical properties of ADI during the aging process which determines the lifetime of ADI for different conditions. Current research presents kinetic results, mechanical properties and microstructural changes of an Mn alloyed ADI during aging at different temperatures. X-ray analysis, TEM, SEM, and metallographical studies were used to investigate the effect of temperature and time on the ADI microstructure. The heat and rate of reactions, and activation energy for the reactions taking place during aging of ADI were measured using DSC analysis. Comparison of mechanical properties and microstructural observations with DSC analysis shows that the lifetime for ADI can be predicted at different temperatures. This study demonstrates that increasing the austenite carbon content, temperature and stress decrease the lifetime of ADI.

4:25 PM
Elimination of Inclusions in a Molten Metal Using a High Magnetic Field: *Kensuke Sasse*[1]; Norihisa Waki[2]; Shigeo Asai[1]; [1]Nagoya University, Grad. Sch. of Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Grad. Student, Grad. Sch. of Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

A new method for separating inclusions from a molten metal using a high magnetic field has been proposed. The principle of this method using a magnetization force has been confirmed in the separation of intermetallic compounds simulating metallic inclusions through series of experiments in Al-Si-Mn-Fe, Bi-Mn and Al-Si alloys. The feasibility study on magnetic separation in a continuous casting of steel has been conducted. In order to measure a magnetic susceptibility, the Gouy's method has been so modified by using a super conducting magnet as to work in a high temperature up to 1200°C. The theoretical expression which is indispensable to evaluate the magnetic susceptibility has been derived. By use of the expression the magnetic susceptibility of the intermetallic compounds used in the magnetic separation

was measured over the range of liquidous to solidous temperatures.

4:50 PM
Application of Equal Channel Angular Extrusion to the Control of Microstructure in Stainless Steels: *N. Saito*[1]; M. Mabuchi[1]; M. Nakanishi[1]; I. Shigematsu[1]; M. Nakamura[1]; G. Yamauchi[2]; T. Asahina[1]; [1]National Industrial Research Institute of Nagoya, 1-1 Hirate-cho, Kita-ku, Nagoya, Aichi 462-8510 Japan; [2]Daido Institute of Technology, 2-21 Daido-cho, Minami-ku, Nagoya, Aichi 457-8531 Japan

Commercial stainless steels (SUS430 and SUS304L) were subjected to intense plastic deformation by using equal channel angular extrusion (ECAE) at room temperature. SUS304L processed by ECAE consisted of many dislocations and dislocation cell structure of about 0.5μ m in size. Room temperature tensile strength of SUS304L processed by ECAE was much higher than that of no ECAE sample. This is probably attributed to fine dislocation cell structure. Annealing at 673K was effective to increase the room temperature tensile strength of SUS304L. Tensile strength of 909MPa was attained by combination of ECAE and annealing at 673K for SUS304L. ECAE was also effective to increase the room temperature strength of SUS430. Annealing at 773K increased both tensile strength and elongation of SUS430 processed by ECAE.

Electronic, Magnetic & Photonic Materials - I

Monday PM Room: Dante
November 6, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: S. Govindarajan, Motorola; Takuo Takeshita, Mitsubishi Materials Company

2:00 PM Keynote
Enabling of Low-Cost, High Rate PVD Processing of Electronic Thin-Films using Intelligent Process Control: *Bruce Lanning*[1]; [1]ITN Energy System, Inc., Wheat Ridge, CO 80403 USA

As both size and performance requirements for electronic, magnetic, and optical devices are pushed to new levels, radical new methodologies in materials processing and manufacturing are necessary in order to create the high performance materials (and corresponding high process yields) that have heretofore been considered too unstable for commercialization. Using fundamental models for simulating material evolution during processing in combination with advanced in-situ sensors for tracking process changes, a new level of interactive, feed-forward control has been created, enabling cost-effective, atomic layer control of thin-film devices such as polycrystalline solar cells, superconductors, and giant magneto resistors for magnetic storage. The combination of models and sensors not only is used to improve process yield and product performance (quality), but is also used as a "virtual" tool for process design and optimization in the development of new and alternative processes. A brief overview of current thin-film control methodologies that have enabled low-cost processing high performance electronic, magnetic, and optical materials will be discussed.

2:30 PM
Processing of Ultrafine Ferrite Particles from Sulfuric Acid Pickling Solutions: *Yasuhiro Konishi*[1]; Kazunari Mizoe[1]; [1]Osaka Prefecture University, Dept. of Chem. Eng., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

This paper describes a new processing of ultrafine, magnetic ferrites from sulfuric acid pickling solution in steel industry. In the ferrite processing, the iron (III) was first supplied by the microbial oxidation of ferrous sulfate in the pickling solution, and then the bio-oxidized pickling solution was treated with solvent extraction to separate the iron (III) into the organic carboxylate solution. After that, the carboxylate solutions of iron (III) and other metal (cobalt or nickel) were hydrolyzed and precipitated crystalline ferrites under hydrothermal conditions. Experimental studies were conducted to collect rate data and investigate various process parameters for the oxidation of iron (II) in the pickling solutions by using iron-oxidizing bacteria. Moreover, the ultrafine particles of cobalt ferrite and nickel ferrite were prepared by hydrolysis of non-aqueous mixed-metal carboxylate solutions using water at 473K. The ferrites were free from contamination by the organic materials.

2:55 PM
Microstructural Effects on the Damping Capacity of Fe-Cr-Mn Alloys: *Kazuya Miyahara*[1]; Wu Kepeng[2]; Kohji Okada[2]; Chang-Yong Kang[3]; Masahiro Sasaki[2]; Naohiro Igata[4]; [1]Nagoya University, Dept. of Molecular Des. and Eng., Furoh-cho, Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Grad. Sch., Furohcho, Chikusa-ku, Nagoya 464-8603 Japan; [3]Pukyong National University, Dept. of Metall. Eng., Yong Dang-Dong, Nam-ku, Puasn South Korea; [4]University of Tokyo, Dept. of Fund. Eng. Sci., Yamazaki, Noda-city, Chiba-Pref 278-8510 Japan

Fe-Mn alloys are known to be one of high damping capacity materials. In the present research, the damping capacity of the Fe-Cr-Mn alloys, in which 10% Cr is added for the improvement of corrosion resistance of the Fe-Mn alloys, is investigated. Particularly the effects of microstructure (including cold work effect) and various phases of α, γ, ε and α′ on the damping capacity of the Fe-10 mass % Cr-5 to 30 mass % Mn alloys are clarified. At the solution treatment state, the alloy containing 3 phases of γ, ε and α′ indicates the largest damping capacity. The alloys composed of γ and α′ or of γ and ε show the medium value. From these results, the interface between α′ and ε is considered to have a beneficial effect to increase the damping capacity of the Fe-Cr-Mn alloys.

3:20 PM
Synthesis of Transition Metal Mixed Sulfides MTS_3 (M=Ba, Pb, T=Ti, Zr) Using Sulfur Melt: *Nobuaki Sato*[1]; Yarong Wang[1]; Kohta Yamada[1]; Takeo Fujino[1]; [1]Tohoku University, Instit. for Adv. Matls. Process., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

Ternary mixed sulfides of transition metals were synthesized by a relatively low temperature process using sulfur melt. The mixture of starting materials, such as, metals, binary sulfides and a large excess of sulfur was reacted in a vacuum-sealed Pyrex ampoule at 623-723K for one to three weeks. After the reaction, the remained sulfur was completely removed by filtration of sulfur melt and dissolution in CS_2. The impurity binary sulfides coexisted in products were removed by rinsing the products in water and acetone. By using the above method, $BaTiS_3$, $PbTiS_3$ and $BaZrS_3$ were obtained in a pure form. The effect of addition of halides on the yields of these ternary sulfides were also discussed.

3:45 PM Break

4:00 PM
Preparation and Properties of Germanium Sulfide Glasses: *Hiromichi Takebe*[1]; Kenji Morinaga[1]; [1]Kyushu University, Grad. Sch. of Eng. Sci., Kasugakouen 6-1, Kasuga 816-8580 Japan

Germanium sulfide (Ge-S) glasses are of interest for infrared optical materials. The Ge-S system has favorable properties for

practical applications including wide glass forming compositions, non-toxicity, chemical stability, and high glass transition temperature. Binary Ge-S glasses were prepared under a careful processing: weighing in a nitrogen-filled glove box and melting in a silica ampoule with a gentle rotation. The effect of impurities on infrared transmission properties is studied. Compositional dependences of properties such as density and refractive index are evaluated systematically. X-ray photoelectron spectroscopy and high resolution X-ray fluorescence analysis are used for evaluating the valency state of Ge and S ions in the glasses. Correlation between properties and structure is discussed.

4:25 PM
Thermodynamic Analysis for Crystal Growth of the III-V Compound: *Katsunori Yamaguchi*[1]; Yoichi Takeda[1]; [1]Iwate University, Dept. of Matls. Sci. and Tech., Ueda 4-3-5, Morioka, Iwate 020-8551 Japan

This work aimed at analyzing the crystal growth processes using newly experimental data of the phase diagram and thermodynamic properties measured by authors. Based on the obtained data of the phase diagram and thermodynamic properties for the III-V systems partial pressure-temperature-composition correlation diagrams of the Ga-As, In-As, Ga-P and In-P systems were contracted. The total vapor pressures of arsenic or phosphorus at melting point of GaAs, InAs, GaP and InP were determined as 101, 10.1, 253 and 1510kPa, respectively, at the corresponding melting points of 1510, 1211, 1739 and 1336K. These diagrams are applied to the analysis of the horizontal Bridgman method, the liquid encapsulated Czochralski method and the liquid phase epitaxial growth technique. On the other hand, the analysis of the equilibrium state of the chloride-CVD process carried out using the data of the Gibbs energy of the compounds obtained by authors.

Control and Analysis in Materials Processing - I

Monday PM Room: Rubens
November 6, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: T. Battles, Dupont Chemicals; Norihiko Fukatsu, Nagoya Institute of Technology, Dept. of Matls. Sci. and Eng.

2:00 PM Keynote
Materials Process Modelling: A General Tool or a Specialty: *Hani Henein*[1]; [1]University of Alberta, Chemical & Materials Engineering, 536 Chemical-Mineral Engr. Bldg., Adv. Materials Processing Lab., Edmonton, Alberta T6G 2G6 Canada

Materials Engineering is enjoying several decades of discovery of new materials and mechanisms, and advancements in tools for materials research and process analysis. These discoveries and advancements range from the formulation of micro-alloyed steels, novel ceramics and electronic materials to the formulation of strengthening mechanisms, the evolution of microscopy and the application of transport phenomena to materials processing operations. This last development was enabled in part by the extensive use of the computer for solving complex models. Over the past two decades the increased accessibility of the computer has enabled the development of software codes that are highly accessible and are user neutral or user friendly. This has in turn empowered anyone to develop mathematical representations of processes. Conducting a basic literature search one learns quickly that models have been developed or proposed for all materials processing operations in use. What then is the future of Process Modelling in our discipline? Is this a mature area of research requiring no specialists? This paper will address some of these issues by exploring the evolution of this field and postulating new directions of research.

2:30 PM Keynote
In-Situ Characterization of Sintering Properties with Non-Contact Ultrasonic Measurements: *James C. Foley*[1]; David K. Rehbein[1]; [1]Ames Laboratory, Metall. and Cer. Pgm., 122 Metals Development, Ames, IA 50011 USA

In-situ characterizations of green state part density and sintering state have long been desired in the powder metal community. Recent advances in non-contact electromagnetic acoustic transducer (EMAT) technology have enabled in-situ evaluations of the acoustic attenuation and velocity as sintering proceeds. Pure aluminum (99.999%) and aluminum alloy powders were prepared with high pressure gas atomization (HPGA). The powders were pressed in a uniaxial die and examined with acoustic waves for changes in velocity and attenuation during sintering. The changes in acoustic properties were correlated with sample microstructures and mechanical properties. Evolution of the acoustic echo train during sintering is shown to provide information on the state of sintering as well as having the potential for detection of interior flaws. This work is funded by a laboratory directed research and development grant and by DOE-BES under contract no.W-7405-Eng-82.

3:00 PM
Evaluation of Frictional Properties of Polycrystalline Diamond at Room Temperature: *Ali Soleman Al-Watban*[1]; [1]Riyadh Technical College, P.O. Box 53699, Riyadh 11593 Saudi Arabia

In this paper, the room temperature friction and deformation properties of various polycrystalline diamonds; i.e. aggregates of diamond crystals with a "binder" phase of cobalt or silicon carbide and a polycrystalline coating produced by a chemical vapour deposition processes (CVD) were studied when softer metallic and ceramic sliders were used. The experiments were conducted at relative low sliding speeds (10 mm/min), in a vacuum of 0.0001 mbr after having cleaned the specimen surfaces by out-gassing them for about 30 min at 800°C. As a result of increasing the number of traversals significant wear of the coated diamond by softer metallic sliders (aluminum and mild steel) was observed. With ceramic sliders, it is shown that the multiple traversals result in the formation of wear groove. With diamond silicon carbide, high friction and adhesion lead to significant metal transfer which then pre-empts cumulative deformation of the harder materials. With the diamond-cobalt composite, the friction is surprisingly low, there accumulation of wear debris on the tip of the slider but no permanent transfer of metal and the harder specimen is not damaged even after 5000 traversals.

3:25 PM
Structure and Performance Evaluation of Hydrogen Sensor for Molten Copper under Industrial Conditions: *Koji Katahira*[1]; Kunihiro Koide[1]; Takashi Iwamoto[1]; Noriaki Kurita[2]; Norihiko Fukatsu[2]; Teruo Ohashi[2]; [1]TYK Corporation, Adv. Matls. Rsch. and Dev. Ctr., 3-1 Ohata-cho, Tajimi 507-8607 Japan; [2]Nagoya Institute of Technology, Matls. Sci. and Eng. Dept., Gokiso-cho, Showa-ku, Nagoya 466-8555 Japan

The galvanic cell-type hydrogen sensor employing $CaZr_{0.9}In_{0.1}O_{3-\delta}$ as the solid electrolyte was designed for the measurement of hydrogen activities in molten copper under industrial conditions. The sensor probe consists of the cap-shaped solid

electrolyte with the porous platinum electrode on the inside and alumina refractory tube. The gas mixture of the composition 3%H_2-1%O_2-Ar was made to flow on the inside electrode as a reference through a nickel tube with which the electrical connection to the inner electrode was made. The hydrogen sensor probe was inserted directly into molten copper and the melt was used as the outer electrode. The effect of oxygen activity gradient to emf was eliminated theoretically by measuring simultaneously the oxygen activity in the melt by an oxygen sensor. The high stability of output and excellent response to the change of hydrogen activity were obtained over the wide range of industrial copper melting conditions.

3:50 PM Break

4:05 PM

Conventional V_s Hot Die Forging of Alloy 685: Process Optimisation for Properties: *V. Gopala Krishna*[1]; Balram Gupta[1]; [1]Government of India Ministry of Defense, Def. Rsch. and Dev., Org. Reg. Ctr. for Military Matls., P.O. Kanchanbagh, Hyderabad 500058 India

Conventional β-forging and hot die α-β forging have been conducted on aerospace grade near-α titanium alloy 685 with low oxygen content at the temperatures ~1025°C and ~960°C respectively using similar amounts of reduction (60%) and strain rates ($1.0s^{-1}$) with a view to optimize processing for mechanical properties. Structural examination revealed distorted and comparatively finer acicular platelike α with more defined platelet colonies resembling a basketweave pattern in the hot die forged pancake, which may be attributed to higher amounts of reduction below β-transus temperature and precise controlling of process variables. On heat treatment, hot die forged material displayed basketwoven structure distinctly with smaller average prior-β grain size, whereas platelike α in transformed β-matrix was observed in conventionally forged material. Characterization studies indicated an overall balance of properties in hot die forged material compared to conventionally forged, due to structural refinement during sub-transus processing. Creep resistance, a mandatory requirement for advanced aoroengine compressor disc applications, has increased by two-fold in hot die material, due to refined α-β interfaces.

4:30 PM

Conduction Electron Generation upon Oxygen Release of Indium Tin Oxide: *Takahisa Omata*[1]; Hiroyuki Fujiwara[1]; Shinya Otsuka-Yao-Matsuo[1]; Naoki Ono[2]; [1]Osaka University, Dept. of Matls. Sci. and Process., Grad. Sch. of Eng., 2-1, Yamada-oka, Suita 5650871 Japan; [2]Mitsui Mining and Smelting Company Limited, Elect. Matls. Sect., 2081, Tohsen, Ohmuta 8360003 Japan

The electrical conductivity and the oxygen release and uptake of sintered indium tin oxide (ITO) under $P(O_2)/P^*=4.9x10^{-4}$ (P^*: atmospheric pressure) atmosphere were simultaneously measured as a function of temperature using a closed-system oxygen gas analyzer. Two kinds of oxygen release and uptake were detected in temperature ranges of 973<T<1130K and 1130<T<1273K. The electrical conductivity increased due to the oxygen release at the higher temperature, although it was not altered by the oxygen release at the lower temperature. Hall measurements showed that the increase in the conductivity upon the oxygen release was caused by increasing of the conduction electron density and two conduction electrons generated by the release of one oxygen atom. Based on the structural chemistry, it was concluded that the oxygen atoms released, by which the conduction electron density increased, were originally at the 16c site in the C-type rare-earth structure as an interstitial excess oxygen, O_i.

4:55 PM

Equal Channel Angular Extrusion (ECAE): from Macromechanics to Structure Formation: *Vladimir Segal*[1]; [1]Johnson Matthey Electronics R&D, Prin. Rsch. Sci., 15128 E. Euclid Ave., Spokane, WA 99216 USA

A special deformation technique to impart very intensive, uniform and oriented simple shear to bulk materials is considered in details. In this case, continual plasticity controls structure and texture evolution at micro/sub-micro scales and is critical for attained effects. More precision analysis of plastic flow that was done before is presented for one step and multi-pass ECAE. Links between macromechanics of simple shear and structure formation such as grain refinement, control of phase and precipitates, materials consolidation and bonding are outlined and some possible applications are considered. The presentation includes: Introduction, Mechanics of ECAE, Multi-pass ECAE, Equivalent strains, Flow localization and Structure formation during ECAE.

Copper, Nickel, Zinc, Lead and Tin - I

Tuesday AM Room: Rubens
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Robert Stephens, Cominco; Mitsuhisa Hino, Tohoku University, Institute for Advanced Materials Processing

8:30 AM Keynote

Thermodynamic Study of Oxygen-Smelting of Copper or Nickel Matte: *Kimio Itagaki*[1]; [1]Tohoku University, Instit. for Adv. Matls. Process., 2-1-1 Katahira Aoba-ku, Sendai 980-8577 Japan

As a fundamental study for smelting copper or nickel matte using oxygen-enriched air or concentrated oxygen as the blowing gas, the phase equilibrium and the distribution of some minor elements between the copper or nickel matte and the iron-silicate or calcium-ferrite base slag were experimentally investigated under controlled partial pressures of sulfur and oxygen at the SO_2 partial pressure of 0.1, 0.5 and 1.0 atm at 1523-1573K. The solubility of copper in the slag was found to be independent of the SO_2 partial pressure at a specified matte grade while that of nickel increased with increasing SO_2 partial pressure. The distribution ratios of cobalt, lead, arsenic, antimony and bismuth also changed with the SO_2 partial pressure. The obtained results were discussed on the basis of thermodynamics.

9:00 AM

Modern View on Composition of Autogenous Smelting Slags and Methods of Their Decoppering: *Valery M. Paretsky*[1]; Andrey V. Tarasov[1]; [1]State Research Institute of Non-Ferrous Metals, Gintsvetmet, 13 Acad. Korolyov St., Moscow 129515 Russia

The knowledge of the true phase composition of the liquid slag is a very important problem, mastering of which grants the possibilities for the scientifically founded choosing of the decoppering method of the autogenous smelting slags. The investigation using different methods of the phase composition of the flash quenched autogenous copper melt's slags has shown, that the most effective method of the slags' decoppering during smelting with the white matte production is the flotation of the recrystallized slags. In this work the authors posed the problem to realize the slags' quenching with the rates similar to those, which are used in the modern investigations devoted to the flash quenching of the iron-containing alloys, with the subsequent study of

the quenched samples using the Mossbauer spectroscopy methods in order to establish the connection between the liquid slag's structure and the metals' losses with the slag.

9:25 AM

Increase in the Productivity of PS-Converters at Saganoseki Smelter & Refinery: *Masatoshi Maeda*[1]; Yushiro Hirai[1]; Fumio Hashiguchi[1]; Toshihiro Nagato[1]; [1]Nippon Mining & Metals Company Limited, Saganoseki Smelt. and Refine., Saganoseki-cho, Oita-pre. 879-2201 Japan

Increase in the copper smelting capacity at Saganoseki Smelter & Refinery was attained through various improvements in the PS-converters and the flash furnace. Two of the major innovations in the converting process were: a new operational technology which avoids increase in the copper content of the converter even when higher copper grade mattes (65%) are being treated in the slag making stage and the technology which shortens the operational interruption time which is caused by coolant charging. The latter innovation was realized by introducing a new automated coolant charging system with which it was made possible to charge the coolant without stopping the blowing into the converter. These improvements brought about a remarkable increase in the production capacity per converter and an annual production of 450,000 tons without adding new furnaces was made possible. In addition, good results have been obtained of the tests with modified tuyeres to further increase the production.

9:50 AM

A Model Study of Splash in a Peirce-Smith Converter: *Yasumasa Hattori*[1]; Kimiaki Utsunomiya[1]; Yoshiaki Mori[1]; Toyokazu Okubo[2]; [1]Sumitomo Metal Mining, Pyrometall. Rsch. Ctr., Niihama Rsch. Labs., Otu 145-1, Funaya, Saijo, Ehime 793-0005 Japan; [2]Sumitomo Metal Mining, Niihama Rsch. Labs., Isouracho 17-5, Niihama 982-0002 Japan

The effect of various operating conditions on splashing behavior in a Peirce-Smith converter has been investigated with a 1/5.7 scale cold model. Dynamic similarity between the cold model and a commercial converter was attained by setting the modified Froude number in the model experiments in the range of the normal converter operation. It is known that for a given modified Froude number the dimensionless penetration length of air into the liquid phase increases as the liquid density decreases. Moreover, the penetration length had a large effect on the splashing behavior. Therefore, a mixture of water and lead concentrate was used as the liquid phase. It is concluded that the amount of splash decreases with the reduction of the buoyancy energy per unit liquid mass and the increase of the horizontal distance between the tuyere tip and the edge of mouth.

10:15 AM Break

10:30 AM

The Q_{235} (A_3) of Seawater Microbe Corrosion in Dayao Gulf: *Wang Li*[1]; Chen Jiafu[2]; Qiao Qianni[1]; [1]Northeastern University, Coll. of Res. and Civil Eng., Shenyang, Liaoning 110006 China; [2]Fushen Petroleum Institute, Mach. Dept., 113001 China

In Dayao gulf natural seawater, the experiments of using $Q_{235}(A_3)$ Tin piece to carry out complete seawater immersion were done, which tested and evaluated the material that was corroded by biotic factor and so on below natural seawater environment. The seawater corrosion mechanism was explored. The Result indicated that seawater pollution and metal corrosion have intimate relationship. Protecting seawater environment quality and observing country emission standard are important measures of controlling seawater biotic corrosion.

10:55 AM

Lead Smelting and Refining at Kosaka Smelter: *Hiroshi Inoue*[2]; Shinsuke Nakaya[1]; Kousuke Inoguchi[3]; Satoshi Nakagawara[1]; [1]Kosaka Smelting and Refining Company Limited, Lead Smelt. Sect., 60-1 Otarube, Kosaka, Akita 017-0202 Japan; [2]Kosaka Smelting & Refining Company Limited, Smelt. & Refining Dept., 60-1, Otarube, Kosaka, Akita 017-0202 Japan; [3]Kosaka Smelting & Refining Company Limited, Tech. Dept., 60-1, Otarube, Kosaka, Akita 017-0202 Japan

The roles of lead smelting section of Kosaka Smelter are; 1) to make the operation of copper smelting section stable by removing the impurities in the complex sulfide concentrates; 2) to recover the valuable metals in the dust generated by flash furnace; 3) to recover the valuable metals in the leach residue from Akita Zinc Refinery; 4) to recover lead in spent lead-acid battery. According to this operating role, Kosaka Smelter has established the technology of electric smelting process with hydrometallurgical dust treatment plant, leach residue flotation plant, spent lead-acid battery crashing-separation plant, and developed the profitable process. This paper described experience of practical operation in the last decade.

11:20 AM

The Oxidation Rate of Pb and Ni in the Copper by Slag: *Kunihiko Nakashima*[1]; Keisuke Yamamoto[1]; Etsuro Shibata[1]; Hiroko Tahori[1]; Katsumi Mori[1]; [1]Kyushu University, Dept. of Matls. Sci. and Eng., Faculty of Eng., 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan

The kinetic experiments were carried out at 1200°C to develop some effective slags for removing Pb and Ni from copper melt and the effect of slag composition on the extent and the rate of the removal was examined. It was found that the slag viscosity and the activity coefficient of PbO were lowered by the addition of B_2O_3. For slags containing B_2O_3, the reaction almost finished within 5 minutes after adding slag and the degree of removal was more than 90%. But the removal degree of Ni was not so high compared with that of Pb. Thus, Ni removal by oxidation was more difficult than Pb removal. For an example, the removal degrees of more than 90% for Pb and of about 50% for Ni were obtained Cu_2O-B_2O_3-Fe_2O_3 slag. The change in metal composition during oxidation reaction could be explained by the coupled reaction model.

11:45 AM

Study of the Influence of Chemical Composition on the Machinability Properties of Brasses: *Cândida Guerra Vilarinho*[1]; Paulo Davim[2]; Fernando Castro[1]; [1]Universidade do Minho, Dept. Eng. Mech., Rua Capitão Alfredo Guimarães, Guimarães 4810 Portugal; [2]Universidade de Aveiro, Secção Autónoma de Eng. Mech., Campus Universitário de Santiago, Aveiro 3810 Portugal

Although brasses are essentially alloys of copper and zinc, they containing also alloying elements which are responsible for the wide variety of properties inherent to these materials. The effect of the chemical composition on the machinability of brasses has been investigated. This study includes commercial alloys and alloys that have been prepared in laboratory, to evaluate the effects of certain elements upon the machinability. The alloying elements studied included lead, aluminium, iron, silicon and tin. Machinability tests have been carried out in a CNC turning under lubricated conditions. The experimental procedure consisted on turning operations, during which cutting forces and surface roughness obtained in brass workpiece were measured. The influence of the various alloying elements in cutting forces, surface roughness and chip class of brasses has been accordingly evaluated.

Rare Metals

Tuesday AM Room: Raphael
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: James C. Foley, Iowa State University, Ames Laboratory; Toshio Oishi, Kansai University, Dept. of Matls. Sci. and Eng.

8:30 AM Keynote
The Development of Processes for the Production of Selected Rare Earth Electronic Materials: *Renato G. Bautista*[1]; [1]University of Nevada at Reno, Metall. and Matls. Eng. Dept., Reno, NV 89557 USA

The development of processing methods for the preparation of three electronic materials with rare earth components is discussed. The resulting processing innovations are the results of a search for an economically competitive pilot scale or industrial production flowsheet without compromising the required properties of the final products. The three cases highlighted in this presentation are:1) The preparation of high energy (Pr-Nd-Ce)FeB magnets for MRI application from a mixed-rare-earth oxide with a narrow range of composition directly prepared from the solvent extraction circuit of bastnaesite ore. 2) The kg production of the giant magnetocaloric effect $Gd_5(Si_xGe_{1-x})_4$ magnetic refrigerant materials from commercial grade gadolinium metal (95 to 98 at. % pure) instead of high purity gadolinium metal (99.8 at. %); and 3) An industrial chemical synthesis flowsheet for the production of the superconductor precursor of $YBa_2Cu_3O_7$.

9:00 AM Keynote
Processing and Applications of Rare Earth Metals and Intermetallic Compounds: *Ken-ichi Machida*[1]; [1]Osaka University, Dept. of Appl. Chem., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

Processing for rare earth metals and intermetallic compounds requires well-controlled conditions in order to avoid the oxygen contamination involved in it due to their high reactivity, as causes the serious decrease in the properties for materials such as magnets, metal hydride for hydrogen storage, and so on. Meanwhile, surface modification is effective to improve the oxidation-resistance of magnets and the hydrogen charge-discharge characteristics of metal hydride electrodes. After glancing about the fundamental properties and main applications of rare earth metals and intermetallic compounds, the results on the nitrogen absorption-desorption characteristics and magnetic property of R-Fe-N intermetallic compounds as obtained by the author's group will be presented and discussed to develop the high-performance epoxy resin-bonded magnets and ammonia-generating nitrogen storage materials.

9:30 AM
Aqueous/Organic Processing of Rare-Earth Carbonate Powders from Rare-Earth Loaded Carboxylate Solutions Using CO_2 Gas: *Yasuhiro Konishi*[1]; Yoshiyuki Noda[1]; [1]Osaka Prefecture University, Dept. of Chem. Eng., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

This paper describes a new processing of rare-earth oxide precursor, in which rare-earth carbonate powders are precipitated by emulsifying rare-earth loaded carboxylate solutions, using CO_2 gas and water. This synthetic route of rare earth carbonate is a combined process of the stripping and precipitation stages in a conventional solvent extraction process for separation and purification of rare earth metals. Using this technique, lanthanum carbonate and yttrium carbonate were prepared at temperatures of 283 to 353K and CO_2 pressures of 0.1 to 3.0 MPa for 120 min. The precipitation kinetics and yield were sensitive to processing parameters such as CO_2 pressure, temperature, and organic-phase composition. The particle size distributions of the carbonate powders were markedly dependent on temperature and CO_2 pressure.

9:55 AM
Rare-Earth-Based Hydrogen Storage Alloys for Batteries: *Nobuhiro Kuriyama*[1]; Tetsuo Sakai[1]; Hideaki Tanaka[1]; Hiroyuki T. Takeshita[1]; Itsuki Uehara[2]; [1]Osaka National Research Institute, 1-8-31 Midorigaoka, Ikeda, Osaka 563-8577 Japan; [2]Industrial Technology Center of Toyama Prefecture

AB_5 type hydrogen storage alloys (A: rare earths, B: transition metals) are extensively applied to a negative electrode of a nickel-metal hydride (Ni-MH) rechargeable battery for portable electric equipments, electric vehicles, hybrid vehicles etc. Performance of a hydrogen storage alloy electrode is affected by various factors, microstructure and surface composition of the alloy and structure of the electrode as well as composition of the alloy. Control of those factors enables us to commercialize the Ni-MH battery in various field.

10:20 AM Break

10:35 AM
Thermodynamic Consideration of Melting of Copper Based Alloys with Rare Earths: *Toshio Oishi*[1]; Motohiro Horiguchi[1]; Kazuo Ichii[1]; [1]Kansai University, Matls. Sci. and Eng. Dept., 3-3-35, Yamate-cho, Suita, Osaka 564-8680 Japan

As a fundamental study to prepare the copper based alloys with rare earths(RE: La, Gd, Nd, Ce and Y) without any inclusions, the solubility of rare earth in the liquid copper was experimentally determined. Liquid copper alloys were equilibrated with carbon and rare earth carbide in a graphite crucible in an argon atmosphere at 1523-1873 K. Solubilities of RE in the liquid copper were determined by approaching the equilibrium values from both the lower and the higher concentrations than those expected in the preliminary experiments. They were expressed as a function of temperature. Oxygen contents and solubilities of carbon were also analysed and thermodynamically discussed.

11:00 AM
Solid State Electrotransport of Oxygen in Praseodymium: *Hiroyuki Sano*[1]; Jun Sugiyama[2]; Hideki Takeuchi[2]; Toshiharu Fujisawa[1]; Chikabumi Yamauchi[2]; [1]Nagoya University, Rsch. Ctr. for Adv. Waste and Emiss. Mgmt., Furo-cho Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Dept. of Matls. Sci. and Eng., Furo-cho Chikusa-ku, Nagoya 464-8603 Japan

Solid State Electrotransport (SSE) is one of the most promising methods for the elimination of gaseous impurities from chemically active metals, such as rare earth metals. In the present work, SSE process was applied for the oxygen removal from praseodymium. The SSE factors, such as effective valence, were measured under the various temperatures, electric field intensities and initial oxygen contents, and effect of those conditions on the SSE process was discussed. Furthermore, solubility of oxygen in solid praseodymium was also measured by using SSE method and metal-oxide equilibrium method.

11:25 AM
Synthesis of La_2S_3 and Its Thermoelectric Properties: Shinji Hirai[1]; *Kazuyoshi Shimakage*[1]; Toshiyuki Nishimura[2]; Yoichiro Uemura[2]; Mamoru Mitomo[2]; Leo Brewer[3]; [1]Muroran Institute of Technology, Dept. of Matls. Sci. and Eng., 27-1 Mizumoto-cho,

Muroran-shi, Hokkaido 050-8585 Japan; [2]National Institute for Research in Inorganic Materials, Tsukuba Japan; [3]University of California-Berkeley, Berkeley, CA 94720-1760 USA

La_2S_3 may be considered to be a useful compound as thermoelectric conversion materials, because γ-La_2S_3 has a high Seebeck coefficient. In the present study, β-La_2S_3 powder was first synthesized via the sulfurization of La_2O_3 powder using CS_2 gas. Next, the synthetic powders of single-phase, β-La_2S_3 has been sintered by a hot-pressing technique to fabricate the sintered compacts of a single-phase, γ-La_2S_3 and a double-phase, γ-La_2S_3 and β-La_2S_3. Seebeck coefficient of sintered compacts was also measured. In the synthesis process of β-La_2S_3 via the sulfurization of La_2O_3 powder using CS_2 gas, La_2O_2S was formed in the initial stage of the reaction at sulfurization temperatures below 973K, and β-La_2S_3 was finally formed for a shorter period of time at higher sulfurization temperatures. For long sulfurization time of 28.8ks, single-phase, β-La_2S_3 was formed by sulfurization at about 873K. The oxygen content in β-La_2S_3 powders decreased gradually with an increase in sulfurization temperature. On the other hand, the carbon content in these powders increase gradually as the thermal dissociation reaction of a part of CS_2 gas proceeded with the rise of the sulfurization temperature. Though it has been reported that β-La_2S_3 was transformed to γ-La_2S_3 at 1723K±50K, the sintered compacts of single-phase β-La_2S_3 was formed at rather high temperature of 1973K when the synthetic powder of β-La_2S_3 containing much oxygen were used. However, phase transformation of β-La_2S_3 to γ-La_2S_3 progressed preferentially with a decrease in the oxygen content of synthetic β-La_2S_3 powder. Seebeck coefficient of double phase, and γ-La_2S_3 and β-La_2S_3 was higher than that of single-phase, γ-La_2S_3 reported previously.

11:50 AM

Hydrogen-Absorbing Alloys Containing Rare-Earth Elements and Their Applications: *Nobuyuki Higashiyama*[1]; Ikuo Yonezu[1]; [1]SANYO Electric Company Limited, New Matls. Rsch. Ctr., 1-18-13 Hashiridani, Hirakata, Osaka 753-8534 Japan

Hydrogen-absorbing alloys containing rare-earth elements were applied for high capacity nickel-metal hydride secondary batteries, hydrogen storage systems for a portable fuel cell and refrigeration systems. New hydrogen-absorbing alloys containing rare-earth elements have been also developed and introduced into these applications. In the case of nickel-metal hydride secondary batteries, higher performance has been strongly demanded by a remarkable progress of portable electric appliances. A large hydrogen absorption capacity, high electrochemical reactivity and superior corrosion resistance in an alkaline solution are required for a hydrogen-absorbing alloy used for nickel-metal hydride batteries. The aim of this investigation is to improve the performance of conventional LaNi5 type hydrogen-absorbing alloys. For example, Mm(Ni-Co-Al-Mn)x type hydrogen-absorbing alloys with various non-stoichiometric compositions were developed. Furthermore, a new process such as a rapidly quenching process was applied for preparing these hydrogen-absorbing alloys to improve their performance.

Electronic, Magnetic and Photonic Materials-II

Tuesday AM Room: Dante
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Bruce Lanning, ITN Energy Systems; Kenji Morinaga, Kyushu University, Dept. of Matls. Sci. and Tech.

8:30 AM Keynote

On the Progress of the HDDR Process as the Microstructure Control Method: *Takuo Takeshita*[1]; [1]Mitsubishi Materials Corporation, Cntr. Rsch. Instit., 1-297 Kitabukuro, Omiya, Saitama 330-8508 Japan

In all branches of materials sciences and engineering, we are engaged in finding new materials and inventing new processes to fabricate them into forms of practical applications. In some cases, novel processing techniques bring new materials as seen in the rapid quenching of the molten metals, which produces amorphous metals of some suitable compositions. It is very important that the processing techniques produce a product with a proper microstructure ranging form single crystal materials such as the semiconductor silicon wafer and the high temperature alloy turbine blade, coarse polycrystalline materials of many metallic parts, nano-crystalline materials of structural parts and functional materials, and to amorphous materials such as inorganic optical glass fibers and metallic glasses of magnetic applications and others. This is because that a practical material will not posses the expected property unless its proper microstructure is realized for each application. Magnetic materials are particularly sensitive to their microstructures as to their magnetic properties. This is seen in almost all magnetic materials from the soft magnetic materials and the hard magnetic materials to magnetic memories. In the case of the NdFeB magnet, since its invention the sintering technique and the rapid quenching with a proper annealing are only two methods to produce permanent magnets before the HDDR process was invented by us. The HDDR process utilizes the phenomenon, in which some hydrogen absorbing intermetallic compounds go through as they are heated in a hydrogen atmosphere, i.e., hydrogenation-decomposition-desorption-recombination. Choosing a suitable process conditioning regarding to hydrogen gas pressure, temperature for the forced desorption, and etc., we can produce permanent magnet powders of good magnetic properties with microstructures of sub-micron size crystalline grains. The progress of HDDR Process will be presented in this lecture comparing with other process techniques.

9:00 AM

Ferrite Thin Film Devices by Low Temperature Fabrication Processing in Aqueous Solution: *Masanori Abe*[1]; Kazuhiro Nishimura[1]; Yoshitaka Kitamoto[1]; [1]Tokyo Institute of Technology, Dept. of Phys. Elect., 2-12-1 O-okayama, Meguro-ku, Tokyo 152-8552 Japan

Ferrite plating enables crystalline spinel ferrite films to be grown from an aqueous solution at 60-100°C. This low temperature, soft-solution processing facilitates fabrication of novel ferrite film devices on non-heat-resistant materials such as plastics. In this paper we review principles and features (especially of low temperature aspects) of the ferrite plating, referring to such applications as magnetic recording media, microwave devices, and biomedical devices. For perpendicular magnetic recording, films of Co and NiZn ferrites were developed to be used as recording

media and high-permeability back layers, respectively. Because of a solution processing, ferrite plating enables us to coat surfaces of fine particles with ferrite layers, which exhibit a high biocompatibility. Thus ferrite-capsulated polymer microspheres were applied to antibody-carriers for enzyme-immunoassay which are actually used for clinical cancer diagnosis. In order to decrease the ferrite-plating temperature down to room temperature, we investigated the potential-pH phase diagram of Fe in solutions. Magnetite (Fe3O4) films were successfully grown at 25°C from an aqueous solution of Fe2 at pH =8-9 which was elevated from that (5-7) at which previous plating was performed at 60-100°C.

9:25 AM

The Role of EDTA and Glycine in the Chemical Mechanical Planarization (CMP) of Copper: Serdar Aksu[1]; *Fiona M. Doyle*[1]; [1]University of California, Dept. Matls. Sci. and Eng., 551 Evans Hall # 1760, Berkeley, CA 94720-1760 USA

CMP will be an essential process in the production of integrated circuits containing copper interconnects. This study aims to improve our mechanistic understanding of the role that organic chelating agents might play in the reactive slurries used in copper CMP. Potential-pH diagrams for the copper-water-EDTA and copper-water-glycine systems were first correlated with the potentiodynamic polarization behavior of copper in EDTA and glycine solutions. This suggested chemistries that were most promising for copper CMP slurries. Slurries with appropriate chemistry, containing alumina abrasive, were used with a SUBA 500 pad to polish a copper foil 0.5 mm thick, mounted on a modified lapping machine. In-situ electrochemical measurements monitored the dissolution of copper. The effect of parameters such as applied pressure, weight percent of alumina abrasive in the slurry, and relative velocity of the polish head with respect to the pad on the polish rate are reported.

9:50 AM

Preparation and Optical Properties of Aluminium Fluoroberyllate Glasses: *Shigeru Fujino*[1]; Kenji Morinaga[1]; [1]Kyushu University, Grad. Sch. of Eng. Sci., Kasugakouen 6-1, Kasuga 816-8580 Japan

Recently, it is important to design for optical systems in the UV region. In particular, fluorine doped silica glasses have attractive and interested applications for stepper lens such as F2 laser lithography with the wavelength of 157 nm. On the other hands, aluminium fluoroberyllate glasses are also interested as UV lithography equipments, because they have good UV-transmitting and low refractive index and dispersion. The optical properties of aluminium fluoroberyllate glasses are predominantly influenced by the glass compositions and its preparation. We will discussed the preparation of high purity aluminium fluoroberyllate glasses. The compositional dependence of the properties has been systematically discussed compared with fluorine doped silica glass and CaF2 crystal.

10:15 AM Break

10:30 AM

Exfoliation and Photocatalytic Activities of Layered Oxides $KSr_2Nb_3O_{10}$: *Michikazu Hara*[1]; Norikazu Ooto[1]; Takeshi Sumida[1]; Junko N. Kondo[1]; Kazunari Domen[1]; [1]Tokyo Institute of Technology, Rsch. Lab. of Resources Utilization, 4259 Nagatsuda-cho, Midori-ku, Yokohama, Kanagawa 226-8503 Japan

We carried out preparation of porous materials composed of niobate sheets by a soft-chemical method that took advantage of the exfoliation of layered perovskite oxide $KSr_2Nb_3O_{10}$, and the characterization and the photocatalytic abilities of them were investigated. The exfoliation of niobate sheets was carried out by stirring in aqueous tetrabutylammonium hydroxide solution. To obtain porous materials that consist of niobate sheets, the supernatant was stirred in HCl aqueous solution until the precipitation was formed. It was evidenced by TEM image that the layered niobate isexfoliated to the monolayer. The BET surface area of $KSr_2Nb_3O_{10}$ was 1 m^2/g,and that of porous materials that consist of niobate sheets was about 300 m^2/g in the maximum. The porous materials showed high activity for photocatalytic H_2 evolution from bulky alcohol solution in comparison with the original $HSr_2Nb_3O_{10.}$

10:55 AM

Magnetic Properties of Electrodeposited Ni-Fe Alloy Films: *Kenji Hara*[1]; Shinji Yamashita[1]; Hisayuki Kaku[1]; Michiaki Ikeda[1]; Shunsuke Kawano[2]; *Takeshi Ohgai*[2]; Tetsuya Akiyama[3]; *Hiroaki Nakano*[2]; Hisaaki Fukushima[2]; [1]Yaskawa Electric Corporation, 2-1 Kurosakishiroishi, Yahatanishi-ku, Kitakyushu 806-0004 Japan; [2]Kyushu University, Fac. of Eng., 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan; [3]Kyushu Sangyo University, Fac. of Eng., 2-3-1 Matsukadai, Higashi-ku, Fukuoka 813-8503 Japan

Ni-Fe alloys have an inverse magnetostrictive effect (Villari effect) in which its permeability varies according to the applied strain. This alloy film has a potential for application as force sensors such as torque sensors, tension sensors and so on. Electrodeposition is promising as a technique for preparing Ni-Fe alloy film because of its convenience and economy. In this paper, the effects of electrolysis conditions on the magnetic properties of electrodeposited Ni-Fe alloy films were investigated. Hysteresis loops of Ni-Fe alloy films on copper substrates from sulfate bath were measured by a vibrating sample magnetometer (VSM). The films of low coercivity, which were preferred when used as a force sensor, were obtained by introducing certain additives to the sulfate type bath.

11:20 AM

Hard Magnetic Properties of Quenched Sm-Fe-N Powders: *Takahiko Iriyama*[1]; Ryo Omatsuzawa[1]; Takayuki Nishio[1]; Norio Okochi[1]; Yuichiro Fujita[1]; [1]Daido Steel Company Limited, Rsch. and Dev. Lab., 2-30 Daido-Cho, Minami-Ku, Nagoya 457-8545 Japan

Sm-Fe-N hard magnetic materials are good candidates for high performance permanent magnets. Melt-quenching method should be highly favorable for the fabrication of high performance isotropic Sm-Fe-N bonded magnet. We report here the magnetic properties of Sm-Fe-N powders produced by several melt-quenching processes. Sm-Fe powders with various chemical compositions were prepared by atomization and melt-spinning processes. Nitrided Sm2Fe17 powder prepared by atomization process had a low coercivity under 1 kOe, since the powder had dendritic solidification structure due to the relatively low cooling rate of the atomizing. On the other hand, Sm2Fe17 powder made by melt-spinning process had fine crystal structure because of its higher cooling rate than the atomization process, and the nitrided powder showed a large coercivity of 5.2 kOe. It was found that Si, C, Al or Ga addition increased the coercivities of nitrided Sm2Fe17 quenched powders. The isotropic bonded magnet made of Sm2Fe18Si0.1Nx quenched powder showed a high (BH)max of 9.6 MGOe.

11:45 AM

Fluorescent Characteristics of Fine Multicomponent Particles of Yttrium and Europium: *Mikio Kobayashi*[1]; Yoshihiro Nishisu[1]; [1]National Institute for Resources and Environment, Matls. Process. Dept., Onogawa 16-3, Tsukuba-shi, Ibaraki-ken 305-8569 Japan

Yttria with europium (Y2O3:Eu3) is very important as a red phosphor. In this work, monodispersed multicomponent particles of Y2O3:Eu3 were prepared from homogeneous solution, and then, they were characterized as a red phosphor under the excitation of ultraviolet?\@rays, 256 nm or 147 nm. The strength of the fluorescence emission was varied with the ratio of europium/yttrium, the particle size and the calcination temperature. In case of excitation using 256 nm UV ray the fluorescent emission intensity was very high around the ratio of 5/95. The tendency that the fluorescence emission intensity increased with the increase of the particle size was observed. The calcination temperature affected crystalization, and so, that also affected the fluorescent emission intensity. The fluorescent emission intensity increased with the increase of crystallinity.

High Temperature Materials

Tuesday AM Room: Cervantes
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: D. Javernick, Los Alamos Natl. Lab.; Keizo Uematsu, Nagaoka University of Technology, Dept. of Matls. Sci. and Eng.

8:30 AM Keynote
The Preparation and Characterization of Boron-Carbon-Nitrogen Nanotubes by a DC Arc Plasma: *Yusuke Moriyoshi*[1]; Yoshiki Shimizu[1]; Takayuki Watanabe[2]; [1]Hosei University, Fac. of Eng., 3-7-2 Kajinocho, Koganeishi, Tokyo 184-8584 Japan; [2]Tokyo Institute of Technology, 2-12-1 Ookayama, Meguroku, Tokyo 152-8550 Japan

B-C-N nanotubes prepared by an evaporation method using a dc arc plasma were mainly characterized by transmission-electron microscopy (TEM) and electron-energy-loss spectroscopy (EELS). The data obtained indicated the presence of three types of nanotubes such as carbon-, boron nitride-, and carbon nanotubes surrounded with boron nitride nanotubes. However, BCXN nanotubes of homogeneous phase could not be obtained. This indicates that thermodynamically stable phase at the experimental conditions is in the mixtures of carbon and boron nitride rather than homogeneous phase of BCXN compounds. Also a relationship between temperatures and nanotube formation indicated that the formation of nanotubes was much affected by heating temperatures and nanotubes were formed at higher temperatures than 3000K. On the other hand, nanocapsules(nano-onions) were formed at lower temperatures than 2500K. Further, the effects of impurities on the formation of nanotubes and nanocapsules were described. Based on the data obtained, a model in the formation mechanisms of both nanotubes and nanocapsules is proposed.

9:00 AM
Origin of the Fracture Strength of Silicon Nitride Ceramics: *Hiroya Abe*[1]; Tadashi Hotta[1]; Makio Naito[1]; Nobuhiro Shinohara[2]; Masataro Okumiya[2]; Keizo Uematsu[3]; [1]Japan Fine Ceramics Center, 2-4-1 Mutsuno, Atsutaku, Nagoya, Aichi 456-8587 Japan; [2]Asahi Glass Company Limited, 1150 Hazawa, Kanagawaku, Yokohama, Kanagawa 221 Japan; [3]Nagaoka University of Technology, Dept. of Chem., 1603-1 Kamitomioka, Nagaoka, Niigata 940-2188 Japan

The relevance of the fracture strength to the microstructure was examined in a variety of silicon nitride ceramics. In the examination of microstructure, several kinds of ceramics including commercially used ones were made into thinned specimens, and their internal structures were observed with an optical microscope in the transmission mode. The strength of all ceramics examined in this experiment was quantitatively correlated with the pore size distribution in the specimens. The origin of large pores was ascribed to problems in the manufacturing process. Clearly, the elimination of these detrimental defects will enhance the reliability of ceramics significantly.

9:25 AM
Kinetic Effect of Moisture on Production of Ceramics: *Yutaka Saito*[1]; Satoshi Tanaka[1]; Nozomu Uchida[1]; Keizo Uematsu[1]; [1]Nagaoka University of Technology, Chem. Dept., Kamitomioka 1603-1, Nagaoka, Niigata 940-2188 Japan

Effect of moisture content must be fully understood for controlling the production of ceramics. It changes the mechanical behavior of spray-dried granules applied for compaction pressing, and governs the structure and properties of ceramics. To understand the time-dependent change in processing, the kinetics was examined for change of moisture content for granules placed in a die and the pressed compacts also. The results were explained in terms of apparent moisture diffusion coefficients. They varied non-linearly with the PVA content in the granules. The results were explained through the non-uniform PVA distribution in the granules.

9:50 AM
Aluminum Oxide Produced by an Oxy-Chlorination Process: *Ana Ester Bohe*[1]; Fabiola Alvarez[2]; Daniel Miguel Pasquevich[3]; [1]Consejo Nacional de Investigaciones Científicas y Técnicas, Unidad Complejo Tecnológico Pilcaniyeu CNEA, Av. Bustillo 9500, San Carlos de Bariloche, Rio Negro 8400 Argentine; [2]Comisión Nacional de Energía Atómica, Unidad Tecnológica Plicaniyeu, Av. Bustillo 9500, San Carlos de Bariloche, Rio Negro 8400 Argentine; [3]Comisión Nacional de Energía Atómica, Unidad Complejo Tecnológico Pilcaniyeu, Av. Bustillo 8400, San Carlos de Bariloche, Rio Negro 8400 Argentine

The oxidation of aluminum and its alloys was extensively studied for improving the corrosion resistance for construction materials having high specific strengths. But this reaction involves the formation of a very thin barrier layer on the metallic pieces. Furthermore the recent development of the directed melt oxidation process for producing metal-ceramic composites has renewed the interest in the study of interaction between gases and molten metals. Oxidation experiments in the presence of different partial pressures of chlorine and at different temperatures were performed in a thermogravimetric balance and microstructures were analysed by electronic microscopy and x-ray diffraction. The oxy-chlorination kinetic was determined and the oxide formed was amorphous at temperatures below 600°C and crystalline at higher temperatures. The appropriate experimental conditions for the fastest completed oxidation was further determined.

10:15 AM Break

10:30 AM
Influence of Slurry P_H on the Compaction Pressing of Alumina Ceramics: *Midori Saito*[1]; Nozomu Uchida[1]; Keizo Uematsu[1]; [1]Nagaoka University of Technology, Chem. Dept., Kamitomioka 1603-1, Nagaoka, Niigata 940-2188 Japan

The state of powder dispersion in the slurry is the major processing parameter, which governs the structures and properties of the granules and thus of the green compacts and ceramics. The granules of various characteristics were prepared by changing the slurry pH, and their properties as well as their influence on

pressing and sintering were studied in alumina ceramics. The granules obtained from the dispersed slurry were dimpled and had a smooth surface and high density. Those from the flocculated slurry were spherical and had a rough surface and low density. The green body fabricated from the former granules had more defects than that made from the latter. The microstructure development during sintering was also studied in the respective compacts.

10:55 AM

Modeling Grain Size During Hot Working of IN 718: *Steve C. Medeiros*[1]; Y.V.R.K. Prasad[1]; William G. Frazier[1]; Raghavan Srinivasan[2]; [1]Air Force Research Laboratory, Matls. Process. Des. Brnch., 2977 P St., Bldg. 653 Rm. 205, Wright-Patterson AFB, OH 45433-7746 USA; [2]Wright State University, Mechanical and Materials Engineering, Dayton, OH 45435 USA

Aerospace gas turbine disks operate in an environment of elevated temperatures and high stresses caused by centrifugal forces. These severe conditions necessitate the need for materials with high temperature strength and good low cycle fatigue resistance. One class of alloys used for this task is the nickel base superalloys, out of which, IN 718 is the most widely used in the aerospace industry. The properties of a gas turbine disk are sensitive to the microstructure, in particular the grain size, which is dependent on the processing history. The ability to precisely control the microstructural development during forging is dependent on controlling the process so the workpiece is deformed within a "safe" region where no microstructural damage or flow instabilities occur. This work describes the effort in establishing a relationship between the grain size and the process parameters i.e., temperature and strain rate, in the hot working of IN 718. The results are validated through the design and optimization of a turbine disk manufacturing process.

11:20 AM

Study on the Corrosion Mechanism of Al2O3-C-ZrO2 Refractory in the Melts of Smelting Reduction with Iron Bath: *Qingcai Liu*[1]; Dengfu Chen[2]; Jing Lin[3]; [1]University of California and Chongqing University, PRC, Dept. of Mech. and Aero. Eng., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA; [2]Chongqing University, Sch. of Matls. Eng., Chongqing 400044 PRC; [3]Chongqing Jianzhu University, Dept. of Appl. Sci., Chongqing 400045 PRC

The corrosion mechanism and corrosion rate of Al2O3-C-ZrO2 refractory in the melts of smelting reduction with iron bath have been studied by rotary immersion and static immersion test method. The key factors that affect the interaction between melts and refractory were also investigated in detail. The results shown that the corrosion rate of Al2O3-C-ZrO2 serials refractory was decreased with the ZrO2 increasing when ZrO2 content among 3% to 10%. Ferrous oxides concentration in melts, rotary speed of the refractory sample in melts and bath temperature were the main factors affecting corrosion rate and corrosion mechanism. The micro-structure and composition change behavior of the refractory in the corrosion process were also studied.

11:45 AM

Synthesis of Highly Oriented Zr(Y)O2 Films by Sol-Gel Method: *Hiroshi Yamamura*[1]; Noritaka Horii[1]; Katsuyoshi Kakinuma[1]; [1]Kanagawa University, Faculty of Eng./Dept. of Appl. Chem., 3-27-1 Rokkakubashi, Yokohama, kanagawa-kenn 221-8686 Japan

ZrO2 and Y2O3-doped ZrO2 gel films were obtained by spin coating of sol from Zr-n-propoxide, n-propanol, acetylacetone, nitric acid, and water on a silica glass as a substrate. The dried gel films crystallized to monoclinic, tetragonal, cubic and their mixed phases, depending on the chemical compoisition of the sol, firing temperature, and coating cycles. The ZrO2 films with highly preferential orientation were successfully obtained from a solution containing a large amount of n-propanol. The highly oriented ZrO2 film was found to have high transparency compared with the non-oriented films. The electrical conductivity of the 8 mol% Y2O3 doped ZrO2 film was also measured.

Thin Films and Coatings-I

Tuesday AM Room: DaVinci 1
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: S. Govindarajan, Motorola, Inc.; Osamu Takai, Nagoya University, Dept. of Matls. Process. Eng.

8:30 AM Keynote

Advanced Processing for the Creation of Functional Thin Films Using Laser Ablation: *Tomoji Kawai*[1]; [1]Osaka University, ISIR-Sanken, 8-1 Mihogaoka, Ibaraki 567-0047 Japan

The Pulsed Laser Ablation method using multi-targets is extremely useful for the creation of functional multi-layers and variety of artificial lattices. The topic of the present talk is on the creation of "Function harmonized artificial lattices by laser ablation" made of the combined magnetic, ferroelectric and superconducting layers. The outline of the talk is: features of Pulsed Laser Deposition method (Laser Molecular Beam Epitaxy) including characterization of thin films by RHEED and STM/AFM; creation of magnetic, dielectric and superconducting artificial lattices by Pulsed Laser deposition; and novel properties of function harmonized artificial lattices; especially on the magnetic and magnetoresistive artificial lattices made of 3d transition metal oxides.

9:00 AM

Mesoporous Silica Coatings Through Triblock-Copolymer Templated Processing: *Jose M. Gomez-Vega*[1]; Atsushi Hozumi[2]; Hiroyuki Sugimura[1]; *Osamu Takai*[1]; [1]Nagoya University, Dept. of Matls. Process. Eng., Grad. Sch. of Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan; [2]National Industrial Research Institute of Nagoya, 1-1 Hirate-cho Chikusa-ku, Nagoya, Aichi 462-8510 Japan

In this work, we report on the preparation of mesoporous silica-based coatings on various substrates: silicon, soda-lime glass and titanium, by a spinning method. Tetraethyl ortho silicate (TEOS) was selected as the silica precursor, and amphiphilic triblock copolymers (poly ethylene-propylene-ethylene oxides type) as the surfactant micellar template or structure-directing agents. Cetyl trimethyl ammonium chloride (CTACl) was also used for comparison. The effect on the mesoporosity of the type of substrate and type and concentration of the templating agent has been investigated. Since mesoporous materials are very interesting as hosts of active molecules that give them specific abilities, an special attention was focused on the fabrication of mesoporous materials highly functionalized (high density of silanols on their inner porous surface) capable to anchor the guest molecules.

9:25 AM

Friction and Wear Performance of Unbalanced Magnetron Sputter-Deposited Diamond-Like Carbon Multilayer Coatings: *Eiji Iwamura*[1]; [1]Kobe Steel Limited, Matls. Rsch. Lab., 1-5-5 Takatsukadai Nishi-ku, Kobe, Hyogo-ken 651-2271 Japan

In various tribological applications, it is necessary for diamond-like carbon (DLC) coatings to have good wear resistance, a low friction coefficient and adequate adhesion in a sufficient level of

combination. In this study, functional DLC multilayered structures were examined with respect to the relationships between microstructures and film properties. Stacking structures with more than a hundred layers of two types of DLC films were prepared by unbalanced magnetron sputtering. It was found that the DLC microstructure was characterized by features of graphite clusters and played an important role in tribological performance. Controlling the size and preferred orientation of the graphite clusters in each DLC layer, the multilayered coatings possessed superior properties to homogenous DLC coatings.

9:50 AM

Preparation of YSZ Thin Films by RF Plasma-Enhanced Metal Organic Chemical Vapor Deposition: *Takashi Nishimura*[1]; Takamichi Ichikawa[1]; Nagahiro Saito[1]; Akio Fuwa[1]; [1]Waseda University, Dept. of Matl. Sci. and Eng., 3-4-1 Okubo, Tokyo 169-0027 Japan

Yttria-stabilized zirconia(YSZ) has high potential to be used as an electrolyte of solid-oxide fuel cell (SOFC). However, the high generation-efficiency of SOFC has not been yet achieved due to the high internal resistance of bulk electrolyte. The resistance becomes lower with the decrease of electrolyte thickness. Thus, the decrease in electrolyte thickness leads to lower internal resistance. The aim of this study is as follows: (a) preparation of the thin films of YSZ by inductively-coupled RF plasma-enhanced metal organic chemical vapor deposition using Zr(t-OC4H9)4,Y(DPM)3 and oxygen, (b) the relationship between the structural characterizations and the process parameters.

10:15 AM Break

10:30 AM

Study on Corrosion Resistance of Electrodeposited RE-Ni-W-P-SiC Composite Coating: *Guo Zhongcheng*[1]; Yang Xianwan[1]; [1]Kunming University of Technology and Science, Dept. of Metall., Kunming, Yunnan 650093 China

Corrosion resistance of RE-Ni-W-P-SiC composite coating and 316L stainless steel in corrosive media of H_2SO_4, HC1, H_3PO_4 and $FeC1_3$ solutions at different concentrations by means of immersion methods and anodic polarization curves obtained by DHZ-1 electrochemical synthetic tester and HDV-7 transistor constant potentiometer. Immersion experiment results show that corrosive rate of RE-Ni-W-P-SiC composite coatings as-deposited in HC1 solutions increases with the rise of HC1 concentrations, on the contrary, the corrosive rate of the composite coatings after heat treatment decreases with increasing the HC1 concentrations. The corrosive rates of the coatings as-deposited and after heat treatment in H_2SO_4 and H_3PO_4 solutions respectively decrease with the rise of H_2SO_4 and H_3PO_4 concentrations. The corrosive rate of the deposits as deposited in $FeCl_3$ solutions decreases with increasing $FeC1_3$ concentrations, while the rate of the composite coatings after heat treatment increases with the rise of $FeC1_3$ concentrations. In addition, the corrosive rate of 316L stainless steel in the corrosive media of H_2SO_4, HC1, H_3PO_4 and $FeC1_3$ solutions respectively at different concentrations increases with rising of concentrations. Furthermore, the corrosive rate of 316L stainless steel in the corrosive media of H_2SO_4, HC1, H_3PO_4 and $FeC1_3$ solutions respectively is much greater than that of the RE-Ni-W-P-SiC composite coatings as-deposited and after heat treatment in the same corrosive media. Anodic polarization curves of RE-Ni-W-P-SiC composite coatings as-deposited and after heat treatment in media of H_2SO_4, HC1, H_3PO_4 and $FeC1_3$ solutions at different concentrations show that corrosion resistance of RE-Ni-W-P-SiC composite coatings after heat treatment is superior to that of RE-Ni-W-P-SiC composite coatings as-deposited in the beginning corrosive time, but the former is inferior to that of the latter with increasing corrosive time. Because there is a layer of film of oxidation in the surface of RE-Ni-W-P-SiC composite coatings after heat treatment, the film of oxidation is dissolved firstly in the corrosive media, and then the RE-Ni-W-P-SiC composite coatings are corroded. In this way, the corrosion resistance of RE-Ni-W-P-SiC composite coatings after heat treatment has advantage over that of RE-Ni-W-P-SiC composite coatings as-deposited in the beginning corrosive time. Supported by Natural Science Foundation of Yunnan Province (95B11-5).

10:55 AM

Effect of Gas Flow on Ion Carburizing of Steel: Naotatsu Asahi[2]; Hirotsugu Takeuchi[3]; Yoshiyuki Funaki[3]; Khoo Khyou Pin[1]; Ryoichi Urao[1]; [1]Ibaraki University, Sch. of Eng., 4-12-1, Nakanarusawa, Hitachi, Ibaraki 316-0033 Japan; [2]Aida Engineering Company, Simomarusawa, Sagamihara, Kanagawa 229-1134 Japan; [3]Nihon Denshi Kogyou Company, Miyashita, Sagamihara, Kanagawa 229-1112 Japan

Steel is used to be ion carburized with mixing gas of methane and hydrogen. In carburizing, carbon in the methane decomposes and deposits on the steel by plasma, so methane gas flow may be in strong influence with ion carburizing of the steel. Nozzles for carburizing gas are set near the sample of steel to investigate the relation between methane gas flow and carburizing of the steel. The surface and section of carburized steel is observed with SEM. The hardness was measured after ion carburizing and quenching. Precipitate was determined by XRD. The steel is carburized deeper near the carburizing gas flow nozzle than far from the nozzle. Hard surface layer was formed by quenching of ion carburized steel.

11:20 AM

Study on Microstructure of Electrodeposited RE-Ni-W-P-SiC Composite Coating: *Guo Zhongcheng*[1]; Zhu Xiaoyun[1]; Zhai Dacheng[1]; Yang Xianwan[1]; [1]Kunming University of Science and Technology, Dept. of Metall., Kunming, Yunnan 650093 China

The components and microstructure of the RE-Ni-W-P-SiC composite coating are analyzed by means of EPXDS, SEM and XRD. The results show that the composite coating containing RE 5-14wt%, SiC 4-7wt%, P 12-15wt% and W 5-6wt% is obtained by use of appropriate bath composition and plating conditions. The composite coating as deposited is amorphous and it becomes mixture when heated temperature is raised from 200°C to 400°C. However, the composite coating is crystal when temperature is over 400°C. Scanning electron microscopy indicates that the heat treatment temperature has no effect on the surface morphologies of the RE-Ni-W-P-SiC composite coating. This is to say that the composite coating has a better heat stability of microstructure and high temperature oxidation.

Electrolytic Processing - I

Tuesday AM Room: Michaelangelo
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Georges Kipouros, Dalhousie University, Dept. of Min. and Metall. Eng., Halifax, Nova Scotia B3J 2X4 Canada; Masazumi Okido, Nagoya University, Dept. of Matls. Sci. and Eng.

8:30 AM Keynote

Electrochemical Implantation to Form Functional Films in Molten Salt Systems: *Yasuhiko Ito*[1]; [1]Kyoto University, Dept. of

Fund. Energy Sci., Grad. Sch. of Energy Sci., Sakyo-ku, Kyoto 606-8501 Japan

Generally speaking, surface film formation by molten salt electrochemical process can be divided into the following three categories: (1) single element film formation on the electrode by electrodeposition, (2) compound film formation on the electrode by electro-codeposition of more than two elements, and (3) compound film formation on the substrate electrode by interdiffusion of substrate element and electrochemically deposited species and/or by direct electrochemical implantation of the component element of the film into the substrate. This talk will be concentrated on the topics of category (3), especially on the electrochemical implantation. Typical example can be seen in the electrochemical implantation of nitrogen atom into Ti metal to form TiN surface film. Another example is the formation of various kinds of intermetallic compound film including Ni_2Y, Ni_2Sc and $LaNi_5$ by the electrochemical implantation of lanthanoid elements into Ni electrode. By combining these two methods, Sm-Fe-N ternary material film can also be formed. The principle and application of the "electrochemical implantation" will be discussed in detail with examples above as well as others also obtained very recently in the author's laboratory.

9:00 AM

Electrodeposition Behavior of Zn-Cr Alloys from Methanol Baths: *Tetsuya Akiyama*[1]; Shigeo Kobayashi[1]; Toshiaki Tsuru[1]; Takeshi Ohgai[2]; Hisaaki Fukushima[2]; [1]Kyushu Sangyo University, Fac. of Eng., Dept. of Indust. Chem., 2-3-1 Matsukadai, Higashi-ku, Fukuoka 813-8503 Japan; [2]Kyushu University, Fac. of Eng., Dept. of Matls. Sci. and Eng., 6-10-1 Hokozaki, Higashi-ku, Fukuoka 812-8581 Japan

Electrodeposition behavior of Zn-Cr alloys from the methanol baths was investigated and was compared with that from aqueous baths. The results obtained were as follows; (1) In the case of low Cr3 concentration in the methanol baths, Cr was incorporated as trivalent state in the deposits. However, Zn-Cr alloys of high Cr content were electrodeposited from the baths having higher Cr3 concentration. (2) The partial polarization curve of Zn was significantly shifted to less positive direction by the simultaneous discharge of Zn2 and Cr3, while the electrodeposition of Cr from the alloy plating baths occurred at the same potential as the one from the single Cr plating bath. (3) The investigation of the effect of water addition to the metahol baths showed that the chemical state of Cr in the deposits changed from metallic to trivalent state with an increase in water content in the baths.

9:25 AM

Recovery of Molybdenum and Rhenium from Sulfide Concentrate by Electro-Oxidation and Precipitation: Tsembel Darjaa[1]; Toru H. Okabe[2]; *Yoshiaki Umetsu*[2]; [1]Tohoku University, Grad. Sch. of Eng., 2-1-1 Katahira Aoba-ku, Sendai, Miyagi 980-8577 Japan; [2]Tohoku University, Instit. for Adv. Matls. Process., 2-1-1 Katahira Aoba-ku, Sendai, Miyagi 980-8577 Japan

With the purpose of extracting molybdenum and rhenium from molybdenum sulfide concentrate, electrolysis of slurry, solvent extraction and precipitation operations were carried out. The slurry of the concentrate in 10 mass% NaCl aqueous solution was continuously circulated into a vertical type 10 stack bipolar electrolytic cell consisted of carbon electrode disks, and the concentrate was dissolved by electrochemically generated oxidizers at 313 K. The current efficiency for molybdenum dissolution was 73 % after 10 hours electrolysis, and the leach solution was found to contain 0.2 mol/l of molybdenum and $4x10^{-5}$ mol/l rhenium. This result shows the dissolution speed is more than 8 times faster than that obtained using a single cell. The leach solution from electro-oxidation was then subjected to a solvent extraction for rhenium, which is recovered as sulfides. After rhenium separation, molybdenum was precipitated by pH adjustment, and solid $MoO_3 H_2O)_x$ crystals, free of sulfate, were obtained from the leach solution containing large amount of SO^{2-}.

9:50 AM

Experimental Study of High Current Density Electrolysis at Tamano Refinery: *F. Furuta*[1]; K. Haiki[1]; M. Hashimoto[1]; Y. Komoda[1]; [1]Hibi Kyodo Smelting Company Limited, Tamano Smelt. and Refine., 6-1-1, Hibi, Tamano City, Okayama Pref 706-8511 Japan

Tamano Smelter and Refinery started its operation in 1972 with high current density electrolysis (350A/m2 of maximum) applying PRC technology. After the start up, four times of expansion have been done, and the cathode production capacity has increased from 84,000 t/y to 191,000 t/y. However, recent refining technology with permanent stainless steel cathode enabled to increase current density up to 300 A/m2 or more without PRC method. In order to increase current density more than 350 A/m2, experimental work using commercial size cell has been carried out at several conditions, such as current density, PRC conditions, electrolyte composition and electrode condition. As a result, it was found that high quality (LME grade) cathode can be produced at such a high current density as 405 A/m2.

10:15 AM Break

10:30 AM

High Throughput of Cathode Copper by New Anode Body Press Machine at Onahama No.1,2 Tankhouse: *Koji Iwami*[1]; Kiyotaka Abe[1]; [1]Onahama Smelting and Refining Company Limited, Onahama Div., 1-1 Nagisa, Onahama, Iwaki, Fukushima 971-8101 Japan

Onahama Smelter and Refinery has been operated since 1965 and currently produces 243,000 tpy of cathode copper. The Onahama tankhouse consists of conventional-type No.1,2 tankhouses with the capacity of 123,000 tpy and No.3 tankhouse with 120,000 tpy, adopting jumbo tank system, using hazelett anode respectively. In October, 1996, for the purpose of improving the anode verticality, new anode copper body press machine has been installed in No.1,2 tankhouses, by increasing Walker type anode copper weight by 35kg to 365kg. Consequently, it is possible to raise current density from 191 to 222 A/m2 and the No.1,2 tankhouse capacity was increased by 15,000 tpy to 138,000 tpy. At present, the refining capacity is 258,000 tpy in total including No.3 tankhouse. This figure is the largest one in Japan.

10:55 AM

Anomalous Codeposition Behavior of Fe-Ni and Zn-Ni Alloys from Methanol Baths: *Shunsuke Kawano*[1]; Takeshi Ohgai[1]; Shigeo Kobayashi[2]; Hiroaki Nakano[1]; Toshiaki Tsuru[2]; Tetsuya Akiyama[2]; Hisaaki Fukushima[1]; Kenji Hara[1]; [1]Kyushu University, Fac. of Eng., Dept. of Matls. Sci. and Eng., 6-10-1 Hakozaki, Higashi-Ku, Fukuoka 812-8581 Japan; [2]Kyushu Sangyo University, Fac. of Eng., 2-3-1 Matsukadai, Higashi-ku, Fukuoka 813-8503 Japan

Electrodeposition behaviors of Fe-Ni and Zn-Ni alloys from aqueous solutions are known to be the anomalous type in which less noble metals are deposited preferentially. In this paper, the electrodeposition behaviors of both systems from methanol baths were investigated and compared with those from aqueous solutions. In Fe-Ni system, the significant suppression of the deposition of more noble Ni was observed over a wide range of electrolysis conditions using both methanol and aqueous solutions. However, the electrodeposition behavior of Zn-Ni alloys from

the methanol baths showed the feature of normal type, which was completely different from that from aqueous solutions. Therefore, it was suggested that the anomalous codeposition behavior should be explained by the mechanism peculiar to the alloy system.

11:20 AM
Effect of Electrolytic Conditions on the Crystal Orientation of Electrodeposited Zinc: *Masao Kurosaki*[1]; Norimasa Yamasaki[1]; [1]Nippon Steel Corporation, Steel Rsch. Labs., 20-1 Shintomi, Futtsu-city, Chiba 293-8511 Japan

The crystal orientation of electrodeposited Zn is an important factor which affects the appearance, formability and corrosion resistance of electrogalvanized steel. Reported so far as items affecting Zn crystal orientation include electrolytic conditions, electrolyte chemical composition and additive elements. In this study, we investigated the effect of different electrolyte chemical composition, flow velocity and current density on the crystal orientation. At the same time, we evaluated the effect of the flow velocity on the mass transport based on the polarization measurement in which Cu was used as the tracer. As a result, it has been clarified that the ratio of the Zn basal plane to the peak intensity of x ray, namely, %Zn (00 x 1) = I Zn(00 x 1)/ΣI Zn(hk x 1) X 100 can be uniformly explained by overvoltage which can be simplistically calculated by the formula: (current density) x (average flow velocity)$^{-0.5}$. In other words, the concentration overvoltage dominantly governs the crystal orientation of electrodeposited Zn.

Aqueous Processing - I

Tuesday AM Room: Parc Ballroom III
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Hani Henein, University of Alberta; Yasuhiro Awakura, Kyoto University, Dept. of Matls. Sci. and Eng.

8:30 AM Keynote
Aqueous Processing of Materials-Where is Our Expertise Taking Us?: *Fiona M. Doyle*[1]; [1]University of California, Dept. of Matls. Sci. and Eng., 551 Evans Hall, #1760, Berkeley, CA 94720-1760 USA

Aqueous processing is a latecomer to the arena of materials preparation. Until comparatively recently, aqueous processing routes were principally used for bulk raw materials and chemicals, rather than for advanced materials with controlled composition, microstructure and properties. The rich capabilities of aqueous processing are rapidly changing this situation. Aqueous processing unit operations are now used in the preparation of many materials, for example in the production and processing of fine ceramic particles, the production of integrated circuits, and the production of materials for biotechnological applications. Future applications, such as the processing of polyelectrolyte multilayers and biomimetic materials will continue this trend. Selected unit processes are explored in terms of the fundamental aqueous processing principles that are the foundation of successful processing, and the future contributions that could be made by the aqueous processor.

9:00 AM
Direct Fabrication of LiCoO2 Films on Papers by Electrodeposition: *Takeshi Fujiwara*[1]; Yoshinori Nakagawa[1]; Seung-Wan Song[1]; Ryo Teranishi[1]; Tomoaki Watanabe[1]; Masahiro Yoshimura[1]; [1]Tokyo Institute of Technology, Matls. and Struct. Lab., Ctr. for Matls. Des., 4259 Nagatsuda, Midori, Yokohama, Kanagawa 226-8503 Japan

We have tried a new method to deposit cobalt oxide films onto the porous films (membrane-filters and paper) by an electorodeposition method. The porous substrate films were placed between a LiOH solution and a CoSO4 solution and treated under electrical currents of ~0.2mA/cm2 using carbon cathode and anode. Effects of temperatures, flow rates and electrical currents have been investigated. We found that LiCoO2 films and Co3O4 films were deposited at 120°C, while CoOOH films were deposited at 80°C.

9:25 AM
Chemical Corrosion Resistance Coating on Aluminum Substrates by the Sol-Gel Process: Shinji Hirai[1]; *Kazuyoshi Shimakage*[1]; Kenji Wada[2]; [1]Muroran Institute of Technology, Dept. of Matls. Sci. and Eng., 27-1 Mizumoto-cho, Muroran-shi, Hokkaido 050-8585 Japan; [2]National Institute for Research in Inorganic Materials, Tsukuba Japan

To improve the corrosion resistance to salt, acid and alkaline attack, a sol-gel film using zirconium tetra-n-butoxide as a starting material has been coated directly to the surface of aluminum substrates by repeating one or twice dip-coating process. The heat treatment at 573K or the UV-irradiation at an ambient temperature after the dipping-withdrawing of aluminum substrate into the sol was performed to form dense coating films of an amorphous zirconiumoxide. In both densification methods of heat treatment and UV-irradiation, corrosion resistance of coating film measured by salt spray test decreased with increasing the surface roughness of substrates. Then, the coating films prepared by a boiling treatment after both densification treatments indicated excellent corrosion resistance to salt attack, regardless of the surface roughness. The surface area of the coating film corroded for 3600ks in salt spray test decreased in the order of sol-gel coating film < chromate film < boehmite film. Furthermore, the mass loss of coating substrates in the copper-accelerated acetic acid salt spray test for 172.8ks decreased with increasing the repetition number of dip-coatings, and the acidic corrosion resistance of coating film formed by over twice dip-coatings was also more excellent than that of chromate film. In addition, the alkaline corrosion resistance of coating film in the electromotive force measurement test was superior to approximately 2.7 times than that of usual anodic oxide films anodized in a sulfuric acid electrolyte. In the measurement of the contact angle against water on coating film surface, sol-gel coating films prepared by both densification methods indicated the water-repellent surface, regardless of surface roughness of substrates. On the contrary, coating films formed on the coarse aluminum substrate of 0.56~2.68 μ m in roughness demonstrated the water-avid surface having contact angle of approximately 3° by a boiling treatment after the heat treatment.

9:50 AM
Hydrothermal Corrosion of TiN, TiAl and CrN Films on SUS-304 in 100 MPa Water: *Sergiy Korablov*[1]; Masahiro Yoshimura[1]; [1]Tokyo Institute of Technology, Matls. and Struct. Lab., Ctr for Matls. Des., 4259 Nagatsuta, Midori, Yokohama, Kanagawa 226-8503 Japan

Hydrothermal corrosion of TiN, TiAlN, and CrN PVD films (3μm thick) deposited on SUS-304 substrate was studied in 100 MPa water at 20-950°C temperature range up to 750h. The beginning temperature of chemical reaction was determined as 200° for TiN and TiAlN and 500°C for CrN films, which are quite low

even for air oxidation of those powders. Very thin oxide scales of TinO2n-1 homologous series phases were formed above 300°C. Anatase TiO2 is observed for TiN and TiAlN films at 400°C. The feature of hydrothermal corrosion of CrN films is CrNxOy phase formation. Distinguishable weight changes were observed at higher temperature than 700°C for TiN, 750°C for TiAlN and 800°C for CrN, where hydrothermal corrosion kinetics were constructed to calculate activation energy of hydrothermal corrosions. On the base of obtained data, hydrothermal corrosion resistance were as follows: TiN-TiAlN-CrN. Facetted oxide crystals were formed at higher temperature than 500°C centigrade for TiN, TiAlN and 850°C for CrN.

10:15 AM Break

10:30 AM

Precipitation Stripping of Samarium Oxalate Particles: *Yasuhiro Fukunaka*[1]; Toshihiro Kuzuya[1]; Masakazu Niinae[1]; Eishi Kusaka[1]; Ryuuji Ishii[1]; [1]Kyoto University, Dept. of Energy Sci. and Tech., Kyoto 606-8501 Japan

As a series of research on the precipitation stripping of rare earth or transition metal oxalate particles, samarium oxalate particles was precipitated from samarium-loaded D2EHPA solution with a conventional impeller. The effect of intensified mixing induced by ultrasonic irradiation was also examined. Morphological variation as well as size distribution of precipitated samarium oxalate particle were observed with processing factors such as pH value, C2H2O4 concentration, samarium metal concentration and D2EHPA concentration under ultrasonic irradiation. The needle-like precipitate was identified as Sm2(C2O4)3·10H2O by XRD. C2H2O4 concentration, samarium concentration and D2EHPA concentration did not significantly influence its shape. However, the size reduction effect caused by ultrasonic irradiation was not so evident as observed in cobalt oxalate precipitation. Ultrasound irradiation operation may prevent from coagulation of precipitated oxalate particles. Plate-like morphology appeared by addition of HCl to aqueous solution.

10:55 AM

Production of Synthetic Rutile from Egyptian Ilmenite Ore by a Direct Hydrometallurgical Process: *I. A. Ibrahim*[1]; E. A. Abdel-Aal[1]; A. A. Ismail[1]; A. K. Ismail[1]; [1]Central Metallurgical R&D Institute, P.O. Box 87, Cairo, Helwan, Egypt

Synthetic rutile for paints industry is conventionally produced by a combined smelting and leaching process. Smelting process is an energy intensive step and represents economic burden in view of the increasing energy costs. Egyptian ilmenite ore containing 40% TiO_2 and 38% total Fe was thus directly leached with sulphuric acid to produce eventually synthetic rutile. The process involves digestion of the ore with sulphuric acid whereby both TiO_2 and Fe are dissolved. The solution was then treated with iron powder to convert Fe^3 to Fe^2 to avoid co-precipitation of iron during the hydrolysis step. After solid/liquid separation and clarification, Fe was crystallised as ferrous sulphate hydrate by chilling. The titanium pregnant liquor was concentrated by evaporation and then hydrolysed to precipitate hydrated titanium dioxide which was then calcined to produce synthetic rutile containing 98.6 TiO_2 and 0.58% Fe_2O_3. The optimum conditions of all the processing stages were determined.

11:20 AM

Room-Temperature Direct Patterning of Crystalline Inorganic Materials on Organic Substrates by On-Site Solution Reaction: *Ryo Teranishi*[1]; Takeshi Fujiwara[1]; Tomoaki Watanabe[1]; Masahiro Yoshimura[1]; [1]Tokyo Institute of Technology, Matls. and Struct. Lab., 4259 Nagatsuta Midori, Yokohama 226-8503 Japan

Crystalline inorganic materials such as metal oxides and metal sulfides thin film have been fabricated by direct patterning with solution reactions. A drop-on-demand jet printer is particularly interesting because it is a non-contact technique. The projected drop of ink travels directly through air to the substrate, thus jet printer allows us to prepare not only 2-D structures in the form of dots, lines, or films but also 3-D ones deposits freely in the right place on the substrate. The crystalline thin films of cadmium sulfide and lead sulfide were fabricated at a room temperature without any firing/heating treatments. In addition the optional line drawing of these materials in the width of 100 micrometer have been succeeded. It means that this method has the capability to produce direct patterning of the crystalline inorganic materials on various substrates at a room temperature.

11:45 AM

The Extraction and Separation of Nickel, Cobalt and Copper Using Solvent Impregnated Resin: *Masayasu Kawahara*[1]; [1]Kumamoto University, Mech. Eng. and Matls. Sci., 2-39-1 Kurokami, Kumamoto 860-8555 Japan

Amberlite XAD-4 resin is used for catching organic substances in the water. LIX84-I has the good ability to extract various metallic elements, and is used in the field of solvent extraction. In this study, solvent impregnated resin (SIR) was devised by absorbing LIX84-I to XAD-4, and extraction properties of the SIR for nickel, cobalt and copper were investigated. The extraction behavior of each metal using the SIR was similar to that of the solvent extraction using LIX84-I. Copper was selectively extracted at the pH of 3, and nickel and cobalt were simultaneously extracted at the pH of 9. The extraction ability of the SIR for metals improved with using finer XAD-4 in particle size and higher concentration of LIX84-I at the making stage of the SIR. Although the stripping of cobalt from the solvent of LIX system is difficult, cobalt was easily stripped in the case of the SIR.

Composite Materials - II

Tuesday AM Room: DaVinci 2&3
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Dennis R. Readey, Colorado School of Mines; Hideharu Fukunaga, HiroshimaUniversty, Dept. of Mech. Eng.

8:30 AM Keynote

Synthesis of Advanced Composite Oxides Thin Films by the Liquid Phase Deposition Method from Aqueous Solution: *Shigehito Deki*[1]; [1]Kobe University, Depts. of Chem. Sci. and Eng., Rokko-dai-cho, Nada-ku, Kobe, Hyogo Pref. 657-8501 Japan

We have been developing a new wet process for the synthesis of various kinds of oxide thin films on substrates from aqueous solution. The process was named as the LPD, Liquid Phase Deposition, method. In this process, the chemical equilibrium, between a metal fluoro-complex and a metal oxide in the solution, is one of key reactions for the film deposition. By adding of the reagent to form more stable complex with fluoride anions, such as metal Al or boric acid etc, the hydrolysis equilibrium is sifted toward the oxide side, and the film-formation on a substrate is brought on. This process can be applied to an on-site synthesis of the functional materials in the thin film technology. In this paper, some current topics on the synthesis of the composite thin oxide films for the advanced materials will be presented.

9:00 AM

Production and Assessment of TiC Reinforced Al-Fe-V-Si Alloy: *Ali Kalkanli*[1]; [1]Middle East Technical University, Metall. and Matls. Eng. Depts., Inonu, Ankara, Bulvari 06531 Turkey

A series of rheocast and sinter specimens were produced and investigated to compare the effect of process conditions on the final mechanical properties of the FVS012 alloy with TiC addition. Two processing techniques were performed to obtain tensile test specimens. First, rapidly solidified FVS-0812 alloy originally developed by Allied Signal Inc. produced by planar flow casting in flakes and fibre forms were mixed with TiC powder, homogeneously mixed at semi solid state, and subsequently solidified under 100 MPa pressure. Secondly, the same alloy powders produced by air atomization were pressed at 375 MPa pressure and sintered at 565-595°C temperature range then sintered at vacuum condition at a pressure of 0.07 torr. Fine and homogeneous distribution of Tic particles with silicide dispersoids were examined. Wear and mechanical tests were performed to the bar shape specimens. These tests were done at 250, 300, 315, and 425°C. Higher strength values were obtained at specimens reinforced with TiC. The silicides were so stable and very little coarsening were observed at 450°C. The presence of quasicrystalline I-phase and silicides were investigated.

9:25 AM

New Process for the Fabrication of Cast Aluminum Alloy Matrix Composites: *Toshiaki Kimura*[1]; Hideki Yamaura[1]; Hideo Nakae[2]; [1]Hitachi Metals, Cast. Tech. Rsch. Lab., 11 Kinugaoka, Moka, Tochigi-ken 321-4367 Japan; [2]Waseda University, Dept. of Matls. Sci. and Eng., 3-4-1 Okubo, Shinjyuku, Tokyo 169-8555 Japan

In 1968 J. N. Reding et al. suggested processes for fabricating composites. According to their patent, decompression atmosphere, which occurs during the reaction between magnesium and oxygen and/or nitrogen in the closed system, causes the spontaneous infiltration of melt into narrow spaces such as ceramic preforms. However, they did not report the proof of this phenomenon or the industrial manufacturing method. Therefore this research measured the pressure change among SiC particles surrounding molten aluminum. As a result, the pressure reduction during the Mg reaction was identified. Moreover, the existence of Mg3N2, which improves the wettability between Al alloy melt and SiC particles on the interface, was observed on the surface of particles. Applying these two phenomena, a new process for the manufacturing of cast discontinuously reinforced aluminum composites (DRA) was developed. This new method is expected to produce DRA without additional pressure.

9:50 AM

A Study on the Mechanical Behavior of ZA-27 Alloy/Quartz Particulate Composites: *P. V. Krupakara*[1]; S. Manjuanth[1]; M. Krishna[1]; Jayagopal Uchil[2]; [1]R.V. College of Engineering, Dept. of Chem., Bangalore, Karnataka 560059 India; [2]Dept. of Matl. Sci., Mangalgangothri, Mangalore

There have been many pioneering efforts over the last forty years in the development of metal matrix composites (MMC's) of new engineering materials, having improved properties, lightweight characteristics and competitive costs compared to traditional materials. Following recent advancements in casting technology and the use of melt stirring devices by foundries, discontinuously reinforced MMC's where prominent alloys are reinforced with particulates or short fibres are being employed in the production of castings targeted for automotive and aerospace applications. The ZA-27 alloy has a high strength compared to zinc or aluminum alloys with a lower melting temperature. The main objective of the present research is to study the effect of quartz particles on the mechanical behavior of ZA-27 alloy composites. SEM analysis of the fracture surfaces, as well as the subsurfaces, are used to explain the observation made. The results showed that the modulus of elasticity, ultimate tensile strength and hardness of the composite gradually increased in the weight percentage of reinforcement. However, the overall ductility decreased with an increase in reinforcement.

10:15 AM Break

10:30 AM

Corrosion Studies of Hematite Reinforced Aluminium-6061 MMC's at Elevated Temperatures: *S. C. Sharma*[1]; N. Shanmukha[1]; M. Krishna[1]; [1]R.V. College of Engineering, Dept. of Mech. Eng., Rsch. and Dev., Bangalore 560059 India

Observations made during the corrosion of aluminium [6061] alloys dispersed with hematite particles were analyzed. The percentage of hematite in the composite was varied from 0 to 6% in steps of 2% by weight and subjected to a temperature range of 200 to 500°C. The vortex method was employed to prepare the composite. With in the experimental temperature range a parabolic weight gain was observed. The weight gain as a function of oxidation time becomes linear after an initial period. As the corrosion was initiated, an oxide scale was found whose morphology depends on temperature, cooling rate and sub-scale formation at the interface between the matrix and reinforcement. The morphology and phase content of the product of oxidation were characterized by EDAX. Detailed analysis by SEM showed that the oxide scales were not homogenous through out, but exhibited several layers, which differ in microstructure and composition with increase in thickness. The presence of hematite particulate reinforcement has very little effect on the corrosion behavior. The interface corrosion rate was found to be higher than the other regions. The exposure time of specimens to corrosion was 1000 minutes at various temperatures [200 to 500°]. The present paper also emphasizes on various reactions taking place during corrosion and the activation energies involved in the oxidation reactions.

10:55 AM

A Case Study on Corrosion Properties of Garnet Particles Reinforced Aluminium Composites: *V. T. Vijayalakashmi*[1]; Jayagopal Uchil[2]; B. M. Girish[3]; K. H. Thipperudrappa[3]; [1]NMKRV College for Women, Dept. of Chem., Jayanagar, Bangalore, Karnataka 560-011 India; [2]Mangalore University, Dept. of Matl. Sci., Mangalore India; [3]RV College of Engineering, Rsch. and Dev., Mech. Eng. Dept., Bangalore 560059 India

The pitting susceptibility of aluminium metal matrix composite reinforced with garnet particulate has been examined using static weight loss method by exposing the metal matrix composite (MMC) specimen to the action on IN HCl acid at laboratory temperature for different time intervals in conformity with the ASTM69 standards. It was found that the corrosion rate decreased with increasing percentage by weight of reinforcement while the corrosion rate was a function of the matrix alloy but not of the reinforcement material. Further, it was found that the rate of corrosion was dependent on the interface region formed between the reinforcement and the matrix. A comparison of the base matrix with the corresponding composite when subjected to heat treatment for varying periods of 4, 8, and 12 hours respectively was made. The comparison revealed that corrosion rate increased with the duration of the heat treatment and this has established a considerable improvement in corrosion resistance when compared with the base matrix. SEM analysis of the corrosion sur-

face, as well as the subsurfaces are used to explain the observation made.

Powder Preparation and Processing - I

Tuesday PM Room: Dante
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Janice Klansky, Buehler, Inc.; Fumio Saito, Tohoku University, Instit. for Adv. Matls. Process.

2:00 PM Keynote
Morphology Control in Particle Preparation by Aerosol Method: *Kikuo Okuyama*[1]; Manabu Shimada[1]; I. Wuled Lenggoro[1]; [1]Hiroshima University, Dept. of Chem. Eng., Higashi, Hiroshima 739-8527 Japan

Synthesis and application of submicron and nanometer particles have become of major interest lately to establish the manufacturing technologies necessary to produce novel functional materials in electronic and chemical industries. Synthesis of functional materials with desirable physical/chemical properties starts with molecular precursors that are brought to conversion into the product. One of the all-around methods for achieving this conversion is the aerosol process (gas-phase method) such as (i) gas-to-particle conversion process (e.g., chemical vapor deposition (CVD) method) and (ii) liquid-to-particle conversion process (e.g., spray pyrolysis). In this lecture, the recent research by the authors on the theoretical and experimental approaches for the preparation of fine particles by the CVD and spray pyrolysis methods is introduced. First, we present the experimental data depicting the changes in number concentration and size distribution under various preparation conditions of CVD processes. A numerical simulation using the discrete-sectional type general dynamic equation for aerosols undergoing chemical reaction, coagulation and sintering is then described to explain the experimental data. Secondly, we review the preparation of various functional particles for luminescent materials etc. by the spray pyrolysis methods. Also, a numerical simulation model is introduced for the evaluation of the morphology of produced particles. The recent progress of aerosol technology for the measurement of size and number concentration of particles in a suspending state is also introduced in this lecture. Finally, the importance of the process engineering approach for aerosol material production is discussed.

2:30 PM
Combined MA and CCS in the TiH_2-B System: *Sedat Ozbilen*[1]; Abdulkadir Gullu[2]; [1]Gazi University, Metall. Edu. Dept., Fac. of Tech., Teknikokullar, Ankara, Turkey; [2]Gazi University, Mech. Edu. Dept., Fac. of Tech. Edu., Teknikokullar, Ankara, Turkey

Combined unreactive mechanical alloying (MA) and controlled combustion synthesis (CCS) was carried out in the TiH_2-B system. Samples with different TiH_2 levels were studied to investigate the influence of the particle size of reactants on the level of exothermicity of the self-propogating reactions together with the other effects of mechanical alloying on CS technique. In the first step, homogeneous mixtures of samples #1 to #3 were converted into fine, crystalline forms without compound formation. For this purpose, unreactive mechanical alloying for up to 8 hours of time under Ar with forced air cooling to keep the temperature at RT were utilized. MAed powder samples were then examined by XRD and SEM study. Homogeneously mixed, unreactively MAed and then cold-compressed pellets of samples #1-#3 (green compacts) were subsequently combustion synthesized in controlled fashion and in thermoexplosion mode under vacuum to develop Ti-boride compounds formation (TiB and TiB_2 depending on the composition). Thermal analysis under vacuum by DTA was carried out on the green compacts. XRD and SEM investigation were used for the examination of unreactively MAed, cold-pressed, and as-reacted pellets via CS.

2:55 PM
Structural and Mechanical Behavior of Boron Modified Low-Carbon PM Superalloys: *K. K. Sharma*[1]; N. Chary[1]; R. K. Sood[1]; [1]Defense Metallurgical Research Laboratory, Hyderabad, A.P. 500058 India

One of the commonly used measures to reduce the tendency of prior particle boundary precipitation in nickel base PM superalloys is to reduce the amount of carbon below 0.02 wt%; but to have the desired temperature capability of such low carbon PM superalloys, it is necessary to add same other grain boundary strengthener. Addition of boron has been shown to restore the grain boundary strength of low carbon PM superalloys besides restricting the formation of prior boundary networks. In the present study aimed at optimizing the addition of boron in a modified version of an advanced cast superalloy, ZhS6K, three alloy variants with different boron contents, i.e. 0.02, 0.045, and 0.08 wt% were prepared by argon atomization of the vacuum induction melted prealloyed ingots and hot isostatic pressing of the atomized powders. Effects of HIP consolidation temperature on the microstructure and mechanical properties were evaluated for all the three alloy variants. The 0.045% boron alloy HIPed at a temperature close to the γ' solvus was found to be virtually free from intergranular precipitation of borides and eutectic γ/γ'. Structural and mechanical behavior of this alloy variant subjected to 40% deformation has also been examined to demonstrate its potential as a turbine material.

3:20 PM
A New Technology for Production of Two Component Composite Powders with Solvent Extraction-Crystallization Stripping: *Junji Shibata*[1]; Shigeno Matsumoto[1]; Hideki Yamamoto[1]; [1]Kansai University, Fac. of Eng., Dept. of Chem. Eng., 3-3-35, Yamate, Suita, Osaka 564-8680 Japan

Some fundamental studies were carried out in order to produce composite powders comprising of two or three metals by using liquid-liquid extraction and develop the control technology of the particle size. The combination of copper and zinc, and cobalt and samarium was selected as the two component composite powders. Both stripping and crystallization occurred at the same time by emulsifying the organic phase loading the two metals extracted with Versatic Acid 10 and the aqueous solution containing oxalic acid. The effect of aqueous pH, oxalic acid concentration and agitation speed was examined on some properties such as crystallization speed, crystallization percent, particle size and shape of each metal powders obtained in this process. The analysis by using EDX showed that the two metals were distributed uniformly at the same ratio as the organic phase composition.

3:45 PM Break

4:00 PM
Study of Quench Gas Injection for In-Flight Nitridation of Molybdenum Disilicide Powders in an Induction Plasma: *Gervais Soucy*[1]; Mohamed Rahmane[2]; Xiaobao Fan[3]; Takamasa Ishigaki[4]; [1]University of Sherbrooke, Plasma Tech. Rsch. Ctr., Sherbrooke, Quebec J1K2R1 Canada; [2]Universite Hassan II, Phys.

Dept., Fac. Sci. and Tech., Mohammadia 20650 Maroc; [3]Nanomaterials Research Corporation, 2620 Trade Ctr. Ave., Longmont, CO 80503 USA; [4]National Institute for Research in Inorganic Materials, 1-1 Namiki, Tsukuba-shi, Ibaraki 305-0044 Japan

In-flight modification of Molybdenum Disilicide ($MoSi_2$) powders has been performed in a thermal induction plasma reactor. The experimental set-up employed for this present study consisted of three major parts: an induction plasma torch connected to a 50 kW radio frequency (r.f) power supply (3-5 MHz), a water cooled cylindrical reactor (0.25m i.d. and 1m long) and an enthalpy probe system coupled with a mass spectrometer. The operating conditions used were: plasma plate power set at 25 kW, an absolute reactor pressure at 40 kPa, a sheath gas flow at 90 l/min STP Nitrogen and a central plasma gas at 30 l/m STP Argon. The effect of the quench gas on the nitridation of the powders was found to be an important parameter and this work will present the results of varying quench gas flowrates and compositions (without quench gas, with Argon, with Nitrogen and with a mixture of Argon and Ammonia) on the increased nitrogen concentration in the $MoSi_2$ matrix. The results demonstrate that there is an optimal flowrate for each quench gas composition that produces the maximum nitridation. Temperature and concentration profiles measured in the reactor are presented to explain the heat and mass transfer limitations that exist under these conditions.

4:25 PM

Low-Temperature Processing of Ba-Doped Lead Zinc Niobate Powder with Chemical Solution Deposition: *Hisao Suzuki*[1]; Toshitaka Ota[2]; Minoru Takahashi[2]; [1]Shizuoka University, Dept. of Matls. Sci., 3-5-1 Johoku, Hamamatsu, Shizuoka 432-8561 Japan; [2]Nagoya Institute of Technology, Cer. Rsch. Lab., 10-6-29 Asahigaoka, Tajimi, Gifu 507 Japan

Perovskite-type lead zinc niobate (PZN) exhibits excellent electrical properties such as high dielectric constant, high electrostrictive coefficient and superior piezoelectricity. Therefore, PZN ceramics are very promising for high performance electronic devices. However, perovskite phase PZN is not stable. Accordingly, it is very difficult to prepare single phase perovskite PZN ceramics. In this study, A-site cation of lead was partially substituted for barium to stabilize the perovskite phase of Ba-doped PZN powder. In addition, molecular design of an alkoxide precursor was controlled for a low-temperature processing. As a result, molecular design to suppress the lead volatilization during calcination was preferred for a low-temperature processing. Processing temperature for a single phase perovskite was depended on the amount of barium dopant. Doping of 20 mole % of barium resulted in the single phase perovskite at 1173K, lower than that of PZN powder by the atmosphere controlled conventional powder processing at 1423K.

4:50 PM

Syntheses of Zeolite-A and Zeolite-X from Kaolinite Activated by Mechanochemical Treatment: *Wantae Kim*[1]; Qiwu Zhang[1]; Fumio Saito[1]; [1]Tohoku University, IAMP, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

Abstract Not Available

Control and Analysis in Materials Processing - II

Tuesday PM Room: Rubens
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: R. Bautista, University of Nevada-Reno; Shiya Otsuka-Yao-Matsuo, Osaka University, Dept. of Matls. Sci. and Process.

2:00 PM Keynote

Hydrogen Sensor for Molten Metals and its Application to Materials Processing: *Norihiko Fukatsu*[1]; Noriaki Kurita[1]; Teruo Ohashi[1]; [1]Nagoya Institute of Technology, Dept. Matls. Sci. and Eng., Gokiso-cho, Showa-ku, Nagoya 466-8555 Japan

An electrochemical sensing device for hydrogen in molten metal has now been available owing to the development of the proton conducting solid electrolyte made of ceramics of metal oxide. The structure of the sensor and the theoretical limitations of its applications are discussed based on the electrochemical properties of the electrolyte used. The practical performance of the sensor is reviewed from the results of test measurements at the plants of aluminum, copper and silver melting industries. The effectiveness of the sensor to the process control of continuous casting of copper is also discussed.

2:30 PM

Software Sensors for Real Time Optimization of Incoherent Materials Exploitation and Processing: *Giuseppe Bonifazi*[1]; Floriana La Marca[1]; Paolo Massacci[1]; [1]Universita Di Roma, Dept. di Ing. Chim. Matl. Mat. Metall., Via Eudossiana 18, Roma 00184 Italy

Integrated hardware and software (HW & SW) solutions are now very cheap and easy to implement at industrial level in many sectors. These innovations can thus profitably applied in the field of bulk handling and processing especially adopting new processing strategies (detection and analysis of particle features) oriented not only to analyze, on routine bases, both at laboratory ("off-line" control) and processing plant scale ("on-line" control), single particle characters, but those characters resulting and extracted from the "particle system" considered as a whole: adoption of specific sampling procedures (easy and correct acquisition of the images necessary for the analysis); identification of those parameters that extracted from the images can describe the characters concerning the particle (morphological and morphometrical parameters, color and/or textural information of single or bulk particles); procedures able to extract the characters of interest from images (points, lines, 2D and 3d analysis); synthesis of the results and the definition of one or more correlation functions able to establish a correspondence between digital information, extracted from the images, and the bulk materials handling and processing characteristics; definition of suitable control strategies, at different level of interactions with the processes, to apply specific control action on the process itself. In the paper will be analyzed, described and discussed the guidelines that can be considered at the base of the definition of digital imaging based procedures applied to bulk solids physical characterization, both during the exploitation and processing stages.

2:55 PM

Numerical Analysis of Influences of Operational Parameters on Fluid Flow and Heat Transfer in Funnel Type Mold of Thin Slab Caster: *Hoseok Nam*[1]; Jong-Kyu Yoon[1]; Hwa-Soo Park[2]; [1]Seoul

National University, Sch. of Matls. Sci. and Eng., San 56-1 Shinrim-2, Dong Kwanak-ku, Seoul 151-742 Korea; [2]Kookmin University, Sch. of Metallu. and Matls. Eng., 861-1 Chongnung-dong, Songbuk-gu, Seoul 136-702 Korea

In a thin slab casting process, control of fluid flow in mold is particularly difficult due to the high casting speed and large aspect ratio of the mold and the understanding of the fluid flow motion as well as heat transfer in mold is regarded as one of the most important informations because the melt delivery is directly related to the problems of shell growth ununiformity, surface turbulence and mold powder entrapment. A mathematical model has been developed for the coupled analysis of fluid flow, heat transfer and solidification in funnel type mold using finite volume method based on body fitted coordinate and the characteristics of the transport phenomena in the mold of thin slab caster were analyzed by numerical simulation. As a result of simulation, the basic flow pattern is characterized as four large recirculations and two small eddies near the narrow faces of mold which is somewhat different from that of parallel type mold or other conventional slab caster. The heat transfer model accurately predicted the mold positions susceptible to cracking as a result of thermal stress and the predicted shell thickness shows a good agreements with the measured shell thickness on breakout product. We also investigated the influences of some operational parameters such as casting speed, nozzle type, submerging depth and mold width on fluid flow, heat transfer and distribution of solidifying shell and various informations for the determination of optimal operating conditions were obtained.

3:20 PM

Development of Bonded Sm-Fe-N Magnet Using Reduction and Diffusion Method: *Kenji Ohmori*[1]; Atsushi Kawamoto[1]; Shoichi Yoshizawa[1]; Takashi Ishikawa[1]; Kaname Takeya[1]; [1]Sumitomo Metal Mining Company Limited, 3-18-5, Nakakokubun, Ichikawa, Chiba 272-8588 Japan

High performance Sm2Fe17N3 magnet powder is developed using a reduction and diffusion method. Typical remanence, coercivity and maximum energy product obtained are 1.40 T, 1.13 T and 323 kJ/m3, respectively. Decreasing samarium content, oxygen content and the amount of alpha-Fe are found to be the important factors to realize high energy product. High performance injection molded Sm2Fe17N3 anisotropic magnet is also developed using the powder and an original binder system developed in order to mold at low temperature. Typical remanence, coercivity and maximum energy product obtained are 0.77 T, 0.78 T and 103 kJ/m3, respectively in the density of 4.77 Mg/m3. The temperature coefficient of remanence is -0.07%/K. The initial irreversible flux loss is 5% at 100°C at a permeance of two.

3:45 PM Break

4:00 PM

Development of an Automatic Short-Circuit Detector at Hitachi Refinery: *Hidenori Okamoto*[1]; Katuto Itoh[1]; [1]Nippon Mining and Metals, Hitachi Refinery, 1-1-2 Sirogane-Cho, Hitachi-Shi, Ibaraki-Ken 317-0056 Japan

In an effort to further increasing productivity at Hitachi Refinery, Nippon Mining & Metals developed a new automatic short-circuit detector to be used in the tank house. It is used in the inspection of short-circuits between anode and cathode and is designed to inspect the 56 pairs of anode and cathode in a cell by fluxgate sensors at once. It inspects 564 cells, automatically and un-manned, and gathers data to be put into a computer. The development of the detector led to a reduction of manpower and achieved a detection accuracy superior to that of a man-held gauss-meter. In addition, statistical and historical processing of the data enabled early detection and early recovery of the cells with poor efficiency. The new system also contributed to raising the refining capacity and reducing the unit steam consumption, because it is possible to inspect even the cells covered with thermal insulation sheets.

4:25 PM

Determination of Trace Arsenic(III), Arsenic(V), Antimony(III), Antimony(V) in Drain by ICP Equipped with Hydridegeneration System: *Hiroshi Ono*[1]; [1]Mitsui Mining and Smelting Company Limited, Corp. R&D Ctr., 1333-2 Haraichi Ageo, Saitama 362-0021 Japan

By newly developed ICP method, trace amounts of As(III),As(V),Sb(III) and Sb(V) were precisely determined within accuracy of sub ng/ml. As(III) and Sb(III) were separated from As(V) and Sb(V) by extracting into xylene as diethyldithiocarbamate complexes using diethylammonium diethyldithiocarbamate (DDDC), and then back-extracted into nitric acid for a quantitative determination by ICP-AES equipped with hydridegeneration system. The recoveries of As(III) and Sb(III) were satisfactory throughout the extraction with 0.04mol/l DDDC in xylene from 1mol/l hydrochloric acid solution and the back-extraction with 7 mol/l nitric acid. As(V) and Sb(V) were reduced to As(III) and Sb(III) by ascorbic acid and potassium iodide, and their quantities were determined as mentioned above. The detection limits were found to be 0.58ng/ml for As(III), 0.63ng/ml for Sb(III) using 50ml of a sample. The proposed method was applied to analysis of the real sample.

4:50 PM

Electrochemical Studies of ZA-27/Flyash Particulate Composites in Acidic Medium: M. S. Koti[1]; *S. C. Sharma*[1]; [1]R.V. College of Engineering, Dept. of Mech. Eng., Bangalore, Karnataka 560059 India

There have been many pioneering efforts over the last forty years in the development of metal matrix composites (MMC's) as new engineering materials, having improved properties, lightweight characteristics and competitive costs compared to traditional materials. Following recent advancements in casting technology and the use of melt stirring devices by foundries, discontinuously reinforced MMC's where prominent alloys are reinforced with particulates or short fibres and are being employed in the production of castings targeted for automotive and aerospace applications. Metal matrix composites are a new range of advanced materials used mainly for elevated temperature applications where existing materials are not suitable for use. Many of the matrices used in the preparation of MMC's are well known alloys, selected mainly on the basis of their already known mechanical properties, which, in turn can be further enhanced by means of a suitable heat or working treatment. The properties of composites are invariably a compromise between those of its matrix and reinforcement. The present investigation aims at exploring the corrosion behavior of zinc-aluminum-27 alloy dispersed with flyash particles in controlled environments, both in the as-cast as well as the heat-treated conditions. The corrodent used is fresh HCl solution of 1 normal concentration. The composites were found to corrode in the HCl solution. In each test, the corrosion rate in HCl decreases with time, probably because of the presence of aluminum in the zinc alloy. As the flyash content is increased, the composite becomes more corrosion resistant. Heat treatment at 320°C for a few hours also enhances the corrosion resistance of the composite. The composites were tested for corrosion characteristics using the weight loss method in accordance with the ASTM standards. It was found that the composites reinforced with flyash showed better corrosion resistance

than the unreinforced ZA-27 alloy probably due to the presence of inert flyash particles. The results obtained will be discussed in detail in the full length of the paper.

Non-ferrous Alloys & Light Metals - II

Tuesday PM Room: Cervantes
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Mark P. Taylor, Comalco Aluminum; Yasuhiro Miura, Kyushu University, Dept. of Matls. Sci. and Eng.

2:00 PM Keynote
Effect of Processing on Mechanical Anisotropy and Formability of Textured Zircaloy Sheet: *K. Linga Murty*[1]; Susan A. Lloyd[1]; [1]North Carolina State University, Nuc. Eng. Dept., P.O. Box 7909, Raleigh, NC 27695-7909 USA

Zirconium alloys are commonly used in light water reactors as thin-walled tubing, intermediate grids, and channel boxes (for BWR's). The choice of Zr-alloys is based on their relatively high transparency to thermal neutrons and excellent corrosion resistance. These alloys have hcp crystal structure with low c/a-ratio at and below the reactor operating temperatures and exhibit preferred orientations or textures. Resulting anisotropic mechanical properties in turn affect their in-service behavior such as in-pile creep-down of the cladding tubes, creep and growth of channel boxes, etc. In addition, the texture and mechanical anisotropy of the starting materials, from which the final products are fabricated, influence the formability that could have technological significance in the productivity of the components. We report here a study on the mechanical anisotropy of Zircaloy sheet fabricated from two different routes: one from a sheet obtained through sheet-forming process (designated NOK) and the other made by cutting out a large diameter thin-walled tube formed through Pilger mill reduction (designated OK). The Q/A-formability tests consisting of punching a boat-type depression revealed cracking in the NOK sheet (formed through sheet-forming process) while that fabricated through Pilger mill process (OK) was found to be satisfactory. Tensile specimens were made along the rolling and transverse directions, from OK and NOK materials, for determining the mechanical anisotropy at ambient temperature. Square grids were electro-chemically etched on one side of the tensile specimens and changes in grid size along the longitudinal and transverse directions were determined by intermittently photographing the grid pattern following different levels of plastic deformation. Thus, the contractile strain ratios, R and P, which are also the mechanical anisotropy parameters in Hill's formulation for generalized stress, are determined. R={ } and P={ } Here, the subscripts, RD, TD and ND refer to the rolling, transverse and normal (thickness) directions respectively of the sheet. The contractile strain ratios (R and P) define the resistance to wall thinning of an anisotropic material, and the Backofen formability parameter (β) is given by the ratio of the stress along the transverse direction under plane strain loading [strain along the RD zero] and twice the stress along the transverse direction for negative equibixial loading [compressive stress along the RD=tensile stress along the TD]: β= The significance of the β parameter lies in the fact that the higher the β value, the better the formability of the material. The experimental results do indicate that the OK sheet has a higher β value than that for NOK. Work is in progress on the texture evaluation and plasticity modeling to predict the mechanical anisotropy and formability of these materials, and progress to-date will be presented. This work has been supported by the National Science Foundation Grant #DMR-9504818.

2:30 PM
The Effect of Scandium on the Microstructure of Al Alloys: *Tadashi Aiura*[1]; Yasuhiro Miura[2]; [1]Kobe Steel Limited, Chofu Plant, Shimonoseki 752-0953 Japan; [2]Kyushu University, Dept. of Matl. Sci. and Eng., Hakozaki 6-10-1, Higashi-ku, Fukuoka 812-8581 Japan

The effect of Scandium(Sc) on the structure and property of the industrial aluminum alloys was investigated. Microstructures, as-solidified and as heat-treated, of the conventional aluminum alloys, the Al-Mg and Al-Mg-Zn alloys, are largely improved by the addition of a small amount of Sc(0.2%). Scandium, added to the Al-Mg alloy, tends to enhance the solidification rate in the casting process, resulting in finer dendrite structure. And consequently, the distribution of the second phase intermetallic compounds, formed between the dendrite arms, also becomes much finer, leading to the good surface quality of the extrusions. The Sc-added Al-Mg alloy has tensile strength 30% higher than that of the conventional alloy after annealing. TEM observation of the Sc-containing alloy revealed a fine dispersion of the coherent, spherical Al3Sc particles, which is thought to be the main cause for the observed improvements in microstructure and property of the alloys.

2:55 PM
Mechanical Properties of Heavily Deformed Copper Alloy: *Koichi Miyake*[1]; Dierk Raabe[2]; Hidefusa Takahara[1]; [1]Mitsui Mining and Smelting Company Limited, Corp. R&D Ctr., Haraichi 1333-2, Ageo, Saitama 362-0021 Japan; [2]Carnegie Mellon University, Dept. of Matls. Sci. and Eng., Pittsburgh, PA 15213 USA

Mechanical properties of a copper wire were examined. The copper wire containing 10 mass % chromium and 3 mass % silver was produced through heavily cold deformation. It was expected that the copper wire was easily broken during cold deformation because of the existence of brittle chromium phase in the alloy. In fact, the copper wire was broken due to large work hardening, when the true strain [η] was 5. However, the copper wire annealed at 1273K for 100 minutes at the middle of deformation process could be smoothly drawn to η=8. After precipitation treatment for silver phase at 473K for 30 minutes, the wire was further drawn to η=8.4. The final deformed wire showed tensile strength higher than 1200MPa.

3:20 PM
A Review of Processing for Metallic Foams and A Basic Study on Bubble Formation in Liquids: Svyatoslav Vitalievich Gnylosukurenko[1]; Takashi Nakamura[1]; *Yoshio Waseda*[1]; [1]Tohoku University, Instit. of Adv. Matls. Process., Rsch. Ctr. for Metall. Process. Eng., 1-1 Katahira 2chome, Aoba-ku, Sendai 980-8577 Japan

Metallic foams, especially light metal foams, have been focused in materials science and engineering due to their characteristics such as lightness and high cushioning ability. Many processes to make metallic foams have been proposed and some of them are commercialized. The processes are classified according to the state of starting materials for the purpose of their use. Making processes of metallic foams were briefly reviewed and some experiments to make small bubbles in water were done in the present study. Bubble size in a liquid depends on the wettability between nozzle materials and a liquid under a certain condition. Bubble foaming experiments were done using model liquids. Smaller

babbles were obtained when a nozzle having a good wettability with the liquids.

3:45 PM Break

4:00 PM

Wettability of Tin-Bismuth-Silver Solders: *Teruo Tanabe*[1]; [1]Kyoto University, Matls. Sci. and Eng., Yoshida Honmachi, Kyoto 606-8501 Japan

The development of lead-free solders is urgent in view of environmental standpoints. In this work the wettability of tin-bismuth-silver solders on copper was studied. Four kinds of fluxes were used. The wettability of conventional eutectic tin-lead solder exhibited an excellent level over the other solders of tin-bismuth systems. Poor wettability of the tin-bismuth solders may be caused by the growth of dendritic copper-tin compounds at copper/solder interface, in contrast to the case of tin-lead solder. The wettability was improved by addition of silver in the solder. In this tin-bismuth-silver system, silver was abnormally concentrated at the copper/solder interface even in the initial stage of contact. The concentration of silver may depress the formation of dendritic compounds.

4:25 PM

Oxidation Behavior of Molten Cu-Be Alloys: *Mitsuru Tanahashi*[1]; Jutaro Miura[1]; Takaharu Iwadachi[2]; Hiroyuki Sano[3]; Toshiharu Fujisawa[3]; Chikabumi Yamauchi[1]; [1]Nagoya University, Dept. of Matls. Sci. and Eng., Furo-cho Chikusa-ku, Nagoya, Aichi 464-8603 Japan; [2]NGK Insulators Limited, New Mets. Div., 1 Maegata-cho, Handa, Aichi 475-0825 Japan; [3]Nagoya University, Rsch. Ctr. for Adv. Waste and Emiss. Mgmt., Furo-cho Chikusa-ku, Nagoya, Aichi 464-8603 Japan

Cu-Be alloys are used for various functional materials, such as electronic devices, machine parts, etc. However, a large amount of beryllium in molten copper is oxidized during the Cu-Be alloy production process, because beryllium is a high-reactive element. The oxidation loss of beryllium deteriorates the production yield of the Cu-Be products, and therefore, should be kept at minimum. In the present study, as a first step for analysis and solution of this serious problem, the oxidation behavior of molten Cu-Be alloys was investigated. By exposing the molten Cu-Be alloys to oxidizing atmospheres, the oxidation rate was measured at 1423 K, and the oxide film formed on the molten alloy was analyzed by EPMA. Based on the results, the effects of the oxygen partial pressure, the composition of alloys, and the small addition of calcium or zirconium (depressant for beryllium oxidation) on the oxidation behavior of the molten alloy were examined.

4:50 PM

Research, Technical and Economical Analysis of the Thermal Methods of Production Alumina (Al_2O_3), Technical Aluminum and Its Alloys at the High Temperature Shaft-Furnace: *Shelkov E.M. Ivtan*[1]; Volovik A.V. Ivtan[1]; [1]1 Samotechny 18, Appt. 61, Moscow 103473 Russia

One of the most power consumption industrial process is production of Al from bocsites–about 45000 KWt. hour electrical energy for production of I ton. of technical Al. This process is based at the low efficiency and productivity electrolysis technology. Last years IVTAN investigations give to us information about possibility to use of high temperature shaft-furnaces for efficiency production of alumina (Al_2O_3, technical aluminum and some alloys on the base of the high temperature carbon-thermal reduction process. At this case power consumption for production of unit of last product will be less at the 3-4 times. The carbon-thermal method is allow to produce alumina (Al2O3), technical Al and its alloys not only from deficient bocsites, but practically inexhaustible reserve of other types of Al-contain raw materials/ cianite, sillimonite and so on. The thermal-chemical and technical-economical analyses of technology is fulfill at the IVTAN, especially for production of alumina. Based on arguments on facts of production at the high temperature shaft-furnace technical Al and its alloys.

Composite Materials - III

Tuesday PM Room: DaVinci 2&3
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Ivar Reimanis, Colorado School of Mines; Keisaku Ogi, Kyusyu University, Dept. of Matls. Proc. Eng.

2:00 PM Keynote

New Processing of Whisker Reinforced Metal Matrix Composites: *Hideharu Fukunaga*[1]; Jin Pan[1]; [1]Hiroshima University, Dept. of Mech. Eng., Kagamiyama, Higashi, Hiroshima 739-8527 Japan

The ceramic whisker reinforced aluminum metal matrix composites (hereafter, referred to MMC) have a large potential for providing high specific strength and stiffness, excellent wear resistance at moderate high temperatures up to 250°C. The silicon carbide, aluminum borate and potassium titanate oxides whisker, titanite dioxides particle and alumina short fiber are excellent and commercial base reinforcements. MMCs are fabricated by squeeze casting process, in which the whisker preform is infiltered with molten metal in the metal die. The main process parameters are molten and preform temperatures, volume fraction of preform, infiltrating rate of the melt and final squeeze pressures. The process conditions to produce the sound MMCs are practiced and predicted, and the typical application of MMCs are also demonstrated. The aluminum matrix composites are nowadays requested to resist at higher temperature service, while the engineering plastic matrix composites are catching up the conventional aluminum matrix composites. The reaction squeeze casting process was newly developed to improve the high-temperature properties of the MMCs by promoting positively the reaction between titanate dioxides and molten aluminum, resulting to the titanate aluminides-distributed aluminum alloy matrix with reinforcement of alumina fibers. This is the in-situ reaction process during squeeze casting, available for strictly narrow process conditions because of high reaction rate and high reaction temperature. Another process with mild reaction was also developed. The MMCs as squeeze cast, consisting of the titanate dioxide and aluminum alloy, are heat-treated at the solid phase temperatures of aluminum alloy. The minute experiments proved that the alkali metal oxide proceeded the solid reaction, which was contained in titanate dioxide as the impurity element. The reaction was found to proceed very slowly, then it was easy to control the hardness of MMCs. Also it is possible to fabricate the hybrid components, partially hardened by selecting the size and shape of preforms.

2:30 PM

Fabrication of Al Matrix Composite by Reactive Infiltration Process: *Makoto Kobashi*[1]; Naoyuki Kanetake[1]; Takao Choh[1]; [1]Nagoya University, Matls. Process. Eng. Dept., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

The spontaneous infiltration of molten aluminum into the SiC based powder compact was investigated. Molten aluminum did not infiltrate spontaneously into the compacted SiC powder even

at 1473K. Titanium and boron carbide (B4C) powders were blended with the SiC powder to promote the spontaneous infiltration. This blended powder shows the exothermic reaction when it is compacted and heated. The spontaneous infiltration of molten aluminum took place at 1073K when sufficient titanium and B4C powders were blended with the SiC powder. The exothermic reaction between titanium and B4C took place at the infiltration front and raised the temperature of the specimen locally, which improved the wettability between molten aluminum and SiC. Titanium boride and titanium carbide, formed by the exothermic reaction, were observed after the infiltration.

2:55 PM

Control of Ag Distribution in YBCO/Ag Composites: *Takashi Kuroki*[1]; Kazuhiro Nishizono[2]; Nobuyuki Mori[3]; Keisaku Ogi[1]; [1]Kyushu University, Dept. of Matls. Process. Eng., 6-10-1Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan; [2]Kyushu University, Dept. of Matls. Process. Eng., 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan; [3]Kyushu University, Depts. of Phys. and Chem., 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581 Japan

The improvement of critical current density and mechanical property is essential to the practical application of the superconductive oxides. A solidification technique is developed in this study for the growth of columnar or single Y123 crystal with finely dispersed Y211 and Ag particles through a monotecto-peritectic reaction of LI Y211=Y123 Ag(LII). Though the large solidification rate and temperature gradient could refine the Ag and Y211 particles, Ag-free region is widely formed along the crystal growth direction in the central part of the Y123 cell. Since this phenomenon occurs because of the cellular growth, it is necessary to narrow Y123 cell width or to grow Y123 crystal with flat freezing interface. By controlling crystal growth direction using a seed crystal, Y123 columnar and single crystal with uniformly dispersed fine Ag particles are obtained. The behavior of Ag and Y211 particles during the solidification of Y123 crystal is simulated by the peritectic growth model of faceted Y123 crystals.

3:20 PM

Pressureless Infiltration into Preformed SiC_P by Al-Si Alloys: *Hideo Nakae*[1]; Hideki Yamaura[2]; Takashi Miyamoto[3]; Takeshi Yanagihara[4]; [1]Waseda University, Dept. of Matls. Sci. and Eng., 3-4-1 Ohkubo, Shinjuku, Tokyo 169-8555 Japan; [2]Hitachi Metals Limited, Cast. Tech. Rsch. Lab., Tochigi, Japan; [3]Waseda University, Dept. of Matls. Sci. and Eng., Student, 3-4-1 Ohkubo, Shinjuku, Tokyo 169-8555 Japan; [4]Waseda University, Dept. of Matls. Sci. and Eng., Grad. Stud., 3-4-1 Ohkubo, Shinjuku, Tokyo 169-8555 Japan

For the production of partially composed MMC's, namely discontinuous MMC's, using a casting process, the interpenetrating phase composites with SiC_P were fabricated by pressureless infiltration using molten Al-Si alloys. The diluted water glass-coated SiC grains passing through a 36 mesh sieve was sintered and putted in a mullite Tammann tube and infiltrated with molten Al-Si alloys in air at 900°C. The aluminum alloy was placed on the sintered SiC_P and heated at 900°C for 90 min. As for the improvement of the infiltration, Fe_3O_4 powder was mixed with the water glass due to the thermit reaction, and pure Ca was alloyed in the melt for the improvement of the wettability. In some experiments, pure aluminum powder was placed on the sintered SiC_P to increase the interfacial area and the chemical reaction between the sintered SiC_P and the melt. By combining these conditions, we succeeded in the complete infiltration without pressure. The mechanism will be discussed based on the wettability of the SiC_P/Al-Si melt.

3:45 PM Break

4:00 PM

Thermal Expansion Behavior of Aluminum-Matrix Composites Processed by Solid- and Liquid-State Routes: *Horacio Elio Nassini*[1]; Mario Moreno[1]; Carlos González Oliver[2]; [1]Comisión Nacional de Energía Atómica, Unidad Tecnológica Pilcaniyeu, Avda. Bustillo 9500, San Carlos de Bariloche, Río Negro 8400 Argentina; [2]Consejo Nacional de Investigaciones Científicas y Técnicas, Unidad Tecnológica Pilcaniyeu, Avda. Bustillo 9500, San Carlos de Bariloche, Río Negro 8400 Argentina

The effect of the processing route on the thermal expansion behavior of an aluminum alloy (Al-4% Cu) reinforced with alumina short fibers has been characterized, both experimentally and theoretically. The metal matrix composites (MMCs) were obtained by solid- and liquid-state processing routes, using powder metallurgy (PM) and squeeze casting (SC) techniques, respectively, and dilatometric testing on heating mode was carried out to investigate the thermal response between 25°C and 500°C. Dilatometric measurements showed a well distinct behavior of the two composites: in the case of the MMCs processed by PM, the thermal strains varied almost linearly with temperature changes in the whole temperature range, while the thermal expansion curves corresponding to the MMCs obtained by SC presented a characteristic knee above a well-defined critical temperature. The different thermal response of the two composites could be explained by analyzing the evolution of internal thermal stresses during heating: when the volume-averaged internal stresses in the matrix were entirely elastic, i.e. for the MMCs processed by PM, the thermal expansion curves were linear, but if the macroscopic yielding of the matrix was expected to occur at a given temperature, the thermal expansion curve deviated from linearity due to the contribution of compressive matrix plastic deformation. Stress analysis was performed with the Eshelby's model coupled with the Mori-Tanaka mean field approach, and a good agreement between experimental results and predictions was obtained.

Thin Films & Coatings - II

Tuesday PM Room: DaVinci 1
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: John J. Moore, Colorado School of Mines; Hiroyuki Sugimura, Nagoya University, Dept. of Matls. Process. Eng.

2:00 PM Keynote

Development of a Novel Ni Coating Technique of Ni(OH)2 Powder for Ni-MH Batteries: *Koji Hoshino*[1]; Takahiro Uno[1]; Kazusuke Sato[1]; [1]Mitsubishi Materials Company, Cntl. Rsch. Instit., 1-297 Kitabukuro-cho, Omiya, Saitama 330-8508 Japan

A chemical reduction technique was developed to reduce the surface of Ni(OH)2 powder as a pretreatment to improve cathodic performance of Ni-MH battery. It was found that Ni(OH)2 powder was reduced by N2H4 without catalyst in an alkaline solution at high temperatures. The reduction rate of the Ni(OH)2 was increased in proportion to the amount of added N2H4. Open cell tests were carried out with the paste type electrodes of the Ni coated Ni(OH)2 powder and non-coat powder, and cell performances were compared. In result, it was found that the Ni coated

Ni(OH)2 powder had superior performance to non-coat powder under the discharging condition of high current densities.

2:30 PM

Surface Reactions in Low-Temperature Plasma Deposition of Silicon-Oxide Films: *Yasushi Inoue*[1]; Jun Iwai[1]; Hirouki Sugiyama[1]; Osamu Takai[1]; [1]Nagoya University, Matls. Process. Eng. Dept., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

Recently low-temperature silicon dioxide (SiO_2) deposition has been in great demand, especially for polymeric materials in order to improve their mechanical and optical properties. Plasma-enhanced chemical vapor deposition (PECVD) using organosilicon reactants is one of the most promising technique for this purpose. In the present, however, the SiO_2 films deposited at low temperatures tend to contain hydrocarbon constituents and a plenty of Si-OH termination of Si-O-Si networks, which degrade the properties of the films. In order to solve the problems, it is necessary to understand the mechanism of SiO_2 deposition at low temperatures. In this work, we report on in-situ diagnostics of the low-temperature PECVD of SiO_2 by means of mass spectroscopy and infrared reflection absorption spectroscopy. The reactions at the film surface are discussed.

2:55 PM

Behavior of Cu-In-Te Electrodeposition from Acid Solution: *Takahiro Ishizaki*[1]; Naghiro Saito[1]; Daisuke Yata[1]; Akio Fuwa[1]; [1]Waseda University, Depts. of Matls. Sci. and Eng., Okubo 3-4-1, Shinjuku-ku, Tokyo 169-0072 Japan

Ternary compound-semiconductor, CuInTe2, film has been deposited cathodically under potentiostatic conditions on titanium substrate from aqueous solution containing CuCl2, InCl3, TeO2 and HCl. The deposition parameters such as electrolytic solution composition, potential and temperature have been optimized for electrodeposition of CuInTe2. Structural characterization of the deposited film has been carried out using XRD and SEM analysis. Electrodeposited films were prepared and analyzed for their chemical composition using ICP studies. The ICP analyses show that the stoichiometry of the films can be controlled by (1) the deposition potential and (2) the electrolytic bath composition, respectively.

3:20 PM

Temperature Programmed Photoelectron Emission Analysis of Copper Surfaces Subjected to Cleaning and Abrasion in Organic Liquids: *Yoshihiro Momose*[1]; Suguru Kohno[1]; Masakazu Honma[1]; Tokirou Kamosawa[1]; [1]Ibaraki University, Dept. of Matls. Sci., 4-12-1 Nakanarusawa, Hitachi, Ibaraki 316-8511 Japan

An extremely sensitive surface analysis technique called temperature programmed photoelectron emission (TPPE) has been developed to easily examine the state of rolled copper surfaces ultrasonically cleaned and subsequently abraded by a screw in various liquids like organics and water. The TPPE apparatus is composed of a Geiger counter with Q gas (He iso-C_4H_{10}). The curve (PE spectrum) of PE intensity vs. wavelength from 300 to 170 nm was repeatedly measured at different temperatures in the temperature-increase process from 25 to 350°C. The plot of the PE total count (the number of emitted electrons in a PE spectrum) vs. temperature exhibited a maximum at 250°C and greatly depended on the liquids. With the surfaces cleaned alone the maximum PE total count decreased in the order n-C_3H_7OH > $(CH_3)_2CHOH$ > C_2H_5OH > CH_3OH > H_2O. With the abraded surfaces the maximum PE total count at 10-min abrasion decreased in the order H_2O > CH_3OH > C_2H_5OH > $(CH_3)_2CHOH$ > n-C_3H_7OH, and tended to appear at higher temperature with increasing abrasion time. The XPS analysis of O1s spectra indicated that the chemical change from the adsorbed oxygen to the oxide oxygen with temperature plays a most important role in the appearance of the maximum PE total count.

3:45 PM Break

4:00 PM

Formation of Al-Cr-N Films by a DC Reactive Sputtering Method and Evaluation of Their Properties: Yukio Ide[2]; *Takashi Nakamura*[1]; Katsuhiko Kishitake[3]; [1]Tohoku University, Instit. of Adv. Matls. Process., Rsch. Ctr. for Metall. Process. Eng., 1-1 Katahira 2chome, Aoba-ku, Sendai 980-8577 Japan; [2]Yamaguchi Prefectural Industrial Technology Institute, Matl. Eng. Dept., 4-1-1 Asutopia, Ube 755-0151 Japan; [3]Kyusyu Institute of Technology, Dept. of Matls. Sci. and Eng., 1-1 Sensui-cho, Tobata-ku, Kitakyusyu 804-8550 Japan

The formation conditions of Al-Cr-N films by a dc reactive sputtering method and their properties were studied in this study. The crystal structure of the Al-Cr-N films changed from hexagonal phase to cubic phase with an increase in Cr content in the films. The resistivity to thermal oxidation of the Al-Cr-N films is superior to that of Ti-Al-N films. A half of the friction coefficient in the Al-Cr-N film was obtained comparing with that in the ordinary Ti-Al-N film at room temperature and the wear rate of Al-Cr-N film was 2 order smaller than that of Ti-Al-N film. The Al-Cr-N film was found to have very high hardness and high performance for the tribological properties.

4:25 PM

Fabrication of Al-pt Coating on Ni-base Superalloys by Sputtering-ion Beam Technique Under Argon Plasma and Their Structure: *Homaira Parchamy Aragy*[1]; M. Goranneviss[1]; A. Sedghi[1]; [1]Islamic Azad University Research Center, Plasma Physics Research Center, I.A.U,Hesarak, Ponak, Tehran, Iran

Thin film coating are formed on the engineering materials to improve their physical and mechanical properties these coating are used mainly in different fields of industrial application and in these respect many research projects conducted to improved their properties. In this paper, therefore, the formation of Al-Pt (Aluminum/Platinum) alloys coatings on nickel based super alloys has been studied. Sputtering-ion beam technique under Argon plasma (which is a PVD technique) and subsequent heat treatment is been used to fabricate these coating. SEM, RBS, GDS and XRD techniques are used to investigate the structure of fabricated coatings. The results show that coatings consist of aluminum-platinum alloys with Pt3Al intermetallic compound. The structure of coatings has a cauliflower feature and trace diffusion of Al in Pt has been performed during sputtering. These results correlates with other investigations and these methods can be presented as a industrial method for fabrication of these coatings.

Solidification Processing-I

Tuesday PM Room: Raphael
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Ben Q. Li, University of Washington, Pullman, WA; Hisao Esaka, National Defense Academy, Dept. of Matls. Sci. and Eng.

2:00 PM Keynote

Modeling Micro Scale Phenomena for Application in Solidification Process Simulations: *Vaughan R. Voller*[1]; [1]University of Minnesota, Civil Eng. Dept., 500 Pillsbury Dr. SE, Minneapolis, MN 55455 USA

Over the last ten years an increasing number of coupled heat and mass balance models of solidification processes have been presented in the literature. A key component in these models is the coupling between thermal and solute fields. This is achieved by developing relationships based on the behavior at the microscopic scale in the casting (typically a secondary dendrite arm space). Early models described the microscopic scale using simple limit assumption for the mass diffusion—Gulliver-Schile or lever—in a fixed arm space. Recently, however, more sophisticated micro scale models that account for finite mass diffusion and coarsening of the arm space have been developed. This paper will explore these models and show how they can be integrated into a large-scale solidification simulation. Three distinct styles of micro models will be introduced (1) those based on a parameterization of the micro phenomena, (2) those based on approximate numerical solutions and, (3) those based on a full numerical treatment.

2:30 PM

Microstructure and High Temperature Phase Studies of Rapidly Solidified Al-Fe-V-Si Alloy: *Suat Kemal Angi*[1]; Ali Kalkanh[1]; [1]Middle East Technical University, Metallu. and Matls. Dept., Inonu Bulvan, Ankara 06531 Turkey

Microstructure and high temperature phase analysis of rapidly solidified Al-Fe-V-Si alloys were investigated. For this purpose, a series of commercial aluminum alloys (FVS 0812) designed for high temperature strength application were prepared and then solidified rapidly in flake and ribbon form by melt spinning technique. The average thickness of the resulting ribbons were in the range of 30 to 250 μm and the corresponding cooling rates were about 10^5-10^7°C/sec. These as-cast products were then heat treated at the temperature range of 150 to 500°C for 1-3 hours. The melt-spun ribbons were examined by X-ray Difractometer, Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) for phase analysis and microstructural morphology at both as-cast and heat treated conditions. Examinations of the melt-spun ribbons revealed that the very fine dispersoids which are uniformly distributed throughout the aluminum matrix in high volume fractions are stable at as-cast and heat treated conditions. In addition, rapidly solidified Al-Fe-V-Si alloys have characteristically cellular type morphology and the nearly spherical second phase particles (silicide dispersoids) decorate the aluminum cell boundaries.

2:55 PM

Influence of Gravity on Monotectic and Eutectic Growth: *Ichiro Aoi*[1]; Makoto Yoshida[2]; Makoto Ishino[1]; Hideo Nakae[1]; [1]Waseda University, Dept. of Matl. Sci. and Eng., 3-4-1 Okubo, Shinjuku-ku, Tokyo 169-8555 Japan; [2]Hiroshima University, Fac. of Eng., 1-4-1 Kagamiyama, Higashihiroshima, Hiroshima 739-8527 Japan

The influence of gravity on the microstructure of unidirectionally solidified Cu-Pb monotectic, Ag-Si and Fe-C eutectic alloys is studied. In order to examine the influence of gravity in a 1g environment, both the upward (opposite to direction of gravity) and downward (direction of gravity) unidirectional solidifications are carried out. The results are as follows: (1) As concerns the upward solidied Cu-Pb monotectic alloy, a banded structure, which consists of Pb-rich layers and Cu-rich layers, is observed. However, such a banded structure is not observed in the downward solidified Cu-Pb specimen. (2) In the case of the upward solidified Ag-Si eutectic alloy, the eutectic Si continuously grows. On the other hand, in the downward solidified Ag-Si alloy, the Si cannot continuously grow and disperses into the α-Ag matrix. (3) No morphological difference in the eutectic growth is observed between the upward and downward solidified Fe-C eutectic alloys.

3:20 PM

Growth Direction of Cellular and Dendritic Interface: *H. Esaka*[1]; H. Daimon[1]; K. Fujita[1]; K. Shinozuka[1]; M. Tamura[1]; [1]National Defense Academy, 1-10-20 Hashirimizu, Yokosuka 239-8686 Japan

It is qualitatively know that growth direction of dendrite almost coincides to the preferred growth direction and is independent on heat flow direction. On the other hand, growth direction of cell is affected by heat flow. However, the quantitative study on growth direction of cell or dendrite has not been carried out. In-situ observation of unidirectional solidification using transparent substance has been performed in this study. Cell or dendrite of which preferred growth direction (θ) is not parallel to the heat flow direction has been observed with various solidification condition and measured their growth direction (δ). Normalized growth angle (δ/θ) is plotted as a function of growth velocity at constant temperature gradient and composition. δ/θ increases with increasing V and is approaching unity. The range where δ/θ sharply increases corresponds to the initiation of side branches.

3:45 PM Break

4:00 PM

Material Properties Improvement of Cast A206 Using Vacuum Assisted Counter Gravity Casting Process: *Scott R. Giese*[1]; Lucas Dix[2]; [1]University of Northern Iowa, Met. Cast. Ctr., 4318 Cherrywood Dr., Cedar Falls, IA 50613 USA; [2]University of Northern Iowa, Indus. Tech. Dept., Cedar Falls, IA 50614-0178 USA

Recently, there is a growing trend in the aluminum foundry industry to develop improved melting and casting processes to produce lightweight, high-strength, and thin-wall castings. One casting method that has demonstrated industrial potential for producing clean, thin wall casting is vacuum-assisted counter gravity casting. This process has the benefit of expeditiously and quiescently filling the casting cavity with a dramatic reduction of furnace related and reoxidation inclusions. In addition, lower casting temperatures can be utilized because the applied pressure overcomes the resistive internal cavity pressure resulting in a finer, as-cast microstructure. By using a lower casting temperature and reducing the number of inclusions, A206 alloy responds more favorable to heat treatment processes than traditional, gravity pouring methods. The paper explores and discusses the processing benefits in improving the as-cast and heat treated mechanical properties of A206 casting alloy.

4:25 PM

Semi-Solid Processing of Hyper-Eutectic Cast Iron: *Masaru Imaizumi*[1]; Hiroyuki Nomura[2]; Mitsuharu Takita[2]; [1]Nagoya University, Dept. of Matls. Process. Eng., Grad. Student, Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Dept. Matls. Process. Eng., Schl. of Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

Cast iron possesses many superior characteristics due to graphite structure, such as castability, wear resistance, solid lubricant, damping capacity. But the more graphite hyper-eutectic cast iron contains, the lower its mechanical strength becomes, because graphite gives a negative effect to strength of cast iron. In addition, primary graphite tends to segregate during solidification, so uniform materials are difficult to be produced. In this study, authors introduced semi-solid processing in order to develop new and high quality cast iron. A series of semi-solid processing experiments were carried out to clarify the effect of stirring condition, cooling rate, solidification temperature and etc. on micro-

structures and property characteristic of cast iron. In particular much attention is paid to the appearance of ternary eutectic zone at the final stage of solidification. As the results advanced hypereutectic cast iron was obtained which is characterized uniquely by mechanical strength and wear resistance.

4:50 PM

The Influence of Thermophysical Properties on the Calculation Accuracy of Solidification Thermokinetics: *Frantisek Kavicka*[1]; Josef Stetina[1]; Jay M. Khodadadi[2]; [1]Technical University of Brno, Mech. Eng. Dept., Brno, Technicka 2 616 69 Czech Republic; [2]Auburn University, Dept. of Mech. Eng., 201 Ross Hall, Auburn, AL 36849-5341 USA

On a steel sample casting of cylindrical shape that was cast in a fireclay or cast iron cylindrical mold, the analysis of influence of the main thermophysical properties of casting and molding materials on calculation accuracy of temperature field in "the casting-mold-environment system" has been performed. As a comparing datum of accuracy, the total time of solidification (crystallization) and mechanism of forming of temperature field of a solidifying casting, represented by isotherms in longitudinal axial section through the casting and the mold, have been selected. For the cast material, the influence of heat conductivity, specific heat capacity, density, and namely in the liquid or in the solid phase, in addition of latent heat of phase transformation also has been analyzed. For metallic or non-metallic molding material, the influence of the same main parameters has been analyzed and, furthermore, their combination, temperature conductivity and heat storage. The last parameter was introduced for the characterization of a mold by foundry technologists. The influence of the individual parameters has been analyzed separately and shown graphically, the other parameters have been taken for that once in their real value. The influence of each parameter has been studied within the range from 50 to 150% of its real value. The order of influence of parameters on calculation accuracy of temperature field of a casting or mold was determined. If a requirement of an accuracy of calculation of solidification thermokinetics, controlled by the total solidification time, will be given in advance, then it is possible to estimate from the obtained graphs what error can affect the used thermophysical parameter.

Waste Management-II

Tuesday PM Room: Michaelangelo
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Robert Stephens, Cominco; Itaru Jimbo, Tokai University, Dept. of Metall. Eng.

2:00 PM Keynote

Application of Thermal Plasma Technology in the Treatment of Waste: *Patrick R. Taylor*[1]; [1]University of Idaho, Plasma Process. Lab., P.O. Box 443024, Moscow, ID 83844-3024 USA

A review of the application of thermal plasma technology to waste is given. Advantages and disadvantages are discussed. Examples are presented for vitrification and in flight calcination and organic destruction. The characteristics of transferred arcs, non-transferred arcs and inductively coupled plasma systems are presented and compared. The potential for this technology is identified and engineering, economic and environmental considerations discussed.

2:30 PM

The Use of Washability Curves to Predict Critical Glass Fragments (Cullets) Size Classes Recovery According to Their Detected Optical-Digital Properties: *Giuseppe Bonifazi*[1]; Paolo Massacci[1]; [1]Universita Di Roma, Dept. di Ing. Chimi., Matl., delle Mat. Prime e Metall., Via Eudossiana 18, Roma 00184 Italy

Glass fragments (cullets) to be recycled present different market values according to their color. Glass recycling plants perform cullets sorting mainly discriminating colored glasses from white and half white glasses. Cullets which are collected without distinctions of color, can be used primarily for the production of green glass and only in part for the production of yellow glass. The production of white glass requires that only cullets of that color be employed. At present, machines for the separation of cullets according to color are not capable of producing an efficient classification of all the different types. In this paper are analyzed the possibility that could be offered by the adoption of a full color imaging based approach to investigate cullets sorting characteristics through the definition of suitable "washability function" (WF), defined on the bases of cullets color and textural attributes. In this study the adoption of WF curves to preliminary characterize the products to be sorted and for the further selection of the "sorting threshold" to realize the separation can represent a powerful tool to increase separation efficiency and a better understanding of the logical path to follow for the separation, on the bases of a selected set of properties. A WF, in fact, is a cumulative plot: on the x-axis are reported the values representative of the physical property investigated; in the present study the value of the cullets color components or textural parameters as they results from the image analysis procedures applied. On the y-axis are reported the number (normalized frequency) of the physical elements (cullets) presenting a value of the color feature equal or less to that of the corresponding abscissa. Each set of cullets, considered as a whole, can be thus evaluated in terms of high or low possibility to be grouped together (sorted) on the base of corresponding color feature.

2:55 PM

Sulfur Recovery from the Leached Residue Produced in the Direct Leaching of Zinc Concentrate: *Toyohisa Fujita*[1]; Atsushi Shibata[1]; Toshio Miyazaki[1]; Eiichi Ruzuno[1]; Kazuo Koike[1]; Hitoshi Matsuda[2]; [1]Akita University, Fac. of Eng. and Resource Sci., 1-1 Tegata Gakuencho, Akita 010-8502 Japan; [2]Akita Zinc Company Limited, Iijima Refinery, Akita 011-0911 Japan

A solid sulfur is more useful to recover and store than a sulfuric acid as a by-product in the zinc hydrometallurgy. In this experiment a zinc concentrate (sphalerite) was leached with ferric sulfate at about 373K for 3 to 5 hours. Zinc was leached more than 95%. After a solid-liquid separation, the leached residue containing solid sulfur was obtained. The particles in the residue were dispersed by lignin and the solid sulfur of approximately 85% was recovered as float product by a flotation with frother in the pH 4. On the other hand in the leached liquid a zinc oxide was added and ferrous iron was oxidized to ferric iron by blowing oxygen. This ferric iron was returned to the zinc leached process after solid-liquid separation. The sulfur recovery from the repeated leached residue was also investigated.

3:20 PM

The Separating of Perovskite from Treated BF-Slag: Junwei Ma[1]; *Zhitong Sui*[1]; Bingchen Chen[2]; [1]Northeastern University, Sch. of Matls. and Metall., P.O. Box 119, Shenyang, Liaoning 110006 PRC; [2]Northeastern University, Dept. of Min. Dress., Shenyang, Liaoning 110006 PRC

In the BF-slag, titanium disseminates in various mineral phases, mainly provskite. Titanium could be concentrated mainly in perovskite, which could be selectively precipitated and grown by optimizing the operation factors such as slag composition, temperature of heat treatment and additive agents. This study aims at establishing the titanium mineralogy and its distribution in the as-received slag and the treated slag, and the methods for the separation of perovskite. After treatment, the grain size grew from 5¡«20¦Ìm to 80¡«200¦Ìm, the distribution of TiO2 in perovskite increased from 48% to 81%, the morphology changed from snow-shaped grain to coarsening dendrites or equiaxed crystals. Gravity separation produced a concentrate analyzing 35.25% TiO2 with a recovery of 68.28% and a tailing analyzing 9.53% TiO2. A concentrate analyzing 40.08% TiO2 and a tailing analyzing 9.28% TiO2 could be obtained by flotation.

3:45 PM Break

4:00 PM

Soil Washing Using Mineral Processing Plant in Hanaoka Mine, Dowa Mining Co. Ltd.: *Toru Nishiyama*[1]; Masaru Tomoguchi[1]; Naoto Sasamoto[1]; [1]Dowa Mining Co. Ltd., Soil Remediation Business Unit, 1-8-2, Marunouchi, Chiyoda-ku, Tokyo 100-8282 Japan

In Hanaoka Mine, which was one of the major "black ore" (Kuroko) mines in northern Japan, mining has finished in 1994, and only mineral processing plant is available now. Recently the mineral processing plant is applied to soil washing. Contaminated soil containing heavy metals is excavated and transported to the plant. The soil is separated to cleaned soil and metal concentration through the process, which consists of scrubbing, classification, extraction, insolubilization, flotation, and so on. The process is flexibly designed for each contaminated soil, in order to the products can be recycled as soil or materials. Arsenic and lead contaminated soil was treated in 1999, which was entrusted from Environmental Agency of Japan.

4:25 PM

Chemical Improvement of Coal Burnt Ash and Slag for Reutilizing as Acid Gas Absorbents: Yuuji Saito[1]; Hiroki Awata[1]; *Shoji Ozawa*[1]; Daisuke Hirabayashi[1]; H. Liwe[1]; Hitoki Matsuda[1]; Yoshimi Goto[2]; Tetushi Iwashita[2]; [1]Nagoya University, Rsch. Ctr. for Adv. Waste and Emission Mgmt., Furo-cho, Chikusa-ku, Nagoya, Pref. Aichi 464-8603 Japan; [2]Yabashi Industry Company Limited, Dev. Div., Ohkubo 4278-1, Akasaka-cho, Ohgaki, Pref. Gifu 503-2213 Japan

We proposed the reutilization of coal burnt ash and slag as absorbents of acid gases such as HCl, SO2 released from thermal treatment of municipal and industrial wastes. The reutilization of these solid wastes contributes to not only saving natural resources but also leading to reducing industrial wastes. The present study is concerned with investigating the effect of chemical treatment on enhancing the absorption capacity of Ca-derived solid wastes for acid gases. Improvement of the reactivity of coal burnt ash and slag toward acid gas absorption were made by slaking under various conditions, using alkaline solution, polyethylene glycol ethanol etc. The reactivities of treated coal burnt ash and slag were evaluated by lab-scale acid gas absorption experiments. Then, the mechanism of the enhancement of the reactivity toward acid gas absorption was made clear by comparing physical structure and chemical composition change before and after chemical treatment.

4:50 PM

A Novel Technique to Recovery Value-Nonferrous Metal Compounds from Metallurgical Slags: *Zhitong T. Sui*[1]; Taiping Lou[1]; Yuhai Li[1]; Nianxin Fu[1]; Guangqiang Li[1]; Zhida Sui[1]; [1]Northeastern University, Sch. of Matls. and Metall., P.O. Box 119, Shenyang, Liaoning 110006 PRC

Based on the case studies in selective precipitating behavior of value-nonferrous metal compounds (VNMC) in molten slags, a novel technique to recovery VNMC is proposed in which three continuous processes are involved. The selective concentrating of dispersed VNMC into designed mineral phase; the selective coarsening of the mineral phase to critical grain size; and the selective separating of the growth mineral phase from tailing by dressing or hydrometallurgy. The features of the technique are efficient, economic, and intensive. The precipitated compounds of Titanium and Boron were summarized respectively as examples of the Technique application. It was confirmed by experiments that the precipitating behavior of the designed phases like perovskite ($CaTiO_3$), Suanite ($2MgO.\ B_2O_3$), ($TiTi_2O_5$) in slags are obviously affected by operation factors such as smelting, temperature, chemical composition, temperature of heat-treatment, cooling rate and additives of the slags. The precipitating kinetics and mechanism of VNMC from slags during solidification processes were also investigated.

Iron & Steelmaking - II

Tuesday PM Room: Parc Ballroom III
November 7, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Gordon Irons, McMaster University Canada; Masahiro Kawakami, Toyohashi University of Technology, Dept. of Prod. Sys. Eng.

2:00 PM Keynote

Prospect for Innovative Refining Process in the 21st Century: *Katsukiyo Marukawa*[1]; Shigeta Hara[2]; Masamichi Sano[3]; [1]Osaka University, Sumitomo Met. Ind. Ltd. Japan; [2]Osaka University; [3]Nagoya University

As for recent research and development in the field of steelmaking, it seems that the emphasis shifted to solidification ingotmaking centering on continuous casting. From a long range point of view, however, needs of unlimitedly developing a refining process have increased but never decreased. For innovative development of a refining process, the time is ripe for its new development, since global environment and depletion. Now that it is necessary not only to introduce electromagnetic application technology and ultrasonic application technology but also to apply new mathematical simulation method for creation of innovative high-efficiency mixture separation reactor, such technological development is required that flexibly includes engineering of other industries and different fields and process elements which had been devised but kept from the public eye without use and resolves issues which had been previously given up.

2:30 PM

Analysis of Mixed Grade Transition in Continuous Thin Slab Casting with EMBR: *Jae-Hwan Ahn*[1]; Jong-Kyu Yoon[1]; Jung-Eui Lee[2]; [1]Seoul National University, Sch. of Matls. Sci. and Eng., San 56-1Shinrim-2 Dong, Kwanak-ku, Seoul 151-742 Korea; [2]POSCO, Kwangyang Iron and Steel. Rsch. Grp., Tech. Rsch. Labs., Kwanyang 545-711 Korea

A concentration change during grade transition operation in steel thin slab casting is predicted with computer simulation, water model and plant trial. Fluid flow and mixing patterns in various tundish levels and flow rates were analyzed through three-di-

mensional mathematical modeling. Comparing with water model experiments, a simple, efficient and accurate computational model has been suggested to predict the concentration profile at outlet of tundish. Based on the model, mixing in/below the mold was analyzed considering EMBR. The predicted concentration profiles show good agreement with the measured values. It is found that the lower vortices in the mold are suppressed by the electromagnetic field and the plug-like flow region develops which decreases the intermixing of two different grades of steel and shortens the length of transition region.

2:55 PM

Hot-Dip Galvanizing Features of Various Grades of Spheroidal Graphite Cast Iron: *Annelize Els-Botes*[1]; Danie Hattingh[1]; [1]Port Elizabeth Technikon, Fac. of Mech. Eng., Private Bag X6011, Port Elizabeth 6000 South Africa

Considerable information is available on the factors influencing the iron-zinc reaction rate at 450°C of low carbon constructional type steels, in comparison there is a scarcity of fundamental information on spheroidal graphite irons. A reason for this is the more complex microstructure and chemical analysis of spheroidal graphite irons. When considering the effects of silicon and phosphorus on the galvanizing characteristics of spheroidal graphite irons, it has to be appreciated that the silicon levels do exceed 2% and the phosphorus content is usually similar to that of steel. Further important differences (compared to low carbon steels) are the presence of graphite nodules and higher percentages of pearlite. Furthermore, the increased percentage of elements present can dissolve into the various phases. Despite the apparent complexity of the iron-zinc reaction on spheroidal graphite irons, the aim of this paper is to contribute towards a better understanding of the mechanism of this reaction.

3:20 PM

Control of Mixing Phenomena between Two Liquids using Lorentz Force: *Toshiyuki Kozuka*[1]; Masayasu Kawahara[1]; [1]Kumamoto University, Matls. Sys. Eng., 2-39-1, Kurokami, Kumamoto 862-8555 Japan

In many metallurgical processes, mixing phenomena between two liquid phases are considered to be important in the mass transport. Lorentz force can suppress the wave motion of metal surface and can also promote the mixing of two liquids. In this paper, new methods of mixing control including wave suppression and separation are proposed, where a direct electric current and a stationary magnetic field are utilized. The model experiments can clarify that upward Lorentz force can suppress the mave motion caused by gravity and intense upward force can scatter metal drop in upper layer, and that downward force can promote the recovery of dispersed drops in another liquid.

3:45 PM Break

4:00 PM

Mathematical Modeling and Cold Model Experiment on Ultrasonic Separation of Inclusions from Molten Metal: *Mamoru Kuwabara*[1]; Tomohito Taki[1]; Masamichi Sano[1]; [1]Nagoya University, Dept. of Matls. Process. Eng., Furo-cho Chikusa-ku, Nagoya, Aichi Prefecture 464-8603 Japan

Theoretical basis and experimental results on ultrasonic separation of dispersed inclusions from molten metal using a standing-plane-wave field are outlined. Numerical solution of the equation of motion of a fine particle in a standing-wave field indicates that the inertia term can be neglected during conventional ultrasonic separation of fine particles. Analytical solutions for the particle speed, the position at which particles are coagulated, and the minimum power for separation, are derived to incorporate such key process parameters as the density difference and the acoustic energy density exerted. Cold model experiments have been carried out to observe transitional coagulation of polystyrene particles in an aqueous sugar solution with the incidence of standing ultrasonic plane wave. Experimental results agree well with the theoretical predictions.

4:25 PM

Deoxidation and Desulfurization of Molten Iron with Magnesium Vapour In-Situ Produced by Carbothermic Reduction of Magnesium Oxide: *Keiji Okumura*[1]; Jiayi Shan[1]; Jian Yang[1]; Masamichi Sano[1]; [1]Nagoya University, Matls. Process. Eng. Dept., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

A new method of deoxidation and desulfurization of molten iron with magnesium vapor has been developed. The immersion tube, which was filled with pellets of the mixture of magnesia and carbon powders, was put into the molten iron. Magnesium vapor produced by carbothermic reduction of magnesia inside the tube was injected directly into the molten iron with argon carrier gas. The effects of temperature and carrier gas flow rate on deoxidation and desulfurization were investigated. A mathematical model of the deoxidation and desulfurization reactions was developed. The rates of deoxidation and desulfurization were controlled by mass transfer of magnesium vapor in the gas-phase and mass transfer of oxygen and sulfur in the liquid-phase. This method may offer high efficiency for magnesium deoxidation and desulfurization of molten iron.

4:50 PM

A New Type of Bell-Less Top for Blast Furnaces: *Yunjin Yan*[1]; Mingchun Lu[2]; [1]University of Science and Technology of Beijing, #912 Bldg. 37, Beijing 100083 PRC; [2]Tianjin Ironmaking Group, Eng. Dept., China

This paper describes a new type of bell-less top installed on two relined blast furnaces at Tianjin Iron Works and another new-built at Echeng Steel Works in China. A compact installation was presented for existing furnace top innovation with the least reformation of existing top structure. A water cooling and sealing gearbox used for driving the chute discards completely the traditional idea about necessity of continuous supply of cooling gas for the gearbox. A multi-ring spreader and its dozens of selectable charging patterns enables an operator to achieve optimal burden distribution easily under the condition of a minimum investment of control equipment. Some useful conclusions have been made through a small-size model and a full-scale model tests. The excellent characteristics and the superiority of the new top have been fully proved at both works.

Solidification Processing - II

Wednesday AM Room: Raphael
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Vaughn Voller, University of Minnesota, MN USA; Hiroyuki Nomura, Nagoya University, Dept. of Matls. Process. Eng.

8:30 AM Keynote

Computer Simulation of Casting-Recent Development: *I. Ohnaka*[1]; J. D. Zhu[1]; T. Ohmichi[1]; S. Kashiwai[1]; H. Yasuda[1]; [1]Osaka University, Dept. of Adapt. Mach. Sys., Grad. Sch. of Eng., Yamadaoka 2-1, Suita-shi, Osaka 565-0871 Japan

Although computer simulation of casting has been greatly developed and now practically used in the foundry industry, still we don't know exactly the casting phenomena especially in the mold. This paper presents and discusses the recent development in the simulation as well as comparison with observation of the real phenomena. The first topic is the comparison of simulation of filling behavior of Al melt in a metallic mold with direct observation through a transparent mold, demonstrating the importance of consideration of surface tension and wetting of the melt with the mold. The second one is the comparison of the simulation of mold filling in the lost foam process with measured pressure change and direct observation using X-ray, showing the possibility of the simulation of the vibration of the gas layer between the melt and polystyrene core and the melt filling behavior. The third one is the comparison of simulation of Al melt flow in a channel and direct observation through a transparent mold, showing that the entrapped gas is expelled ahead through the melt and the simulation needs the consideration of viscosity change with temperature and very fine meshes. These phenomena affect the position and degree of porosity or inclusion defects. Finding new phenomena requires the comparison of the simulation and observation.

9:00 AM

New Composite Cast Alloys Based on Copper and Technological Peculiarity of Processing: *B. A. Kyryyevsky*[1]; V. I. Dubodelov[1]; V. A. Seredenko[1]; H. V. Seredenko[1]; V. V. Khristenko[1]; [1]Physico, Tech. Instit. of Mets. and Alloys of the NAS, Kyiv, Ukraine

During the last decades an investigators of many countries show increased interest to alloys which are characterized by miscibility gap in a liquid state. It is connected with essential possibilities of control of structurization in such alloys because of the melt for at certain temperature parameters is emulsion with disperse phase inclusions. These ensure essential influence on structurization. The multicomponent alloys of system of copper -(Fe-Cr-C)-alloy concern to number of advansed compositions. The copper alloys with a structure of a type "frozen emulsion" in which strengthening a phase is a chromium pig-iron are characterized by high thermal stability in the extreme operating conditions. The mechanism of inclusions forming is emulsifying of a melt in miscibility gap at temperatures close to temperature of binodal under superimposition of external effects. Thanks to a selective action of electromagnetic fields on liquid phases with various electroconductivity relative velocity of phases is regulated the ratio of their "apparent" specific gravity is changed. Excitation of pulsing force effects with the certain frequency of interphase surface allow actively to influence on process of a dispersion of a liquid metal medium.

9:25 AM

Reactive Casting of NiAl and Simultaneous Joining to Steel: *Kiyotaka Matsuura*[1]; Masayuki Kudoh[1]; [1]Hokkaido University, Div. of Matls. Sci. and Eng., Kita 13 Nishi 8, Kita-ku, Sapporo, Hokkaido 060-8628 Japan

Molten aluminum and nickel cast onto a steel block exothermically react and produce molten nickel monoaluminide, NiAl, which is simultaneously joined to the steel. The heat of the exothermic reaction is transferred to the steel block, and steel near the surface of the block is melted. The depth of the melted steel increases with both the initial temperature of the steel block and the thickness of the produced NiAl. Iron from the melted steel dissolves in the molten NiAl, and an intermetallic compound of NiAl-Fe pseudo-binary system is produced. During solidification of the intermetallic compound, it is strongly joined to the steel. The NiAl-Fe intermetallic compound has an excellent resistance to ware, oxidation and corrosion.

9:50 AM

Plasma-Arc Zone Refining of Silicon: *Kouji Mimura*[1]; Manabu Kishida[1]; Minoru Isshiki[1]; [1]Tohoku University, Instit. for Adv. Matls. Process., 2-1-1 Katahira Aoba-ku, Sendai, Miyagi 980-8577 Japan

Purification of metallurgical grade silicon through the plasma-arc zone melting has been examined. The process consisted in the horizontal displacement of a molten zone heated by Ar- and Ar H_2- plasma arc under atmospheric pressure. This technique, especially using hydrogen plasma, promoted the segregation of impurities by zoning from the head to the tail of the bar and the vaporization of impurities of higher vapor pressures. Metallic impurities such as Fe, Ti, Al, Cr and Mn were reduced to less than 1 mass ppm at the head side of the Si bar. Non-metallic impurities such as oxygen, carbon, boron and phosphorous were also reduced by the zoning effect and, furthermore, by activated hydrogen atoms dissociated in the high temperature plasma-arc, according to the following reaction: O (in liq.Si) $2H=H_2O$.

10:15 AM Break

10:30 AM

Numerical Simulation of Ostwald Ripening using the Phase-Field Model: *Machiko Ode*[1]; Seong Gyoon Kim[2]; Won Tae Kim[3]; Toshio Suzuki[4]; [1]University of Tokyo, Grad. Sch., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan; [2]Kunsan National University, Dept. of Matls. Sci. and Eng., Kunsan 573-701 Korea; [3]Chongju University, CNM and Dept. of Phys., Chongju 360-764 Korea; [4]University of Tokyo, Dept. of Metallu., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan

Numerical simulation of Ostwald ripening using a phase-field model is performed. The model proposed by Kim et al is adapted and the phase-field parameters are determined at the thin interface limit condition. The model reproduces the local equilibrium condition at interface and enables us to calculate with large particle size comparable to the experimental data. The time history of the change in the solute distribution during the ripening process is shown. The shape deviation of solid particles and the particle agglomerating are qualitatively analyzed The average particle radius with time and the change of the particle size distribution with the solid fraction are compared with LSW law and experimental observation under wide range of the solid fraction.

10:55 AM

New Process and Technology-VEAT-In Directional Solidification of Eutectics: *Paul Christian Olaru*[1]; [1]FAUR SA, Sr. Metall. Air. Eng., B-dul Basarabia nr. 256, Sector 3, Bucuresti 73429 Romania

VEAT process consists of the simultaneous application of a stationary magnetic field B_0 and of a variable magnetic field b(t) in the vicinity of the molten metal during the course of solidification. It shows that the grain refinement can be effective when an alloy is electromagnetically vibrated under favorable conditions concerning primarily the amplitude of the oscillating pressure. Results from experiments with eutectic Al-Al_3Ni and Al-Al_3Fe alloys are shown, demonstrate the facility is working and the accuracy of the optical temperature measurements can be evaluated. Also, results revealed that the size and shape of the crystals can be controlled both by the application either of various magnetic fields or of a combination of magnetic fields of sufficient strength and by an adequate heat extraction rate.

11:20 AM

Metal/Mould Interfacial Heat Transfer during Solidification of Cast Iron against Cast Iron Chills: *Narayan K. Prabhu*[3]; William

D. Griffiths[2]; [1]University of Manchester and UMIST, Manchester Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK; [2]University of Birmingham, IRC in Matls., Elms Rd., North Campus, Edgbaston, Birmingham B15 2TT UK; [3]Karnataka Regional Engineering College, Surathkal, India

The casting/chill interfacial heat flux and heat transfer coefficients were measured using an inverse modelling approach. Chills of thickness 100 mm and 10 mm were used, to simulate both gravity die casting conditions, and the use of chills in sand moulds. In both cases the transient heat transfer, measured by the interfacial heat flux and heat transfer coefficient, declined from initially high values in the first few seconds of solidification, to values about an order of magnitude lower, which then persisted for the remainder of the experiments. These results were then interpreted by studying the resistance to heat transfer from the casting offered by the casting/chill interface and the chill itself. This is influenced by the surface roughness of the casting and the chill surfaces, their deformation and the subsequent relationships between the two surfaces. Finite element modelling of the chill surface demonstrated that it probably underwent a small convex deformation, which must also be considered in conjunction with the casting surface deformation.

11:45 AM

No-Inner Core Continuous Casting of Metal Pipe: *Zhongming Ren*[1]; [1]Shanghai University, Matls. Dept., 149 Yan Chang Rd., Shanghai 200072 China

A new process of near net shape continuous casting of metal pipe has been put out. The specialty of the process is that no inner core is used. The principle of the process has been presented, and the effect of several factors on the thickness of the pipe has been investigated experimentally and numerically. It is shown that the temperature and the level of the molten metal, cooling intensity of the mold, and the velocity of casting influenced the thickness of the pipe apparently. In order to improve evenness of the thickness of the pipe, the mold was dedicated designed, and special methods were taken. By this process, a metallic material like "banboo" was fabricated.

Cu, Ni, Zn, Pb and Sn - II

Wednesday AM Room: Rubens
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Hong Y. Sohn, University of Utah, Metall. Eng. Dept., 135 S 1460 E, Rm. 412, Salt Lake City, UT 84112-0114 USA; Takahiko Okura, Nikko Techno Service Company

8:30 AM Keynote

Technologicial Advances for Reducing Production Costs of Commodity Metals: *Robert L. Stephens*[1]; Douglas J. McKay[1]; [1]Cominco Research, P.O. Box 2000, Trail, British Columbia V1R 4S4 Canada

It can be argued that the most important property of any material is cost, since this property determines the applications for which a material can be considered. Despite growing demand and production for commodity metals such as Cu, Ni, Zn, Pb, and Sn, the cost of these metals has been steadily decreasing in real terms. The only way that metal producers can remain financially viable in the long term is by continuing to lower their cost curve through the implementation of new metals production technologies. This paper reviews technological developments in the production of Cu, Ni, Zn, Pb, and Sn that have or threaten to redefine the cost curves for these metals.

9:00 AM

Phase Equilibrium between Nickel Alloy Containing Minor Elements and FeO_X-MgO-SiO_2 Slag: *Hector Mario Henao*[1]; Mitsuhisa Hino[1]; Kimio Itagaki[1]; [1]Tohoku University, Instit. for Adv. Matls. Process., Katahira 2-1-1, Aoba-ku, Senda 980-8577 Japan

The experimental study on the phase equilibrium between the Ni-Fe alloy and MgO saturated FeO_X-MgO-SiO_2 slag was conducted under the p_{O2} range from $10^{-6.5}$ to $10^{-9.5}$ atm by using CO-CO_2 mixed gas at 1773 and 1873K, in which each minor element of Cu, Co or Cr was dissolved. The distribution ratios of major and minor elements between the nickel alloy and slag phases were calculated and dissolution of the elements into the slag was analyzed based on the relation with the oxygen potential in a ferro-nickel smelting process. Effect of CaO or Al_2O_3 on the distribution ratios was also experimentally investigated. The activity coefficients of nickel, copper, cobalt and chromium oxides in the FeO_X-MgO-SiO_2 slag were estimated from the distribution ratios combined with the activity coefficients in the alloy phase. Then, the behavior of the minor elements in the slag is thermodynamically analyzed.

9:25 AM

Activity Measurements of Several Molten Sulfide Systems: Kazuo Koike[1]; *Mitsuhisa Hino*[2]; Akira Yazawa[2]; [1]Akita University, Faculty of Eng. and Res. Sci., Tegata-Gakuen-choh 1-1, Akita 010-8502 Japan; [2]Tohoku University, Instit. for Adv. Matls. Process., Katahira 2-1-1, Sendai 980-8577 Japan

The vapor pressures of MS (PbS,SnS and Sb_2S_3) in the molten Cu_2S-MS, FeS-MS pseudo-binary and Cu_2S-FeS-MS ternary systems were measured with a transportation method in the temperature range of 1323 and 1373K to calculate MS activities in the sulfide melts. The activities of MS in these systems show slightly negative deviation from ideal behavior. The activities of the Cu_2S-FeS pseudo-binary system which is the fundamental system of copper matte were estimated from the activity values in the ternary systems and exhibit negative deviation from ideality. The distribution behavior of tin between the matte and silica saturated slag was investigated under the controlled partial pressures of SO_2 at 1523K. The activity coefficient of SnO in the slag was estimated based on the distribution ratio of tin combined with the activity coefficient of tin in the Cu_2S-FeS-SnS matte which was measured in the present study.

9:50 AM

Recent Operation in the Gresik Smelter and Refinery: *Teruyuki Matsutani*[1]; Toshihiko Igarashi[1]; Moto Goto[1]; [1]PT Smelting, Gresik Smelt. and Refine., Desa Roomo, Kecamatan Manyar, P.O. Box 555, Gresik, East Java 61151 Indonesia

The Gresik Smelter and Refinery, PT Smelting in Indonesia owned by Mitsubishi Materials Corporation (60.5%), Freeport Indonesia (25.0%) and some other Japanese companies (14.5%), has commissioned in middle of December, 1998. It employs Mitsubishi Continuous Process as smelting unit and ISA permanent stainless steel type as copper refinery designed to produce 200,000 tpy of copper cathode from concentrate supplied exclusively from Grasberg Mine of PT Freeport Indonesia in Irian Jaya. The paper will review the design concept and show the actual operational performance.

10:15 AM Break

10:30 AM
Recent Improvements in Waelz Operation for Zinc Leach Residue: R. Yoneoka[1]; Y. Ohmizo[1]; *S. Sakamoto*[1]; S. Karasawa[1]; [1]Toho Zinc Company Limited, 1443 Nakajuku, Annaka, Gunma 379-0197 Japan

The Annaka Refinery has been processing the leach residue by the waelz process for 17 years. Recently, the quantity of the leach residue has increased due to a decline in the quality of zinc concentrate. Meanwhile, negative factors (e.g. deterioration of water balance due to improved electrolytic power consumption rate) have increased in the processing of the leach residue. We have implemented various measures in response to this situation while striving to increase processing capacity and achieve stable operation. As we have largely completed assessing these measures' anticipated effectiveness, we report on our recent process improvements.

10:55 AM
High-Performing Flash Smelting Furnace at Saganoseki Smelter & Refinery: *Chikashi Suenaga*[1]; Takayoshi Fujii[1]; Yoshiaki Suzuki[1]; Mitsumasa Hoshi[1]; [1]Nippon Mining & Metals Company Limited, Saganoseki Smelt. and Refine., Saganoseki, Oita-pre. 879-2201 Japan

In a bid to renovate its Saganoseki Smelter & Refinery and to establish a leading production unit in terms of global competitiveness, Nippon Mining & Metals undertook major restructuring efforts since 1994. One of its themes was the shift from two-flash furnaces operation to a single-flash furnace operation. The shift was successfully completed in 1996 while maintaining the production at the same level (330,000mtpy) by adopting such measures as improvements in pneumatic concentrate dryers, the concentrate feeding system, concentrate burners, and the furnace cooling system. With a view to realizing high matte grade operation (at 65%Cu) and increasing concentrate feeding rate, further steps were taken : a new cryogenic oxygen plant was constructed and technical improvements were added, including optimization of the smelting reaction in the furnace shaft. As a result, the smelting capacity, as of the year 2000, has increased to 450,000 tons per year of copper.

11:20 AM
New Operational Calculation Process for the T-FSF: *Soichirou Tanaka*[1]; [1]Hibi Kyodo Smelting Company Limited, Tamano Smelter and Refinery, 6-1-1, Hibi, Tamano City, Okayama Pref 706-8511 Japan

T-FSF (Tamano Type Flash Smelting Furnace) has been operated as compact facility without slag cleaning furnace, with low dust trouble and low copper loss in slag. After start up, with proceeding operation technology, finally Coke Combustion Technology had developed for T-FSF. Water Cooled Uptake had been also developed, it contributes to decrease dust trouble at boiler and to recover the energy. For this new smelting method, new operational calculation process has been developed. This is based on the precise heat balance and mass balance calculation toward the various kinds of coke feeding. It is programmed into the computer, the operator can carry out the real-time simulation for various directions before execution of change operation. As a result, the optimum quantity of change can be obtained. Therefore T-FSF control is improved comparing with old calculation process. In this paper these improvements are described.

11:45 AM
Balance of the Circulation Materials and Behavior of the Various Components in the Lead Smelting Process at Chigirishima Refinery: *Koichi Yamazaki*[1]; Toshio Kurikami[1]; [1]Toho Zinc Company Limited, Chigirishima Refinery, 5562-1 Higashino-cho, Toyota-gun, Hiroshima-pre. 725-0222 Japan

Toho Zinc Co., Ltd. Chigirishima Refinery has the production capacity of electrolytic lead for 95000t/year by sintering-blast furnace-electrolytic process. Lead raw materials are lead concentrates, spent batteries and dusts that contains lead from other companies, and these are handled by existing smelting process. There are many circulation materials in the process, because the system has been constructed with improving the recovery rate and protecting the environment. This report describes material balance in each process, distribution of the lead and the various components, and it examines present state of the processing. In addition, we report the result that we have tried to improve the work environment.

Non-ferrous Alloys & Light Metals - III

Wednesday AM Room: Cervantes
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: T. Battles, Dupont Chemicals; Teruo Tanabe, Kyoto University, Dept. of Matls. Sci. and Eng.

8:30 AM Keynote
Silicon in the World: *Masafumi Maeda*[1]; Takashi Ikeda[1]; [1]The University of Tokyo, Instit. of Indust. Sci., Roppongi 7-22-1, Minato-ku, Tokyo, Japan

Demand for solar cell is increasing linearly in the world. Last year its production was 5000t in the world. Market size of solar silicon will increase more because of the world demand to decrease carbonaceous emission from energy consumption. Silica is reduced by carbon in an electric furnace. The purity of silicon at this stage is one nine, namely, 90-97%. Major impurities are Fe, Ti, Ca and Al, and minor ones are P, B and carbon. The Metallurgical process is no more effective to remove the transition metals because those are less noble than silicon. Phosphorous and boron must be reduced as low as 0.001 ppm for semiconductor use. The common process for high purity silicon is Siemens C process. That is basically distillation of hydro-chlorinated silicon followed by CVD of $SiHCl_3$. The cost of such silicon is far more expensive for its popular use for solar cell. Currently, those are supplied by scrap silicon of the semiconductor production. In this paper, the world demand of silicon will be reviewed and some processes proposed will be summarized.

9:00 AM
Non-Isothermal Gravimetric Investigation on Kinetics of Reduction of Magnesia by Carbon and Aluminum: *Keiji Okumura*[1]; Hong Lan[1]; Masamichi Sano[1]; [1]Nagoya University, Matls. Process. Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

Magnesium has been well known as one of the excellent agent of desulfurization, deoxidation, etc. in iron- and steelmaking because of its high reactivity. Since its production is energy intensive, the price is very high. Thus magnesium has not widely been used as an agent in metal refining processes. In the present work, reduction of magnesia by carbon and aluminum is examined. Because the reaction is very sensitive to temperature, non-isothermal technique is applied to investigate the reaction kinetics. The effects of heating rate and reaction interface area on the reaction kinetics are studied. A kinetic model is proposed based on the experimental results.

9:25 AM

Effect of Carbon Addition on the Microstructures and Mechanical Properties of IMI 205: *Zhiqiang Chen*[1]; Yuegang Li[1]; Michael H. Loretto[1]; [1]The University of Birmingham, Interdisc. Rsch. Ctr. in Matls. for High Perform. Appls., IRC in Materials, Birmingham, Edgbaston B15 2TT UK

As-cast, as-forged, as-solution-treated, and solution treated and aged (STA) microstructures of Ti-15Mo (IMI 205) with and without carbon additions were examined using SEM and TEM. Tensile tests were carried out at room temperature. It was found that carbon addition in IMI 205 acts primarily as a titanium carbides former. Titanium carbides increase slightly the tensile strength, but decrease the ductilityof IMI 205. These observations are interpreted in terms of the morphology of titanium carbides and in terms of the phase diagrams of Ti-Mo, Ti-C, and Mo-C.

9:50 AM

Effect of Extrusion Manufacturing and Materials Processing on the Mechanical Properties and Microstructure of Aluminum-Lithium Alloys: *James Mark Fragomeni*[1]; [1]Ohio University, Dept. of Mech. Eng., Coll. of Eng. and Tech., 251 Stocker Eng. Ctr., Athens, OH 45701-2979 USA

The purpose of this study was to determine the effect that extrusion processing had on the variation in mechanical properties and microstructure as a consequence of mechanical and material processing of aluminum lithium alloy extrusions that were direct extruded at various extrusion temperatures, extrusion geometries, and extrusion ratios. An aluminum-lithium and aluminum-lithium-copper 2090 alloy billets were direct extruded at varied extrusion temperatures, strain rates, geometries, and ratios. The extrusion temperature, extrusion ratio, billet temperature, extrusion geometry, and strain rate of the extrusion were correlated to both the microstructure, and the precipitation heat treatments. The Zener-Hollomon parameters or the temperature compensated strain rates were determined for all of the extrusion processing conditions. The extrusion processing variables and microstructure were correlated to the Zener-Hollomon parameter or the temperature compensated strain rate. It was found that the smaller subgrain sizes were correlated to larger values of the Zener-Hollomon parameter. The plastic deformation was studied through finite element computer simulations of the plastic metal flow for varying conditions during simulated extrusion deformations. Standard ASTM tensile testing was performed to determine the mechanical properties of both the as-extruded, and the heat treated conditions.

10:15 AM Break

10:30 AM

Semi-Solid Processing of Al-Wrought Alloys: Andreas Wolf[2]; Kurt Steinhoff[3]; Peter J. Uggowitzer[1]; *Gian-Carlo Gullo*[1]; [1]Swiss Federal Institute of Technology Zürich, Instit. of Metall., ETH Zentrum Sonneggstrasse 3, Zürich CH-8092 Switzerland; [2]University of Stuttgart, Instit. for Met. Form. Tech., Holzgartenstrasse 17, Stuttgart D-70174 Germany; [3]SM AG, Development & QM, Altdorf CH-6460 Switzerland

With the increasing acceptance of the Thixoforging process in the last few years, also the technical and commercial requirements for this forming technology and its components increased. So far, a great deal of work has been done in the development of an efficient process and control design. However, less attention has been paid to the development of new specifically tailored aluminum wrought alloys for this specific manufacturing process. In this paper a new AlMgSi-alloy (A6082), optimized for the Thixoforging process, is presented. In the first part, the technical potential of the new alloy is specified by its microstructural evolution and flow characteristics in the semi-solid state. In the second part, the mechanical properties of thixoforged components of different conventional AlMgSi-alloys are illustrated and compared to the new alloy.

10:55 AM

Mitsubishi Materials' High Performance Oxygen Free Copper and High Performance Alloys: *Yutaka Koshiba*[2]; Tsutomu Masui[1]; Norihisa Iida[1]; [1]Mitsubishi Materials Corporation, Sakai Plant, 1-9,3-cho, Chikko Shinmachi, Sakai, Osaka 592-8331 Japan; [2]Mitsubishi Materials Corporation, Copper & Copper Alloy Prod. Dept. Non-Ferr. Alloys Co., 5-11-cho, Marunouti, Chiyodaku, Tokyo 100-8222 Japan

Mitsubishi Materials has many years of experience in the production of oxygen free copper (OFC) and its alloys with a fully continuous casting process. Four grades of OFC, commercial, electronic, superconductor, and ASTM class 1 are available according to user needs such as stabilizer for superconductivity, magnetron, and ultra vacuum systems. Along with Cu-Ag and Cu-Sn, we offer OFC based copper alloys such as Cu-Cr-Zr, Cu-Fe-P, Cu-Ni-Si and others, that are suitable for lead frame and connectors. Within the maximum sizes of 260mm X 1050mm for cakes and 385mm diameter for billets, we are very flexible and can adjust the ingot size to customer's requirements. Other products that we can supply are semi finished products such as hot rolled sheet, forged blank, extruded rod and finished products like mold for continuous casting of steel, ring mold for nonferrous casting and backing plate for sputtering target.

Thin Films & Coatings-III

Wednesday AM Room: DaVinci 1
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: S. Sastry, NMKRV College, Bangalore; Nobuaki Sato, Tohoku University, Instit. for Adv. Matls.

8:30 AM Keynote

TEM and SIMS Study of Duplex Structure of Anodic Films on Aluminum: *Sachiko Ono*[1]; Noboru Masuko[1]; [1]Chiba Institute of Technology, Dept. of Metall., 2-17-1 Tsudanuma, Narashino, Chiba 275-0016 Japan

The structure and the formation behavior of anodic films formed on aluminum in the mixture of boric acid/ammonium borate solution have been investigated with respect to electrolytic conditions. The film is composed of a boron-containing outer layer and a boron-free inner layer with disc-like crystals present at the interface. The film thickness formed at constant current density up to the same voltage decreases with increasing current density. It increases remarkably with elevated electrolyte temperature. The transport number of Al3 estimated from the thickness ratio of the outer to inner layers is 0.4 when the film is formed at the current density of 20 Am-2. It increases to 0.45 when the current density is 200 Am-2. The ratio decreases with increasing temperature. A porous cell structure is developed when the film is formed at a current density higher than 100 Am-2.

9:00 AM

Structure of Ni-Mn-Ga Films Prepared by a Sputtering Method: *Makoto Ohtsuka*[1]; Kouki Chiba[1]; Minoru Matsumoto[1]; Kimio Itagaki[1]; [1]Tohoku University, Instit. for Adv. Matls. Process., 2-1-1 Katahira Aoba-ku, Sendai 980-8577 Japan

The ternary intermetallic compound Ni_2MnGa is an intelligent material, which has a shape memory effect and a ferromagnetic property. The films of this compound can be applied for an actuator of micro-machines. The Ni-Mn-Ga films were deposited on a poly-vinyl alcohol substrate by a radio-frequency magnetron sputtering apparatus. The properties of Ni-Mn-Ga films such as chemical composition, microstructure, crystal structure and transformation temperature were investigated with respect to sputtering conditions in the present study. The composition of the films depended on the radio-frequency generating power. Each deposited film had a columnar grain structure. The Heusler type cubic crystal structure of this film, which was annealed at 1073K for 3.6 ks after separating from a substrate, changed to the tetragonal one by the martensitic structural transformation during cooling. The transformation temperature increased with increasing nickel content of films.

9:25 AM

Field Emission from CVD Diamond Fabricated by Pulse-Modulated Microwave Plasma: *Yukihiro Sakamoto*[1]; Matsufumi Takaya[1]; [1]Chiba Institute of Technology, Dept. of Precision Eng., 2-17-1, Tsudanuma, Narashino, Chiba 275-0016 Japan

Electric field emission properties from CVD diamond fabricated using pulse-modulated microwave plasma CVD were studied. Diamond was synthesized by microwave plasma CVD apparatus equipped with a pulse-modulated microwave power supply. Pulse conditions were varied from 1 to 10 seconds for pulse periods and from 0.1 to 1Hz for pulse frequency either. Surface morphologies of the deposits were differed by pulse frequency and duty cycle. As a result, FE properties prepared by different pulse duty cycle were different from continuous film which synthesized using continuous microwave significantly. Highest current density was obtained from the uneven morphology diamond film prepared at 17.5% pulse duty cycle. FE properties of the CVD diamond were depended on surface morphologies that related to the pulse conditions.

9:50 AM

Deposition of Tantalum Oxide Thin Film by Laser Ablation: *Nobuaki Sato*[1]; Junichi Hasegawa[1]; Masato Nakazawa[1]; Kohta Yamada[1]; Takeo Fujino[1]; [1]Tohoku University, Instit. for Adv. Matls. Process., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

Tantalum oxide thin films were deposited on quartz substrates by laser ablation of Ta_2O_5 target using Nd:YAG pulse laser. Deposition was observed when a laser light with its energy higher than 200 mJ and/or its repetition rate higher than 3 Hz was used. A thin film of 0.1 μm thickness was obtained by the ablation using a laser light of 300mJ and 6 Hz for 2 sec. The thickness of thin film increased with increasing energy, repetition rate and irradiation time of laser. By the SEM observation, it was found that island-like particles were deposited on quartz substrate forming a thin film. The obtained thin film, which was identified as δ-TaO by X-ray diffraction, was oxidized to Ta_2O_5 by the heat treatment in air at 1273K for 3 h.

10:15 AM Break

10:30 AM

Electron Field Emission from Nitrogen-Doped Amorphous Carbon Thin Films Prepared by Arc Ion Plating: *Hiroyuki Sugimura*[1]; Yoshiki Sato[1]; Nobuhiro Tajima[1]; Osamu Takai[1]; [1]Nagoya University, Matls. Process. Eng., Furo-cho, Chikusa, Nagoya 464-8603 Japan

Currently, there is much interest in electron field emission from amorphous carbon thin films doped with nitrogen (a-C:N) as a potential cold cathode material for flat panel displays. Here we report on the electron field emission properties of a-C:N thin films of prepared by shielded arc ion plating. The films of 150 nm in thickness were deposited on p-Si(100) substrates, as well as a-C films for control experiments. The applied electric field of ca. 10 V/micrometer was needed in order to obtain emission current from the a-C:N films at a density of 1 nA/cm2, while the a-C films showed no detectable emission even when a filed of 20 V/cm was applied. Nitrogen doping was found to be effective for lowering threshold voltage for field-emission. Dependency of the filed-emission characteristics of the a-C:N films on their preparation conditions will be further discussed.

10:55 AM

Production of Lead Oxide Film by PVD and Its Application to a Thin Film Solid Cell: *Masami Taguchi*[1]; [1]Akita University, Dept. of Matls. Sci. and Eng., 1-1 Tegata Gakuenmachi, Akita 010-8502 Japan

It is found that lead monoxide has an ionic conductivity, and the all solid state electrochemical cell using the oxide as an electrolyte has become of interest. In this study, thin film PbO2/PbO/Pb solid cells were produced on glass substrates and the discharge properties were investigated. The PbO2 layer was obtained by sputtering of a Pb target in oxygen plasma. The PbO electrolyte, which is an oxygen ion conductor, was deposited from beta-PbO powder in vacuum. The open-circuit voltage of the cell was in agreement with the theoretical value based on the thermodynamic data and the closed-circuit voltage was improved by the addition of a uni-valent impurity such as sodium or potassium to the PbO electrolyte. Moreover, the connection of the cells in series and in parallel brought out the higher electromotive force and the larger capacity.

11:20 AM

$In_xGa_{1-x}As$ Islands Grown on CaF_2 /Si(III) by Molecular Beam Epitaxy: *Y. Takeda*[1]; Y. Moriya[1]; Y. Sadayoshi[1]; Y. Nonogaki[1]; [1]Nagoya University, Dept. of Matls. Sci. and Eng., Grad. Sch. of Eng., Nagoya 464-8603 Japan

Though semiconductor/semiconductor heterostructures are widely used for many device applications, small band off-sets of the conduction bands and the valence bands restrict the utilization of the intra-subband transitions to far-infrared regions. With InAs as the quantum well and fluorides as the barrier layers, we can design three-electronic-level systems in the infrared region, which is the low loss transmission window of the silica optical fibers. To realize those structures, we have grown $In_xGa_{1-x}As$ islands on CaF_2 thin films that were epitaxially formed on Si(111) substrates by molecular beam epitaxy. By the droplet heteroepitaxy that we invented, the $In_xGa_{1-x}As$ islands with height of -30nm have been obtained successfully. Optimum processing to obtain atomically smooth surfaces of Si and CaF_2 and high quality InAs islands will be described in detail.

Powder Preparation & Processing - II

Wednesday AM Room: Dante
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Raj Mendiratta, Indian Institute of Technology, Delhi, India; Mitsuhiro Ito, Taiheiyo Cement Corporation, Eng. & Techn. Dept.

8:30 AM Keynote

Nano-Particulate Design and Preparation for Targeting and Controlled Release of Drugs: *Yoshinobu Fukumori*[1]; Hideki Ichikawa[1]; Hiroyuki Tokumitsu[1]; Futoshi Shikata[1]; Masahito Miyamoto[1]; Testuya Watanabe[1]; [1]Kobe Gakuin University, Fac. of Pharm. Sci., 518 Arise, Ikawadani-cho, Nishi-ku, Kobe 651-2180 Japan

Nanoparticles have been attracting much attention in the pharmaceutical field as drug carriers and functional components of dosage form. In this presentation, we describe nanoparticles of lipid and polysaccharide, chitosan, for cancer therapy prepared by emulsification processes, and thermosensitively drug-releasing microcapsules constructed with specially designed nanoparticles. The size of particles used in cancer therapy is an important factor that determines medical treatment strategy. The lipid nanoparticles were designed in drug targeting through blood vessels for their particle sizes to be in the range of 50 to 100 nm. The chitosan nanoparticles were designed to be around 500 nm for intratumoral administration. Finally, we will demonstrate that composite nanoparticles can be designed for thermosensitive mode of drug release from microcapsules when they are used in fine particle coating process.

9:00 AM

Continuous Hydrothermal Synthesis of Metal Oxides in Sub- and Supercritical Water: *Tadafumi Adschiri*[1]; [1]Tohoku University, Dept. of Chem. Eng., Aoba-ku Aramaki Aza, Aoba-07, Sendai 980-8579 Japan

Supercritical water can provide an excellent reaction atmosphere for hydrothermal crystallization of metal oxide particles. Due to the drastic change of properties of water around the critical point, including density, dielectric constant and ionic product, the reaction phase with O_2 or H_2 gas and the reaction rate and equilibrium can be varied to synthesize new materials or define particle morphologies. In this work, hydrothermal crystallization experiments are performed with several types of flow apparatus that allow convenient manipulation of variables such as temperature, pressure, and residence time. The proposed supercritical hydrothermal synthesis method has the following desirable features: 1) ultra fine particles can be produced, 2) morphology of the produced particles can be controlled with small changes in pressure or temperature, and 3) a reducing or oxidizing atmosphere can be applied by introducing oxygen or hydrogen or other gases. An overview of this method is given for functional materials synthesis of significant industrial interest including barium hexaferrite magnetic particles, YAG/Tb phosphor fine particles and lithium cobalt fine crystals.

9:25 AM

Preparation of Lead Zirconate Titanate Through a Spark Plasma Sintering Technique: *Kazuyuki Kakegawa*[1]; [1]Chiba University, Grad. Sch. of Sci. and Tech., 1-33 Yayoi-cho Inageku, Chiba-shi, Chiba 263-8522 Japan

Sintered body of lead zirconate titanate (PZT) was prepared through the spark plasma sintering (SPS) technique. When the mixture of raw materials was calcined at 800°C and sintered at 800°C for 10 min. with SPS technique, the sintered body had almost theoretical density but was a mixture of lead titanate (PT) and PZT. When a calcining temperature of 1100°C was employed, sintered body had a single PZT phase but a lower density. High dielectric constant was obtained for a sample calcined at 800°C, sintered with SPS technique and fired again in the air at 1100°C.

9:50 AM

Preparation and Characterization of a Pure, Ultrafine, and Partially-Stabilized Zirconia Powder from Technical-Grade Zirconium Oxychloride and the Effluent of an Acrylic Acid Plant: *G. Y. Guo*[1]; Y. L. Chen[2]; [1]Shanghai Jiao Tong University, Dept. of Matls. Sci. and Eng., 1954 Hua Shan Rd., Shanghai 200030 China; [2]Shanghai University, Coll. of Chem. and Chem. Eng., Shanghai 200072 China

In recent years the superior mechanical, thermal, electrical and chemical properties of stabilized zirconia have become the focus of growing scientific and technological interest, and hence resulted in various structural and functional applications. The great potential of the material has given rise to numerous preparation approaches to obtain the material with the excellent properties; nevertheless, an extensive number of programmes are underway worldwide aimed at understanding and improving both the preparation and behavior of stabilized zirconia ultrafine powders. This study resulted from our efforts to treat the effluent from an acrylic acid plant. The effluent contained low molecular weight carboxylic acids (acrylic and acetic acids) which show strong resistance to biological and chemical oxidation, and hence exhibited very high chemical oxygen demand and total organic carbon levels. Incineration appeared to be only an option for the effluent in order to meet the discharge legislation set by countries. A metalorganic precipitation process using zirconium oxychloride has been devised in seeking an alternative technique to conventional incineration treatment of the effluent. This study demonstrates that a precursor of advanced zirconia ceramics as well as practically pure acetic acid can be obtained by the present process from the effluent, suggesting that near-zero discharge can be achieved in the production of acrylic acid. In addition, this study shows that the polymer precursor route employing carboxylic acids in an aqueous environment may eliminate the need for toxic solvents and anhydrous atmosphere used by well-known metal alkoxide methods for the preparation of zircoia powders with well-defined particle size, purity, and crystal polymorph. Further, the present process is the first example of preparing pure, nanoscale, partially stabilized zirconia powders by using technical-grade zirconium oxychloride and the effluent from an acrylic acid plant.

10:15 AM Break

10:30 AM

Controlled Structure of Nano-Porous Silica Particles through the Use of the Spinodal Decomposition-Type Process: *Kimio Imaizumi*[1]; Toshiaki Nakai[1]; Hiroaki Shirasaka[1]; Kunihiko Takeda[1]; [1]Graduate School of Sibaura Institute of Technology, Dept. of Matls. Sci. and Eng., Sibaura 3-9-14, Minato-ku 1088548 Japan

The new type of nano-porous silica particle was considered to constructed by the spinodal decomposition-type process which was found to have narrower size of pores than those of conventional type of porous silica prepared from boron-silicate or silca balloon. Nano-porous silica particles were fabricated by the calcination of silica gel mixed with well-dispersed complex salts composed of phosphorous and molybdenum compounds. Both silica and other inorganic salts were not perfectly dissolved in the suspension solution. After removing water, the silica-inorganic salts complex was volatized and successively calcinated at 650°-750°. Then, the inorganic salts were removed by dissolving with hot water. Other inorganic salts such as potassium halides were also found to be effective as a pore size controller.

10:55 AM

A New High Quality EAF Charge: *Mingchun Lu*[1]; Jianliang Zhang[2]; Zhijia Liu[1]; Xueqi Zhang[1]; [1]Tianjin Iron Corporation, Handan 056000; [2]University of Science and Technology, Beijing 100083 Beijing

Abstract text is unavailable.

Electrolytic Processing - II

Wednesday AM Room: Michaelangelo
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: P.V. Krupakara, N.M.K.R.V. College, Bangalore; Tetsuya Akiyama, Kyushu Sangyo University, Dept. of Chem. Eng.

8:30 AM Keynote
Challenges in Electrolytic Processing of Materials: *Georges J. Kipouros*[1]; [1]Dalhousie University, Dept. of Min. and Metall. Eng., P.O. Box 1000, 1360 Barrington St., Halifax, Nova Scotia B3J 2X4 Canada

In an effort to produce low-cost materials for high technology applications various methods of production have been employed. Electrolytic processing is the most prominent in the field of the continuous, high volume metal and alloy production, and in the field of the surface modification of the final product in the form of various coatings. The fundamental scientific principles in both fields are the same although different techniques are applied. At the meantime as the metals of interest are also difficult to produce in a pure state, the first challenge is the cleanliness of the metal or alloy. Producing high purity metal electrolytically from impure raw materials requires extreme control of the electrolytic process. A second challenge comes from the extensive use of powder metallurgy techniques in the manufacturing of parts for use in automobiles. Production of metal powders, suitable for powder metallurgy processing, by electrolytic processing is feasible. Separating and collecting the metal powder from the electrolyte in high temperature operations is a challenge. Similarly, in the surface modification arena the success of the electrolytic process depends largely on techniques to control the current distribution. The fundamental principles and their applications for the success of the electrolytic processing are discussed using examples from the recent developments in the production of magnesium, lithium and titanium.

9:00 AM
Structure of Zn-Cr Alloy Electrodeposited on Steel at High Current Densities: *Masao Miyake*[1]; Tetsuji Hirato[1]; Eiichiro Matsubara[2]; Yasuhiro Awakura[1]; [1]Kyoto University, Dept. of Matls. Sci. and Eng., Yoshida-hommachi, Sakyo-ku, Kyoto 606-8501 Japan; [2]Tohoku University, Instit. for Matls. Rsch., 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi 980-8577 Japan

Zn plating film containing around 15 wt%Cr has been found to gives much higher chemical corrosion resistance to steel than the conventional zinc plating. These Zn-Cr plating will be performed at high current densities around 1 A/cm^2 and for 10 to 20 sec using acidic aqueous mixed solutions of $ZnSO_4$ and $Cr_2(SO_4)_3$ with some additives in iron-steel industry, and so the plating process should be proceeding under the condition of unsteady state. The structure and composition of the plating film thus deposited would be cahnged drastically in a direction of thickness. This work describes the structure of Zn-Cr alloy films electrodeposited on steel at high speed, which was examined using mainly XRD technique. The effects of elecrtrochemical conditions such as pH of plating bath and current density on the structure of deposit are also discussed in this presentation.

9:25 AM
Electrochemical Behavior of Copper in Ammonium Imide Type Room Temperature Molten Salts: *Kuniaki Murase*[1]; Koji Nitta[1]; Tetsuji Hirato[1]; Yasuhiro Awakura[1]; [1]Kyoto University, Dept. of Matls. Sci. and Eng., Yoshida-hommachi, Sakyo-ku, Kyoto 606-8501 Japan

Room temperature molten salts (RTMS), or room temperature ionic liquids, is a promising electrolytes for base-metal electrodeposition. In this work, a quaternary ammonium-imide type RTMS, trimethylhexylammonium bis((trifluoromethyl) sulfonyl) amide ($TMHATf_2N$), was synthesized and redox behavior of copper in $TMHATf_2N$ was investigated. Melting point of $TMHATf_2N$ was 27 degrees Celsius and an electrochemical window measured with graphite-carbon electrode was ca. 5.5 V. An electrolysis of $TMHATf_2N$ containing copper(II) bis((trifluoromethyl) sulfonyl)amide, $Cu(Tf_2N)_2$, using platinum cathode and copper anode gave a copper film with metallic luster on the cathode. Current efficiency calculated from the deposited mass of Cu metal, whose deposition is assumed to be 2-electron reduction, and the total quantity of charge passed during the electrodeposition was more than 100%, suggesting that the deposition of Cu metal occurs by the reduction of Cu(I) species which generated on copper anode as $Cu^2Cu=2Cu$.

9:50 AM
Production of Strong Reducing Solvent by Electrolyzer Equipped with Bipolar Membrane: *Satoshi Yamashita*[1]; Noboru Masuko[1]; [1]Chiba Institute of Technology, Dept. of Metall. Eng., 2-17-1 Tsudanuma, Narashino-shi, Chiba 275-8588 Japan

Newly prepared bipolar membrane is promising to become a key material of an electrolyzer for a production process of strong reducing solvent. This research is basic research for using bipolar membranes as electrolyzers of reducing agents, Cr(II), Ti(II), Ti(III) and V(II) on the laboratory scale. In this report, a bipolar membrane was tried with an electrolyzer, reducing chromic chloride to chromous chloride. An analysis of mass balance demonstrates advantages of it over the former electrolyzer equipped with an anion and/or cation exchange membrane. The effects of current density, concentration of Cr(III), circulating rate of electrolyte and interelectrode distance on the reduction rate of Cr(II) and the current efficiency were examined. No deterioration of the bipolar membrane resulted from the electrolytic reduction of Cr(III).

10:15 AM Break

10:30 AM
Formation of Hydrogen Absorption Film by Electrodeposition in Molten Salts: *Hiroaki Yamamoto*[2]; Takahiro Sakamoto[2]; Kensuke Kuroda[1]; Ryoichi Ichino[2]; Masazumi Okido[1]; [1]Nagoya University, CIRSE, Furo-cho Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Dept. Matls. Sci., Furo-cho Chikusa-ku, Nagoya 464-8603 Japan

Lanthanun-Nickel alloy film can be formed on a nickel substrate in KCl-NaCl-$LaCl_3$ molten salts by electrolysis-diffusion process. $LaNi_5$ alloy was formed by mutual diffusion between La and Ni after La deposition on Ni substrate. Though several kinds of La-Ni compound existed in deposits, the alloy compounds on Ni substrate changed by electrolysis condition. According to the open-circuit potential and CV measurements in molten salts for electrolysis, the formed compounds had a relation to the open-circuit potential. There was some potential plateau with time after La deposition. This plateau potential region corresponded to two phase coexistence region. $LaNi_5$ alloy was in stable between 1.42 V and 1.68V vs 0.1M Ag/AgCl reference electrode in molten salts.

10:55 AM
The Effect and Automatic Control of the Electrolysis of Metal Aqueous Solution by Organic Additive: *Li Shixiong*[1]; Zhang

Chuanfu[1]; [1]Central South University of Technology, Dept. of Metall., Changsha 410083 PRC

In the electrolysis, electrode or electroplating of aqueous solution of copper lead zinc nickel manganese etc, with the increase in cathodic overpotential the reducing rate of metal and the cathodic current efficiency increases, the deposit becomes more compact and smooth. According to this principle, a method and equipment of the auto-control of electrolyte quality is invented. The industrial application shows that the method turns the experienced operation after electric deposition into auto-control before electric deposition. The quality and quantity of metal is improved greatly and the consumption of electric energy is reduced.

11:20 AM

Electrowinning of Titanium in Fluoride Melt by Using Hydrogen Gas Oxidation as Anodic Reaction: *Toshihide Takenaka*[1]; Nao Murakami[1]; Takashi Matsui[1]; Masahiro Kawakami[1]; [1]Toyohashi University of Technology, Dept. of Prod. Sys. Eng., Hibarigaoka 1-1, Tempaku-cho, Toyohashi 441-8580 Japan

The electrowinning of titanium have been investigated in LiF-NaF-KF melt including K_2TiF_6 at 773-923K. The electrochemical oxidation of H_2 gas at the gas electrode of carbon was used as anodic reaction which should be a key in this process. The cathodic behavior of titanium and the anodic behavior of H_2 gas were studied by voltammetry in detail, so that the electrolytic condition for the process was clarified. The electrowinning of titanium was carried out by potentio-static electrolysis. Stable electrolysis was achieved under suitable condition without breaking out anode effect. Titanium metal was electrodeposited on the cathode though the deposits were usually powdery. It is concluded that this process is potential if the morphology of the deposit can be improved.

11:45 AM

Electrodeposition of Zn-Te Semiconductor Film from Acidic Aqueous Solution: *Ryoichi Ichino*[2]; Kensuke Kuroda[1]; Masazumi Okido[1]; [1]Nagoya University, CIRSE, Furo-cho Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Dept. of Matls. Sci., Furo-cho Chikusa-ku, Nagoya 464-8603 Japan

We investigated an electrodeposition of II-IV compound semiconductor (ZnTe) from the acid aqueous solution. The effect of electrodeposition condition such as bath contents, potential, bath temperature etc., on Zn-Te contents and morphology was estimated by SEM, EDX and XRD. Electrodeposition mechanism was evaluated by an electrochemical QCM method. On the other hand, additional AC current in the cathode electrode formed local high temperature reaction field. Using this heating electrode, we examined the effect of high temperature reaction field on Zn-Te deposit properties. Electrochemical Te ion behavior influenced to the properties of electrodeposits. Stoichiometric composition of Te for ZnTe was obtained in the deposits by using the heating electrode method.

Aqueous Processing - II

Wednesday AM Room: Parc Ballroom III
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Fiona Doyle, University of California-Berkeley; Junji Shibata, Kansai University, Dept. of Chem. Eng.

8:30 AM Keynote

New Approaches for Electrochemical Preparation of CdS Film: *Hideki Minoura*[1]; [1]Gifu University, Grad. Sch. of Eng., Yanagido 1-1, Gifu 501-1193 Japan

In recent years, much attention has been paid to materials preparation by the solution processing because of its advantage in lowering the cost as well as the stress to the environment upon the production, as compared to the conventional gas phase techniques which unavoidably require high temperature and/or vacuum. We have been trying to explore novel techniques for preparing functional compound semiconductors by the aqueous solution process. Semiconductors which we have been mainly focusing on are CdS and ZnO, which are important semiconductors finding the application in various devices. In this talk, I will explain some of the techniques which we have recently established for the preparation of highly crystallized CdS finding a possibility of electrochemical epitaxial growth. The first technique is an electrochemically induced chemical deposition (EICD), where cathodizing a conductive sustrate in an acidic chemical bath containing cadmium salt and thioacetamide gives highly crystallized CdS film. This technique realizes long-range atom-by-atom growth of CdS, yielding a film composed of highly crystallized CdS. The crystal sizes are as large as 300 nm, unreachable by any of the previously reported chemical/electrochemical methods in aqueous systems. Detailed examinations of the reaction processes including its kinetic analysis have shown that the electroreduction of protons at low current densities triggers a growth of highly crystallized CdS thin films. Selective formation of hexagonal and cubic forms of CdS by choosing counter an ion of cadmium salts was also found. Q-particulate CdS film has been also formed with this technique by adding species such as mercaptoethanol in the deposition bath which acts as adsorbent to hinder the crystal growth and promote the formation of nuclei. The other technique is electroreduction of aqueous Cd^2-SCn-complex. An advantage of this technique lies in no formation of solid particles in the bulk of the bath, which is in contrast to previously reported techniques for the preparation of CdS film. This excludes an incorporation of foreign substances into CdS film and therefore this technique can be regarded as purely electrochemical growth of CdS from a single solution.

9:00 AM

Electrochemically Deposited $LiCoO_2$ Film on Platinum Metal Substrate under Hydrothermal Condition: *Seung-Wan Song*[1]; H. Fujita[1]; K. S. Han[1]; M. Yoshimura[1]; Y. Sato[1]; [1]Tokyo Institute of Technology, Matls. and Struct. Lab., 4259 Nagatsuta Midori, Yokohama 226-8503 Japan

$LiCoO_2$ film as a cathode for rechargeable lithium microbatteries has been fabricated on the platinum metal substrate by Soft Solution Processing[1-3] in a concentrated LiOH solution at 100-200°C. Three electrode system which consists of two Pt metal plates as anode and cathode and temperature controlled Ag/AgCl as ref-

erence electrode was set up within autoclave involving the LiOH solution and 100-200 mg of cobalt metal powders in the teflon beaker. And it was hydrothermally-electrochemically treated at a constant current density of 0.01-2.5 mA/cm^2. The film fabrication has been understood in terms of dissolution of cobalt metal powders and subsequent anodic oxidative deposition on the substrate. The crystal structure and electrochemical properties were characterized using XRD, XPS, micro-Raman spectroscopy, Co K-edge XANES spectroscopy, SEM, charge-discharge experiment and cyclic voltammetry.

9:25 AM

Aqueous Oxidation of Sodium Phosphite by Dissolved Oxygen Using Sulfur Dioxide in a Catalytic Reaction: *Satoshi Yamashita*[1]; Takabumi Kondo[1]; Noboru Masuko[1]; [1]Chiba Institute of Technology, Dept. of Metall. Eng., 2-17-1 Tsudanuma, Narashino-shi, Chiba 275-8588 Japan

The effects of agitation speed, concentration of SO_2 in oxygen, temperature, pH and phosphite concentration on the oxidation of phosphite ions were examined. Dissolved oxygen alone can not oxidize sodium phosphite in aqueous solution, but when sulfur dioxide is coexistent, cooperative oxidation processes for both sulfite and phosphite can occur in aqueous solution. Stoichiometric studies are conducted according to a working hypothesis, in which hydrogen peroxide assists the oxidation of phosphite. Oxygen oxidizes sulfite to sulfate with generation of hydrogen peroxide which oxidizes phosphite to phosphate. Sulfur dioxide itself is a reducing agent, but it reacts as an oxidizing agent. So sulfur dioxide is a promising cheap reagent for waste phosphite processing.

9:50 AM

Ambient-Temperature Precipitation of Heavy-Metal Ions from Aqueous Solutions as Ferrite-Type Compounds: *Oscar Perales Perez*[1]; Kazuyuki Tohji[3]; Atsuo Kasuya[1]; Yoshiaki Umetsu[2]; [1]Tohoku University, Ctr. for Interdis. Rsch., Aramaki aza Aoba, Aoba-ku, Sendai, Miyagi-ken 980-8578 Japan; [2]Tohoku University, Instit. for Adv. Matls. Process., Katahira 2-1-1, Aoba-ku, Sendai, Miyagi-ken 980-8577 Japan; [3]Tohoku University, Dept. of Geosci. and Tech., Aramaki aza Aoba Aoba-ku, Sendai, Miyagi-ken 980-8578 Japan

The present work investigated the conditions to produce magnetite and various M-bearing ferrites (M: Zn, Cu, Co, Ni, Cd, etc...) from aqueous solutions at 25°C by simultaneous control of the oxidizing conditions and pH. The formation of the solids was followed by monitoring the ORP and proton release during the aerial oxidation at constant pH. Only mildly oxidizing conditions and a moderate oxidation rate were conducive to crystalline ferrites without heating of the precipitates. It was also found that increasing the Fe(II)/M mole ratio in starting solutions enhanced the stability of the ferrites. When the formation of the ferrite was incomplete, aging of the solids in their mother liquors at 25°C promoted their crystallinity. Furthermore, the formation of the ferrites permitted the elimination of the heavy metal ions to sufficiently low concentration. The characterization of the precipitates evidenced their magnetic nature (M_s>60emu/g) and the effective incorporation of metal ions into the ferrite structure.

10:15 AM Break

10:30 AM

Preparation of Mono-Dispersed Droplet using Ultrasonic: *Tsuyoshi Arai*[1]; Kimio Imaizumi[1]; Kunihiko Takeda[1]; [1]Sibaura Institute of Technology, Dept. of Matls. Sci. and Eng., Sibaura 3-9-14, Minato-ku 1088548 Japan

The study on the preparation of mono-dispersed droplet was performed using ultrasonic as one of the steps of producing the perfectly mono-dispersed particles. The ultrasonic system was constructed with 39kHz generator, an amplifier, a electrostrictive element, and a bolted Lanjevin type horn which was installed in cell. For the first time, a liquid pillar under a nozzle of the cell was stably formed. Then, dense and coarse parts were alternatively constructed by oscillating the wavelength of the ultrasonic. The diameter of those droplets was found mono-disperse, therefore, it was measured by the gmulti-images photograph method using the synchronized stroboscope with the ultrasonic. From the results, more than 30,000 of perfectly uniform droplets were observed by this method.

10:55 AM

Surface Conditioning of ABS for Metallization-A Study on Cleaner Processes: *Luiz Alberto Teixeira*[1]; Marcela Costa Santini[1]; [1]Catholic University of Rio de Janeiro, Dept. Matls. Sci. and Metall., CP. 38008, Gavea, Rio de Janeiro, RJ 22453-900 Brazil

Surface conditioning of plastics prior to metallization is generally conducted with sulphuric chromic acid solutions. Although these baths perform well technically, the presence of chromium VI imposes serious operating problems of an environmental nature. The present work reports the results of a study of surface conditioning of ABS using solutions based on sulphuric acid, with hydrogen peroxide and/or nitric acid, replacing chromic acid, as oxidants. Process performance was evaluated by SEM and by the uniformity and adherence of electroless nickel coatings applied on selected conditioned samples. The results showed that the evaluated types of solutions could be optimized to perform as well as the chromic-based baths, with a much superior environmental quality.

11:20 AM

New Hydro Metallurgical Process of Copper Anode Slimes at Saganoseki Smelter & Refinery: *Akinori Toraiwa*[1]; Yoshifumi Abe[1]; [1]Nippon Mining and Metals Company Limited, Saganoseki Smelter and Refinery, Saganoseki-cho, Oita-pre. 879-2201 Japan

As part of its efforts to reinforce its copper business, Nippon Mining & Metals launched a fully hydro-metallurgical process capable of processing 2,000 tons of slimes per year in October 1997 at Saganoseki Smelter & Refinery. The new process, although facilitated by Dr. Hoffmann's proposal, was completed as a practical one building mainly on Nippon Mining & Metals' own technologies and operational know-how. Its major features are hydrochloric leaching of decopperized slime, solvent extraction of precious metals, and three-stage reduction of selenium-containing solution. The new process is superior to the conventional pyro-metallurgical one in terms of operation costs and more environmentally-friendly. In addition, a remarkable increase in gold purity (more than 99.997%) and a reduction by half of the gold inventory time from 16.5 days to 8 days were registered. The current annual production rates of major products are 30 tons for gold and 380 tons for silver.

11:45 AM

Ultra-High Purification of Transition Metals by Anion Exchange Applying Various Oxidation States: *Tamas Kekesi*[1]; Masahito Uchikoshi[2]; Kouji Mimura[1]; Yongfu Zhu[1]; Minoru Isshiki[1]; [1]Tohoku University, Inst. for Adv. Matls. Process., 2-1-1 Katahira Aoba-ku, Sendai, Miyagi 980-8577 Japan; [2]MRC Japan, Inc., R&D Div., 2-1-15 Meigetsu, Tagajo, Miyagi 985-0843 Japan

Anion exchange is shown to be an efficient way of separating the major transition metals in chloride solutions. It is demonstrated that complexation by chloride ions may lead to the formation of sorbable anionic species in cases of a number of metal-

lic elements, depending on the oxidation state and the hydrochloric acid concentration. Predominance of different species has been estimated by computations, based on the available stability data, while anion exchange sorption functions have been determined by the equilibrating method for various metals in different oxidation states. Separation schemes to eliminate all the impurities from the solutions of a number of transition metals are proposed according to these fundamental results. Anion exchange separation processes, applying laboratory and pilot scale columns, have been developed and tested for the purification of copper, zinc, cobalt and iron. The efficiency of operation is assessed by analysis of the obtained elution curves.

Composite Materials - IV

Wednesday AM Room: DaVinci 2&3
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: S. C. Sharma, R.V. College of Engineering India; Naoyuki Kanetake, Nagoya University, Dept. of Matls. Proc. Eng.

8:30 AM Keynote
Processing and Characterization of Cu-Al_2O_3 Nano-Scale Composites Produced by In-Situ Reduction: Marcelo Senna Motta[1]; Eduardo A. Brocchi[1]; P. K. Jena[1]; *Guillermo Solórzano*[1]; [1]PUC-Rio, Matls. Sci. and Metall. Dept., P.O. Box 38090-Gávea, Rio de Janeiro, RJ 22451-970 Brazil

The synthesis of the Cu-Al_2O_3 (1wt%) and Cu-Al_2O_3 (5w%) cermet were carried out by a three step procedure. Initially, a powder mixture consisting of CuO and Al_2O_3, was cold pressed. This was followed by the preferential reduction of the copper oxide with hydrogen and by sintering at 900°C. The procedure leads to an in-situ formation of the composite, imparting to it special microstructural features. FEG SEM has suggested that the alumina particles are distributed in a nanometric scale. TEM diffraction contrast imaging have shown well-developed copper crystals surrounded by a fine dispersion of Al_2O_3 nano-scale particles. It has also been observed that cermets with 5 wt% Al_2O_3 are capable of developing, in the sample, an even finer microstructure where Cu crystals, in the order of 50nm size dispersed and exhibiting twining, are surrounded by even smaller particles of Al_2O_3. Current investigation is being developed to assess the evolution of the microstructure resulting from cold rolling followed by annealing.

9:00 AM
Damping Behaviour of Aluminite Particulate Reinforced ZA-27 Alloy Metal Matrix Composite: *Shanta Sastry*[1]; *Jayagopal Uchil*[2]; *M. Krishna*[3]; [1]N.M.K.R.V College for Women, Phys. Dept., Jayanagar, Bangalore, Karnataka 560011 India; [2]Mangalore University, Matl. Sci. Dept., Mangala Gangotri, Dakshina Karnataka, Mangalore, Karnataka 574199 India; [3]R.V. College of Engineering, Mech. Eng. Dept., Rsch. and Dev., Bangalore, Karnataka 560059 India

Damping behaviour of aluminite particulate reinforced ZA-27 alloy metal matrix composites processed by compocasting method is studied. The weight percentage of particulate reinforcement is 0,1,2,3, & 4%. A dynamic mechanical analyzer is used to measure the damping capacity and dynamic modulus at the natural frequency of the specimen over a temperature range of 30°C to 300°C both in the heating and cooling cycle. The damping capacity of the materials is shown to increase with increasing temperature and weight percentage of aluminite reinforcement although this is accompanied by a decrease in dynamic modulus with temperature. The damping capacity and elastic modulus exhibit a sharp decrease in their values around 300°C as ZA-27 alloy undergoes a first order phase transition in this region which is confirmed by Differential Scanning Calorimetric studies. Several damping mechanisms have been evaluated theoretically for the material under study. The damping capacity at low temperatures is attributed primarily to coefficient of thermal expansion mismatch induced dislocations and high intrinsic damping of the matrix. At higher temperatures the damping capacity is attributed to ZA-27 aluminite interface, phase transformations and Thermoelastic damping.

9:25 AM
Prediction of Galvanic Corrosion Rates for Garnet/LM-6 Metal Matrix Composites in Sodium Chloride Solutions: *V. T. Vijayalakshmi*[1]; Jayagopal Uchil[1]; K. H. Thipperudrappa[1]; M. Krishna[1]; [1]N.M.K.R.V. College for Women, Dept. of Chem., Jayanagar III Block, Bangalore 560011 India

Observations made during the galvanic-corrosion behaviour of aluminum-LM6 alloys reinforced with garnet particles were analysed in sodium chloride solutions of different normalities using potentiodynamic polarisation technique. Aluminium reinforced with garnet particulate in percentages of 4, 8, and 12% by weight were prepared by the melt stirring process. Both composites and the corresponding base alloys were subjected to identical test conditions to understand the influence of the reinforcement on alloy corrosion behaviour. As normality of Sodium Chloride (NaCl) increased, numerous geometrical pits were found to be nucleated in both composite and matrix. Composites are more prone to corrosion and pit formation than the matrix alloy, which may be due to residual contaminants of MMC's during processing and the formation of inter-phases between reinforcement and matrix. The reinforced/matrix interfaces were found to be favourable sites for nucleation of corrosion pits. On the other hand the test reveals that corrosion resistance of both alloy and composites were found to decrease with exposure time, which may be presumably due to the pit formation on the surface, which leads to higher exposed area in corrosive media. The corrosion behaviour of Lm-6/hematite metal matrix composite was also studied by scanning electron microscopy.

9:50 AM
Damping Behavior and Mechanical Properties of a MnB Particle-Reinforced INCRAMUTE Matrix Composite: *Fuxing Yin*[1]; Yoshiaki Ohsawa[1]; Akira Sato[1]; Kohji Kawahara[2]; [1]National Research Institute for Metals, Matls. Creation Station, Sengen1-2-1, Tsukuba, Ibaraki 305-0047 Japan; [2]B.B.Materia Company Limited, Asumigaoka, Chiba 267-0066 Japan

INCRAMUTE alloy (nominally 45Mn-53Cu-2Al wt.%) has a specific damping capacity as high as 40%, through a solution and aging treatment. The damping behavior of the alloy is highly temperature-dependent and shows a peak near room temperature. The inadequate strength in the high damping condition restrained the practical application of the alloy. Ferromagnetic MnB particles (1-5wt.%) were dispersed in the alloy during induction re-melting in an argon atmosphere. Surface modification of the MnB particles by MA and suitable control of the addition process caused a homogeneous distribution of MnB particles in the alloy. In the composite containing 2wt.% MnB particles, temperature dependence of damping capacity was improved and higher damping capacity was retained in the temperatures over 100°C. Meanwhile, the Young's modulus and tensile strength were

also increased by about 20%. The roles of MnB particle in improving both damping behavior and mechanical properties were also discussed.

10:15 AM Break

10:30 AM
Elastic Behavior of Aluminite Particulate Reinforced ZA-27 Alloy Metal Matrix Composite: *Shanta Sastry*[1]; Jayagopal Uchil[2]; *S. C. Sharma*[3]; M. Krishna[3]; B. M. Satish[3]; [1]NMKRV College for Women, Dept. of Phys., Jayanagar, Bangalore, Karnataka 560011 India; [2]Mangalore University, Dept. of Matl. Sci., Mangalore India; [3]RV College of Engineering, Rsch. and Dev. Dept., Dept. of Mech. Eng., Bangalore 560 059 India

A well-considered composite of ZA-27 reinforced with weight percentage 0, 2, 4, and 6% of aluminite particulate has been made by compocasting technique. The elastic properties of ZA-27/aluminite particulate composite were determined by using UTM. The results showed that the modulus of elasticity and UTS of the composite gradually increases with increase in the weight percentage of reinforcement. However, the overall ductility decreased monotonically with an increase in reinforcement. The results of these tests as well as the predictions of several models for composite strengthening available in the literature, were compared with the experimental results. The data observed from Young's modulus measurements studies were compared with theoretical results predicted by Hashim's model and Esnelby's model, and it was observed that a better agreement was with those of the later model. SEM analysis of the fracture surface as well as the subsurfaces are used to explain the observation made.
ech. Edu., Teknikokullar, Ankara 06500 Turkey

Powder Preparation & Processing-III

Wednesday PM Room: Dante
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Fumitaka Tsukihashi, The University of Tokyo, Dept. of Adv. Matls. Sci.; K. K. Sharma, Defence Metall. Research Lab., Hyderabad, India

2:00 PM
Analysis of Anionic Polymer Dispersant Behavior in Dense Silicon Nitride and Silicon Carbide Suspensions by Using ANAFM: *H. Kamiya*[1]; M. Nojiri[1]; H. Hasegawa[1]; S. Matsui[1]; Y. Fukuda[1]; M. Tsukada[1]; [1]Tokyo University of Agriculture and Technology, Koganei, Tokyo, Japan

The present paper focused on the action mechanism of anionic polymer dispersants in dense Si_3N_4 and SiC suspension. By using atomic force microscope, the relationship between macroscopic suspension viscosity and microscopic adsorbed structure of polymer dispersant at solid/liquid interface and surface interaction in suspensions were analyzed in various pH and additive conditions of dispersant. The addition of anionic polymer dispersant decreased Si_3N_4 and SiC suspension viscosity and increased short-range steric repulsion force on nonoxide surface in solution at pH>6 which was iso-electric point of each materials. It was assumed that the increase of repulsive force was promoted by a hydrophobic adsorption of polymer dispersant on nonoxide particles.

2:25 PM
Structure and Properties of an F75 Co-Cr-Mo Superalloy Produced by Fluidized-Bed Nitriding of Gas-Atomized Powders, HIP-Consolidation, and Heat-Treatment: *Wade L. Karlsen*[1]; [1]Helsinki University of Technology, Lab. of Eng. Matls., Otakaari 4, P.O. Box 4100, Espoo, Hut 02015 Finland

Revision surgery to replace joints in the human body requires materials that are strong, long-lasting, and biocompatible. Metallic implants made of cobalt-chromium-molybdenum alloys are already popular because of their proven corrosion and wear resistance properties, but there is an on-going effort to economically improve their performance. Alloying with inert nitrogen can enhance the mechanical properties of the alloy, enhance its corrosion resistance, and elevate the temperature at which the metallic implants can be thermally treated, without increasing the threat to the human body. The current work has produced a high-nitrogen Co-Cr-Mo alloy through HIP-consolidation of metal powders that have undergone nitriding in a mechanically fluidized bed. The already fine microstructure of the powders combined with the increased nitrogen content resulted in significant property gains in the consolidated materials. Significance: Of interest to those seeking to improve the performance of superalloys through processing, or involved with nitrogen alloying, or with enhancing the performance of biomedical components.

2:50 PM
Production of Fine Powder of Tantalum by Reduction of Tantalum Chloride Vapor with Hydrogen: *Fumitaka Tsukihashi*[1]; Hitoshi Iijima[2]; [1]The University of Tokyo, Dept. of Adv. Matls. Sci., Grad. Sch. of Front. Sci., 7-3-1 Hongo, Bunkyo-ku, Tokyo, Tokyo 113-0033 Japan; [2]Showa Cabot Supermetals K.K., New Prod. Rsch. Off.

Fine powder of tantalum is used as materials of electric condenser. It is important to control the distribution of particle diameter of tantalum fine powders for the commercial use. In this study, fine powders of tantalum were produced by reducing tantalum chloride vapor with hydrogen gas at 1673 to 1773K. The tantalum chloride vapor was reduced with hydrogen and the produced fine particles of tantalum were collected to observe the distribution of particle diameter. The effects of reaction temperature, partial pressure of hydrogen gas and vaporization temperature of tantalum chloride on the distribution of particle diameter and reaction efficiency of reduction were determined. The mechanism of reduction reaction of tantalum was discussed.

3:15 PM Break

3:30 PM
Processing of Soft Ferrites by the Citrate Precursor Method: *Raj Gopal Mendiratta*[1]; Anjali Verma[1]; Tara Chand Goel[1]; [1]Indian Institute of Technology Delhi, Phys. Dept., Hauz Khas, New Delhi, Delhi 110016 India

Processing of ferrites has gained tremendous importance in recent times to meet the high performance demands on ferrites in keeping with the fast emerging technologies. The quality of ferrite powders has strong influence on the performance of final device. Fabrication of high performing ferrite components requires starting ferrite powders of precise stoichiometry. purity and microstructure. It is difficult to obtain these attributes by the conventional ceramic method. The key to obtaining quality powders lies in the solution techniques. With this in view, soft ferrites have been prepared by a wet chemical process, known as the citrate precursor method, which involves the formation of citrate complexes of the constituent cations. The advantages of this process are the ease of preparation and close control of composition, homogeneity, purity and particle size. Preliminary measurements

on ferrites prepared by the citrate precursor method have shown resistivity higher by two orders and electrical and magnetic losses lower than those for the conventionally prepared samples.

Waste Management-III

Wednesday PM Room: Michaelangelo
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Georges J. Kipouros, Dalhousie University, Halifax Canada; Toshiharu Fujisawa, Nagoya University, Res. Cen. for Adv, Waste And Emiss, Mgt.

2:00 PM Keynote

Industrial Wastes Management for Construction Materials: *V. A. Mymrin*[1]; Alfonso J. Vazquez Vaamonde[2]; P. A. Alexeev[3]; [1]Profsouznaia Str., 152-4-119, Moscow 117321 Russia; [2]Avda Gregorio del Amo 8, Madrid 28040 Spain; [3]Profsouznaia Str. 152-4-71, Moscow 117321 Russia

Such industrial wastes of Spain and Russia as mud of Jarosite, dust of electric steel production (EAFD), refuses of Pyrite, "red mud" of Bauxite's and liquid alkaline wastes of Al-production, white and black slags of metal works, incinerator ash and slag, burnt refuses of coal mining and processing, all kinds of slag dumped by iron and steel industry, some kinds of nonferrous metal work slags (Ni, Al, etc.), foundry sand wastes of machine building plants, slime of silica brickyards, dry or sluiced ashes of thermal power plants, liquid, sludge or powdered wastes of chemical, paint and varnish, petrochemical, engineering, and other industries can be used for the producing of different construction materials. If they are not recycled, they will consume arable areas and need significant financial investments and efforts for neutralization and subsequent disposal. These are findings of laboratory and field tests in different parts of Russia as roads and airfields bases. The road strength and elasticity increase more than twice in comparison with traditional materials. The strength of these materials has been continuously improving over more than 15 years of use; as a result, the base of a road maintains its high quality for an infinite period. The values of the properties can be considerably improved with changing of proportions in the initial mixtures. The environment is conserved both by recycling the industrial wastes and by slowing the expansion of areas taken up by quarries.

2:30 PM

Extraction of Valuable Compounds from Egyptian Zinc Concentrate: Kamilia A. El-Barawy[1]; Samir Z. El-Tawil[1]; *M. B. Morsi*[1]; M. M. Nasr[1]; Zaki I. Mohamed[1]; [1]Central Metallurgical Research and Development Institute, Pyrometall. Div., P.O. Box 87, Helwan, Cairo Egypt

In Egypt, considerable reserves of oxidized zinc ore are present in Um Ghieg area eastern desert. The possibility of extraction of zinc from these ores and choosing an acceptable technology for processing these deposits commercially are of vital importance to the national economy. The present investigation deals with the sulphation of zinc concentrate by ammonium sulphate as sulphatizing agent at different conditions to achieve the appreciable formation of soluble sulphates. The maximum zinc recovery as soluble sulphate equals to 95% at optimum conditions (1.5 times stoichiometry ammonium sulphate, at 600°C for 1 hour). The obtained leached zinc sulphate is subjected to purification process to prepare high pure zinc metal and zinc compounds. The products obtained are analyzed chemically and identified by X-Ray. The mechanism and kinetics of sulphatization of zinc concentrate are considered and the activation energy is calculated and equals to 8.93 Kcal/mol. A process flow sheet for the sulphatization process of zinc concentrate by ammonium sulphate is proposed to prepare zinc sulphate, zinc metal and other valuable zinc compounds.

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Model of Reaction between Steelmaking Dust and Polyvinyl Chloride: *Masahiro Hirasawa*[1]; [1]Tohoku University, Instit. for Adv. Matls. Process., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

As a fundamental study on pyrometallurgical process that involves recycling steel-making dusts, of which major components are oxides of Zn and Fe, and waste polyvinyl chloride simultaneously at a temperature below 1273K, the reactions between the mixture of ZnO Fe_2O_3 and PVC or the compound $ZnFe_2O_4$ and PVC are investigated in a series of small scale furnace experiments under Ar atmosphere at 1000 to 1200K. From the experimental results, it is shown that the chlorination of Zn in the oxides by Cl in PVC, the vaporization of $ZnCl_2$, the reduction of iron oxide to metal Fe by C and H in PVC, and, thereby, the separation of Zn and Fe in the oxides take place simultaneously at the temperature ≅ 1200 K, and the reaction mechanisms are discussed from kinetic and thermodynamic viewpoints.

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Bioleaching of Waste Nickel Condenser Powder: *Hiroshi Nakazawa*[1]; Wu Shouming[1]; Hayato Sato[1]; Yasuo Kudo[1]; [1]Iwate University, Fac. of Eng., 4-3-5, Ueda, Morioka-shi, Iwate Prefecture 020-8551 Japan

In order to recover copper from a waste nickel condenser powder, bioleaching experiments were carried out using Thiobacillus ferrooxidans in a shaking flask. In preliminary study using metal copper powder, the dissolution of copper was accelerated with addition of ferric iron in the bioleaching of copper powder while not in the absence of ferric iron. Ferric iron oxidizes metal copper, and Thiobacillus ferrooxidans oxidizes ferrous iron resulting from the oxidation of metal copper, whereby the dissolution of copper proceeds. The copper content of a nickel condenser powder used in this study was 3.5%. Xray analysis showed the peak of barium titanate. The dissolution rate of copper increased with increase in the concentration of ferric iron in the bioleaching of the waste nickel condenser powder.

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The Roles of Metallic Compounds on Dioxins Generation-Decomposition Behavior from Thermodynamics and Quantum Chemistry: *Nagahiro Saito*[1]; Mituhito Hirota[1]; Takahiro Ishizaki[1]; Akio Fuwa[1]; [1]Waseda University, Depts. of Matl. Sci. and Eng., 3-4-1 Okubo, Shinjuku-ku, Tokyo 169-0027 Japan

Owing to their extremely high toxicity in the environment, the dioxins have been attracting social, technical and scientific attention in recent years. The authors theoretically predicted the thermodynamic properties of all the dioxins via quantum chemistry and statistical thermodynamics and elucidated the generation-decomposition behavior of dioxins in the equilibrium calculation of C-H-O-Cl system. In actual combustion processes, it is said that metallic compounds have much influences on the dioxins behavior. The aim of study is that the roles of metallic compounds the generation-decomposition behavior of dioxins is revealed from the viewpoints of thermodynamics and quantum chemis-

try; equilibrium calculation, potential stability diagram and potential energy profile.

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Zinc and Lead Recovery from Model Ash Compounds: *Shin-ji Abe*[1]; Tadakazu Kagami[1]; Katsuyasu Sugawara[2]; Takuo Sugawara[2]; [1]Mitsubishi Material Company, 1-3-25 Koishikawa, Bunkyo-ku, Tokyo 112-0002 Japan; [2]Akita University, Fac. of Eng. and Res. Sci., 1-1 Tegata Gakuen-cho, Akita City, Akita Pref. 010-8502 Japan

In order to design efficient recovery processes of heavy metals from ash, release behavior of zinc and lead was followed during various heat treatment conditions from model ash compounds consisted of zinc oxide, lead oxide, silicate and calcium chloride etc. with the use of a thermogravimetric analyzer and fixed beds. Rapid release of both chlorides of zinc and lead from solid phase can be explained by several steps including liquid phase reactions at the temperatures up to 1000°C. The kinetic papameters of these reaction steps were determined and applied to the simulation of chlorides recovery from actual incinerator ash.

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Recovery of Nickel from Spent Electroless Nickel Plating Solution by Solvent Extraction: *Mikiya Tanaka*[1]; Mikio Kobayashi[1]; Mansour Ahmed S. AlGhamdi[1]; Kenji Tatsumi[2]; Hirotaka Senba[3]; Yukinori Saiki[3]; [1]National Institute for Resources and Environment, Matls. Process. Dept., 16-3 Onogawa, Tsukuba, Ibaraki 305-8569 Japan; [2]National Institute for Resources and Environment, Hydro. Environ. Protect. Dept., 16-3 Onogawa, Tsukuba, Ibaraki 305-8569 Japan; [3]Japan Kanigen Company Limited, 3-11-10 Edagawa, Koto-Ku, Tokyo 135-0051 Japan

Spent electroless nickel plating solution contains low concentrations of iron and zinc as impurities. In order to recover nickel ion in the solution and, at the same time, to remove such impurities, a flowsheet employing solvent extraction of nickel by a chelating extractant followed by the stripping by acid is proposed. By this flowsheet, the solution concentrated in nickel ion with high purity is obtained, and this nickel ion would be reused in the electroless plating process.

Iron & Steelmaking-III

Wednesday PM Room: Parc Ballroom III
November 8, 2000 Location: Renaissance Parc 55 Hotel

Session Chairs: Patrick R. Taylor, University of Idaho; Masanori Iwase, Kyoto University, Dept. of Engy. Sci. and Techn.

2:00 PM Keynote
Influence of the Preforming Process of IF-Steel on the Impact Behavior: *Angelika Spalek*[1]; Gerd Reisner[1]; Ewald Werner[1]; Andreas Pichler[2]; Peter Stiaszny[2]; [1]Christian Doppler Laboratorium für Moderne Mehrphasenstähle, Lehrstuhl A für Mechnik; TU-München, Boltzmannstr. 15, Garching D-85747 Germany; [2]VOEST-ALPINE STAHL Linz GmbH, Rsch., Dev. and Test. Div., Voest-Alpine Str. 3, Linz A-4034 Austria

Interstitial free (IF) sheet steels are used for deep drawing parts of complex geometry because of their excellent formability. Unfavorable production conditions may cause a susceptibility to brittle fracture, when a deep drawn cup is subjected to a high-speed deformation at low temperature. This phenomenon is called secondary work embrittlement (SWE). A comparison of test results obtained from various IF-steel grades shows the influence of the processing conditions on the susceptibility of the material to SWE. The correlation between the parameters of the preforming process and the impact behavior is studied. The test allows to identify the temperature at which a crack can be initiated by impact deformation.

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The Fracture Toughness of Cold Work Tool Steels: *Johann Blaha*[1]; Ewald Werner[1]; Werner Liebfahrt[2]; [1]Christian-Doppler-Laboratorium, Lehrstuhl A für Mechanik, TU München, Boltzmannstrasse 15, Garching D-85747 Germany; [2]Böhler Edelstahl GmbH, Mariazellerstraße 25, Kapfenberg A-8605 Austria

To meet the rising demands for an improved lifetime and shorter processing cycles tool steels are increasingly more produced via the powdermetallurgical (PM) route. In addition to the benefits of a net-shape technology this production route makes possible to set up a homogeneous microstructure consisting of a tough matrix with uniformly distributed hard carbides of globular shape. In this work the properties of several PM-cold work tool steels are characterized by means of plane strain fracture toughness testing, "Ultra-Mikro"-indenting and quantitative analysis of their microstructure. The fracture toughness depends mainly on the volume fraction, size, shape and distribution of the primary carbides, since crack propagation frequently takes place along the carbide/matrix interface due to matrix decohesion. If, however, the crack propagates through the matrix, more energy has to be spent, resulting in higher fracture toughness values K_{1C}. The mechanical in-situ properties of the matrix material are characterized by indentation tests performed with an ultra-mikro indenter in a scanning electron microscope. These tests help to quantify the local material properties responsible for a overall behaviour of the compound.

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Grain Refining Process and Fracture and Toughness of Ultra-Fine Low-Carbon Steel Bars: *Toshihiro Hanamura*[1]; Tohru Hayashi[1]; Hiroshi Nakajima[1]; Shirou Torizuka[1]; Kotobu Nagai[1]; [1]National Research Institute for Metals, 1-2-1 Sengen, Tsukuba 305-0047 Japan

For structural materials, it is required to improve both its strength and toughness simultaneously. The authors have been studying high strength ferrite steels by grain refining. There has been no study that examined the effect of grain refining on the toughness in the 1μm region of grain size. In this study, fracture surfaces of ultra-fine grain steels after impact tests were examined based on the effective grain size of fracture. The difference of impact properties between transverse and longitudinal orientations of the groove-rolled fine-grain steels was also examined. It was confirmed that even in the ultra-fine grain structure steels, the concept of effective grain size of fracture affirmed in the QT steels is applicable. Also, the effect of grain refining on the fracture behavior was confirmed. However, the effective grain size of fracture for the fine-grained steels is a few times that of the ferrite grain size.

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Formation of Austenite Grain in Hypo-Peritectic Carbon Steels: *Masayuki Kudoh*[1]; Tohru Maruyama[2]; Yohichi Ito[1]; [1]Hokkaido University, Div. of Matls. Sci. and Eng., Grad. Sch. of Eng., Kitaku N13W8, Sapporo 060-8628 Japan; [2]Hokkaido University, Div. of Matls. Sci. and Eng., Grad. Student, Kitaku N13W8, Sapporo 060-8628 Japan

When a hypo-peritectic carbon steel is unidirectionally solidified, three regions of delta dendrite, fine columnar and coarse

austenite (γ) grains are observed. The delta dendrite is corresponding to the (L ζ) phase, and fine columnar γ grain is (L ζ γ) and (ζ γ) phases. The narrow size of the fine columnar γ grain is related to the primary dendrite arm spacing. The γ grain boundaries is formed in the skeleton of the delta dendrite. The fine columnar γ grain becomes suddenly coarse when the delta phase completely disappears. Therefore, it seems that the delta phase in hypo-peritectic carbon steel acts as the prevention of the growth of the γ grain.

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Modelling of Austenite Decomposition of Hot-Rolled Dual Phase Steels during the Cooling in Run-Out Table: *Andrej Samoilov*[1]; Yuri Titovets[2]; Nikolai Zolotorevskii[2]; Gottfrid Hribernig[1]; Peter Stiaszny[1]; [1]VOEST-ALPINE STAHL LINZ, R and D Hot Rolled Sheet, WFC, Voest-Alpine-Strasse 3, P.O. Box 3, Linz A-4031 Austria; [2]St.-Petersburg State Technical University, Metalphy. & Comp. Tech. for Matl. Sci. Dept, Politechnicheskaya 29, St. Petersburg 195251 Russia

The model has been developed allowing to predict simultaneously both transformation product portions and mean ferrite grain size from the same common principles as a result of austenite decomposition during any cooling path of dual phase steels. The transformation products considered are polygonal ferrite, pearlite, bainite and martensite. The model is based on the classical equations of nucleation-growth theory and also contains some empirical parameters. The chemical driving forces for nucleation and composition of elements at the phase interfaces are derived by computation thermodynamic. The empirical parameters related to the model were determined on the basis of a series of austenite transformation dilatometric curves and grain size measurements for continuous and interrupted cooling of several steel types. The model was applied to complex cooling conditions of the run-out table of the hot strip mill at VOEST-ALPINE STAHL LINZ GmbH.

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Phase Equilibria of the MnO-SiO_2-CrO_x System: *Mitsuru Tanahashi*[1]; Nobuo Furuta[1]; Tsuyoshi Taniguchi[1]; Toshiharu Fujisawa[2]; Chikabumi Yamauchi[1]; [1]Nagoya University, Dept. of Matls. Sci. and Eng., Nagoya, Aichi 464-8603 Japan; [2]Nagoya University, Rsch. Ctr. for Adv. Waste and Emiss. Mgt., Furo-cho Chikusa-ku, Nagoya, Aichi 464-8603 Japan

In order to control non-metallic inclusions formed by the deoxidation of stainless steel with silicon, it is essential to clarify the equilibrium relations among non-metallic inclusions of the MnO-SiO_2-CrO_x system and molten stainless steel. In the present study, the phase equilibria of the MnO-SiO_2-CrO_x system were measured by conducting the following experiments under controlled oxygen partial pressure, PO_2 at 1873K: (1) Phase equilibria of the MnO-Cr_2O_3 binary system were measured for PO_2=$2x10^{-6}$-$2x10^{2}$ Pa. (2) Phase equilibria of the MnO-SiO_2-CrO_x ternary system were measured for PO_2=$2x10^{-6}$ Pa. Based on the results, the phase diagram of this ternary system was established under the condition of the Si-deoxidation process of stainless steel (PO_2=$2x10^{-6}$ Pa and T=1873K).

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Phase Diagrams of FeO-Containing Slags Derived from Activity Measurements: *M. Myochin*[1]; K. Wakimoto[1]; Yuichi Uchida[1]; Masanori Iwase[1]; Alexander Mclean[2]; [1]Kyoto University, Ferrous Metall. Rsch. Grp., Dept. of Engy. Sci. and Tech., Yoshida-Honmachi, Sakyo-ku, Kyoto 606-8501 Japan; [2]Kyoto University, Ferrous Metallu. Rsch. Grp., Dept. of Ene. Sci. and Tech., Yoshida-Honmachi, Sakyo-ku, Kyoto 606-8501 Japan

By using an appropriate thermodynamic model, it is possible to derive chemical potentials of components in binary, ternary, quaternary or even multi-components systems. In turn, phase diagrams can be derived from chemical potentials of components, determined through appropriate experimental techniques. The present paper is aimed at documenting such derivations of the phase diagrams by using experimental data for the activities of FeO in the systems, FeO-Al2O3-CaO, FeO-BaO-BaF2, FeO-BaO-BaCl2, FeO-BaO-SiO2, FeO-CaO-CaCl2 and FeO-SrO-SrCl2.

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