JMR Abstracts

Editor-in-Chief: Robert A. Laudise • Associate Editors: Shigeyuki Sōmiya, Heinrich J. Wollenberger

Abstracts for October 1998 Journal of Materials Research *Volume 13, Number 10*

TABLE OF CONTENTS

October JMR issue includes a SPECIAL SECTION:
Photovoltaic Energy Conversion

ABSTRACTS

SPECIAL SECTION
PHOTOVOLTAIC ENERGY CONVERSION

Photovoltaics characterization: A survey of diagnostic measurements
L.L. Kazmerski
(National Renewable Energy Laboratory)

The advancement of the photovoltaic technology is closely linked to the standard evaluation of the product, the diagnosis of problems, the validation of materials and cell properties, and the engineering and documentation of the ensemble of device properties from internal interfaces through power outputs. The focus of this paper is on some of the more common, visible, and important techniques dealing with physical-chemical through electro-optical parameters, which are linked intimately to the performance quality of materials and devices. Two areas, defined by their spatial-resolution qualities, are emphasized: macroscale and microscale measurement technologies. The importance, strengths, and limitations of these techniques are stressed, especially their significance to photovoltaics. Included are several techniques that have been developed specifically to address problems and requirements for photovoltaics. The regime of measurement literally covers arrays through atoms.

Order No.: JA810-001

Photovoltaic silicon produced by thermal plasma/influence of atomic hydrogen on oxygen elimination and passivation of the crystal defects
D. Morvan, I. Cazard-Juvernat, J. Amouroux
(Université P&M Curie)

The photovoltaic properties of the polycrystalline silicon depend on the crystallinity and the purity of the material. The thermal plasma process gives us an alternative way for silicon preparation since it is possible to produce an ultrahigh purity simultaneously with a passivation of crystalline defects and active impurities. We demonstrate the efficiency of the plasma purification process and particularly the influence of the atomic hydrogen in an argon thermal plasma on the photovoltaic properties of silicon. The results of the diffusion lengths measured by photoelectrochemical method show that locally it rises up to 200 μm. We correlate these photovoltaic measurements with the properties of the crystal (defects and purity) by means of measurements by Fourier transform infrared spectroscopy (FTIR) at low temperature (6 K), four probes resistivity technique, scanning electronic microscopy, inductively coupled plasma (ICP) and neutronic activation analyses. We show that the increase of the purity explains the high measured diffusion lengths. Nevertheless the thermal conditions of the crystallization of the silicon, due to the specificity of the plasma, lead to defects such as dislocations for which density is particularly high ($> 10^6$ dis/cm^2).

The results show that chemical reactions between the atomic hydrogen of the plasma and the oxygen of the silicon occur. They decrease the oxygen content in silicon from 3.10^{17} at/cm^3 down to 2.10^{16} at/cm^3 while the residual hydrogen in silicon is close to 2.10^{15} at/cm^3. This passivates the dangling bonds of ultra pure silicon with a high thermal stability up to 1000 K. The objective of this paper is to demonstrate that the hydrogen in the plasma modifies the electronic properties of the material to achieve a very good photocurrent even though the dislocation density of the silicon is very high.

Order No.: JA810-002

Multicrystalline silicon material: Effects of classical and rapid thermal processes
J.C. Muller*, S. Martinuzzi+
*(*Université Aix-Marseille, +CNRS)*

For photovoltaic applications silicon is still the predominant material. Besides monocrystalline Czochralski wafers (Cz-Si), multicrystalline sheets (mc-Si) play an important role in the terrestrial power applications (almost 50%). Large mc-Si ingots (up to 150 kg) are now produced in large scale by the industry using various directional solidification methods in appropriate crucibles (or molds).

However if the crystallographic properties are now quite satisfactory (columnar structure with large grains of more than one cm^2, few dislocations and intra-grains defects), multicrystalline silicon contains larger quan-

tities of impurities than single crystalline silicon which can have detrimental effects on the bulk minority carrier diffusion length ($L_{n,p}$). These impurities, including metals as well as high concentrations of carbon and/or oxygen, can degrade the photovoltaic properties of the solar cells.

Thermal treatments as Gettering, performed in a classical or rapid thermal furnace, studied separately or in conjunction with the doping steps, can limit or avoid the degradation of the bulk diffusion length, but its efficiency is strongly dependent on the presence of these impurities in Si.

Order No.: JA810-003 **© 1998 MRS**

Growth of silicon sheets for photovoltaic applications

R.O. Bell, J.P Kalejs
(ASE Americas Inc.)

Several techniques for the sheet growth of silicon for solar cell substrates are reviewed here. These techniques usually offer an economic advantage over growth in the form of bulk crystals. At least 16 different sheet growth systems have been proposed but only five, that are actively being pursued for commercialization, are discussed here. These include dendritic web, string ribbon, edge-defined film-fed growth (EFG), ribbon growth on a substrate (RGS) and Silicon-Film™. The growth systems and the characteristics of the resulting solar cells are briefly described. A discussion of their current status concludes the review.

Order No.: JA810-004 **© 1998 MRS**

Cadmium-telluride—Material for thin film solar cells

D. Bonnet*, P. Meyers+
*(*ANTEC GmbH, +ITN Energy Systems)*

Due to its basic optical, electronic, and chemical properties, CdTe can become the base material for high-efficiency, low cost thin film solar cells using robust, high-throughput manufacturing techniques. CdTe films suited for photovoltaic energy conversion have been produced by nine different processes. Using n-type CdS as a window-partner, solar cells of up to 16% efficiency have been made in the laboratory. Presently five industrial enterprises are striving to master low cost production processes and integrated modules have been delivered in sizes up to 60 x 120 cm^2, showing efficiencies up to 9%. Stability, health and environmental issues will not limit the commercial potential of the final product. The technology shows high promise for achieving cost levels of 0.5 $\$/W_p$ at 15% efficiency. In order to achieve this goal scientists will have to develop a more detailed understanding of defect chemistry and device operation of cells, and engineers will have to develop methods for high throughput manufacturing.

Order No.: JA810-005 **© 1998 MRS**

Advances in amorphous silicon photovoltaic technology

D.E. Carlson, K. Rajan, R.R. Arya, F. Willing, L. Yang
(Solarex)

With the advent of new multijunction thin film solar cells, amorphous silicon photovoltaic technology is undergoing a commercial revival with about 30 megawatts of annual capacity coming on line in the next year. These new a-Si multijunction modules should exhibit stabilized conversion efficiencies on the order of 8%, and efficiencies over 10% may be obtained in the next several years. The improved performance results from the development of amorphous and microcrystalline silicon alloy films with improved optoelectronic properties and from the development of more efficient device structures. Moreover, the manufacturing costs for these multijunction modules using the new large-scale plants should be on the order of $1 per peak watt. These new modules may find widespread use in solar farms, photovoltaic roofing, as well as in traditional remote applications.

Order No.: JA810-006 **© 1998 MRS**

Crystalline silicon thin films: A promising approach for photovoltaics?

A. Slaoui*, R. Monna*, J. Poortmans+, T. Vermeulen+, O. Evrard+, K. Said+, J. Nijs+
*(*UPR 292 CNRS, +IMEC)*

In this paper we review the achievements in the field of silicon crystalline thin film solar cells and correlate these with the different types of growth techniques and substrates. As a starting point we discuss the characteristics of photovoltaic devices based on the use of highly doped monocrystalline substrates as mechanical carrier for the thin films. These films are epitaxially deposited from the gas (CVD) or liquid phase (LPE). The comparison of both techniques is extended to growth on defective silicon substrates, i.e., multicrystalline wafers or silicon ribbons. The intrinsic grain boundary recombination activity in the thin films is assessed as a function of the deposition technique. Bulk passivation by hydrogenation considerably improves the recombination properties. The optimization of the hydrogen passivation conditions is looked at in conjunction with the used surface passivation process. This review is completed with the approaches to realize thin film cells on non-silicon substrates, including recrystallization in solid and liquid phases.

Order No.: JA810-007 **© 1998 MRS**

COMMUNICATIONS

Synthesis of Y-Ba-Cu-O films with EDTA complexes assisted by excimer laser ablation

N. Tanaka, H. Wakabayashi, S-I. Mochizuki, S. Ohshio, H. Saitoh
(Nagaoka University of Technology)

Y-Ba-Cu-O films were prepared on yttria stabilized zirconia polycrystalline substrate using KrF excimer laser irradiation with EDTA complexes as a target material. The results of x-ray θ-2θ scan showed that the films grown above 750°C were preferentially oriented with c-axis normal to the substrate. The value of full width at half maximum of (005) reflection reduced from 4.4° to 2.1° with an increase in the laser power density in a range between 0.8 and 3.8 J/cm^2. The transcription of the compositional ratio was improved by approaching the target-substrate distance from 45 to 20 mm. The films obtained at 800°C consisted of many islands showing well-developed spiral growth, suggesting that the droplets of metal-EDTA complexes do not form random oriented crystalline particles on the surface of the substrate.

Order No.: JA810-008 **© 1998 MRS**

Formation and catalytic activity of amorphous $Ni_{50}Pd_{40}Si_{10}$ alloy powder by mechanical alloying

H.F. Zhang, J. Li, Q.H. Song, Z.Q. Hu
(Academia Sinica)

Amorphous $Ni_{50}Pd_{40}Si_{10}$ alloy powder was prepared by mechanical alloying. The surface states and catalytic activities of amorphous and crystalline $Ni_{50}Pd_{40}Si_{10}$ alloy powder pretreated with HF solution were studied. The results show that amorphous Ni-Pd-Si alloy powder was easily prepared by mechanical alloying. After treating with HF solution, the number of Pd atoms on the surface of amorphous Ni-Pd-Si powder was obviously higher than that of the crystalline alloy and catalytic activity of amorphous Ni-Pd-Si powder was three times higher than that of the crystalline one.

Order No.: JA810-009 **© 1998 MRS**

Electrorheological fluids using Bi-dispersed particles

W. Wen, W.Y. Tam, P. Sheng
(The Hong Kong University of Science and Technology)

We report very large enhancement of static yield stress for electrorheological fluids by adding ferroelectric nanoparticles of lead zirconate titanate (PZT) or lead titanate ($PbTiO_3$) to ER fluids consisting of 50 μm glass spheres. It is found that the enhancement peaks at certain nanoparticle/microparticle ratios for fixed solid/liquid volume fractions. The results are explained by calculations using an effective medium approach, based on the physical picture that the nanoparticles modify the properties of the liquid and solid components.

Order No.: JA810-010 **© 1998 MRS**

Preparation of free-standing diamond films by a two-step-growth method

Q.H. Fan, E. Pereira, J. Grácio
(University of Aveiro)

A two-step-growth method is presented to prepare free-standing diamond films using copper substrate. The method includes a short pre-growth, for obtaining non-continuous but sufficient nucleation, followed by a quick ramp down in plasma power and temperature for stress release, and a final growth. With this method, the common problem of film cracking is avoided and the possibility of preparing large area, free-standing diamond film is demonstrated.

Order No.: JA810-011

ARTICLES

Control of epitaxial growth orientation in $YBa_2Cu_3O_{7-\delta}$ films on vicinal (110) $SrTiO_3$ substrates

D.S. Linehan, E.P. Kvam, L. Hou, M.W. McElfresh
(Purdue University)

Films of $YBa_2Cu_3O_{7-\delta}$ (YBCO) were grown on (001), exact and vicinal (110), and (111) $SrTiO_3$ single crystal substrates by pulsed laser deposition, and evaluated by x-ray diffraction and scanning force microscopy (AFM). It was observed that the YBCO was always epitaxially aligned to the substrate with the [001] (c-axis) parallel to a substrate cube axis direction. For the exact (001), (110), and (111) surfaces, there were one, two, and three orientations, respectively. For the vicinal (110) surfaces, however, there was usually only one discernible c-axis orientation, corresponding to a single {013} film surface orientation. The reduction of the (110) surface two-fold symmetry by use of a vicinal substrate thus allowed controlled growth of a YBCO single crystal with an inclined c-axis orientation.

Order No.: JA810-012

Relation between internal boundaries and critical current in textured $YBa_2Cu_3O_{7-\delta}$: Transmission electron microscopy observations

A. Khalfi*, G. Trolliard*, B. Soulestin*, D.S. Smith*, J.P. Bonnet*, D. Bourgault+, R. Tournier+
*(*ESA-CNRS 6015, +UPR CNRS 9033)*

The local critical current (I_c) at 77 K measured at the mm scale in a 60 mm long sample of $YBa_2Cu_3O_{7-\delta}$ prepared by a melting zone process is correlated to the microstructure. Lower values of I_c (< 20 A) were obtained in a part of the sample which optical microscope examination showed to be generally polycrystalline. In contrast the rest of the sample, consisting mostly of large textured domains, gave values for I_c of 120 A and above. Transmission electron microscope observations revealed that the textured domains contain internal boundaries. Depending on the scale of observation the misorientation angles across the boundaries could vary from a few tenths of a degree up to a few degrees. This seems characteristic for boundaries in textured material which allow strong coupling of the superconducting current across themselves.

Order No.: JA810-013

Floating zone partial melting and solidification of SmBCO superconductor under low oxygen partial pressure

M. Sumida*, S. Matsuoka+, Y. Shiohara+, T. Umeda*
*(*The University of Tokyo, +SRL-ISTEC)*

Microstructure control of SmBCO superconductor was carried out using the floating zone partial melting and solidification method under 0.01 atm oxygen partial pressure of which atmosphere is preferable to obtain a crystal with stoichiometric SmBCO. The growth rate, initial composition and addition of small amount of platinum dependences on the microstructure formations of the (Sm211+L) mixture during melting and the Sm123 or Sm123/211 during solidification were investigated. Furthermore, superconductive properties of the solidified Sm123/211 were measured by SQUID after appropriate oxygen annealing. Estimated critical current density of the single crystalline Sm123/211 was 3.6×10^4 A/cm^2 at 77 K, 1 T.

Order No.: JA810-014

The effect of excess neodymia on the grain growth of $Nd_{1+x}Ba_{2-x}Cu_3O_y$ solid solutions

R.B. Rogenski*, K.H. Sandhage*, A.L. Vasiliev+, E.P. Kvam+
*(*The Ohio State University, +Purdue University)*

The grain growth of dense, fine-grained $Nd_{1+x}Ba_{2-x}Cu_3O_y$ (x = 0.1–0.4) specimens has been examined in pure O_2(g) at 938°C and 967°C. No detectable change in average grain size was observed for $Nd_{1.4}Ba_{1.6}Cu_3O_y$ within 72 h at 967°C; however, a significant increase in average grain size developed between 18 and 24 h at 967°C for $Nd_{1.3}Ba_{1.7}Cu_3O_y$, and within 8–12 h at ≤ 967°C for $Nd_{1.2}Ba_{1.8}Cu_3O_y$ and $Nd_{1.1}Ba_{1.9}Cu_3O_y$. Microstructural analyses revealed that sudden changes in average grain size coincided with the formation of relatively large (abnormal) grains. A broadening of the grain size distribution was also observed. TEM analyses revealed that grain boundaries were free of second phases. The possible role of anisotropy in grain boundary energy and/or mobility on grain growth is discussed.

Order No.: JA810-015

Probing of microvoids in high-rate deposited a-Si:H thin films by variable energy positron annihilation spectroscopy

X. Zou*, D.P. Webb*, Y.C. Chan*, Y.W. Lam*, Y.F. Hu+, S. Fung+, C.D. Beling+
*(*City University of Hong Kong, +The University of Hong Kong)*

In this paper, positron annihilation measurements have been carried out on a-Si:H thin films deposited by PECVD at high and low rates by means of the variable energy positron beam Doppler-broadening technique. The depth profiles of microvoids in the films grown under different conditions have been determined. We found a smaller void fraction in the surface region of all films compared to the bulk, and a smaller void fraction in low rate than in high growth rate films. By plotting S and W parameters in the (S, W) plane, we have shown that the vacancies in all of the high-rate and low-rate deposited intrinsic samples, and in differently doped low-rate samples, are of the same nature, although there appears to be a higher density of defects in the boron than phosphorus doped films. The depth profiles of the microvoid-like defects in the a-Si:H films are extracted by use of the VEPFIT program.

Order No.: JA810-016

Highly-adherent diamond coatings deposited onto WC-Co cemented carbides via barrier interlayers

J.M. Lopez*, V.G. Babaev+, V.V. Khvostov+, J.M. Albella#
*(*Cayetano Heredia University, +Lomonosov Moscow State University, #Instituto Ciencia de Materiales)*

Diamond coatings have been deposited by plasma enhanced chemical vapor deposition (PECVD) onto WC-Co cemented carbides by use of specially developed barrier interlayers, well compatible with cemented carbides. The barrier interlayer comprises a Ti-based layer adjacent to the substrate, which completely prevents both substrate decarburization and Co diffusion from the substrate, and a diamond-bonding layer needed to obtain high adhesion to the diamond coating. The diamond-bond layer is obtained by seeding the surface with nano-grained diamond particles by laser ablation. Diamond deposition under controlled parameters allows to obtain fine-grained and uniform diamond coatings. The diamond coating obtained in this way has a high adhesion to the cemented carbide substrate due to the enhanced interaction through the nano-grained diamond interlayer.

Order No.: JA810-017

Porous NiTi alloy prepared from elemental powder sintering

B. Li, L. Rong, Y. Li
(Chinese Academy of Sciences)

An elemental powder sintering (EPS) technique has been developed for the synthesis of porous NiTi alloy, in which Ni and Ti powders are used as the reactants and TiH_2 powder is added as a pore-forming agent and active agent. Effects of various experimental parameters (sintering temperature, sintering time and TiH_2 content) on the porosity, pore size and pore

distribution as well as phase composition in experimental alloys are investigated. It is found that in order to avoid the formation of carcinogenic pure Ni phase, the porous NiTi alloy should be synthesized over a temperature of 1223 K. This gives NiTi as the main phase without any elemental phase. Substitution of Ti by TiH_2 is more economic and more favorable to obtain homogeneous porous NiTi alloy. A proper selection of initial powders, ball-milling, pressing and sintering process makes it possible to achieve the porous NiTi alloy with desired properties.

Order No.: JA810-018 **© 1998 MRS**

Young's modulus and tensile strength of CuNi(Mn) thin films on polyimide foils by tensile testing

F. Macionczyk, W. Brückner, W. Pitschke, G. Reiss
(Institute of Solid State and Materials Research Dresden)

Force-strain curves were measured for 1.0 μm and 1.5μm thick $Cu_{0.57}Ni_{0.42}Mn_{0.01}$ films on 8 μm thick polyimide foils by tensile testing. By separating the force working on the polyimide foil from that working on the metal polyimide compound, stress-strain curves for the CuNi(Mn) films were obtained. Young's modulus and tensile strength were determined for as-deposited and annealed [350°C, 1 h, N_2/H_2(5 vol. %) atmosphere] films by this method. Crack propagation starts at the end of the elastic region at 0.2 to 0.7% strain depending on the film thickness and the thermal treatment. The cracking behavior is described by a steady-state approximation.

Order No.: JA810-019 **© 1998 MRS**

Wetting and interface microstructure between Sn-Zn binary alloys and Cu

K. Suganuma*, K. Niihara*, T. Shoutoku+, Y. Nakamura+
*(*Osaka University, +National Defense Academy)*

Sn-Zn binary alloys have been examined as a lead-free solder. Zn distributes in a Sn matrix as platelets. The hypoeutectic alloys show two endothermic peaks in DTA, which correspond to the eutectic and the liquidus temperatures. Three reaction layers are formed at the Sn-Zn/Cu interface without containing Sn, the thick γ-Cu_5Zn_8 adjacent to the solder, the thin β'-CuZn in the middle and the thinnest layer adjacent to Cu. Although many non-wetting regions and voids are formed at the interface because of poor wetting, soldering at 290°C can form a rigid interface and tensile strength reaches about 40 MPa.

Order No.: JA810-020 **© 1998 MRS**

Fabrication of surface barrier layer capacitor on $BaTiO_3$-based composite containing particulate SiC

H.J. Hwang, K. Niihara
(Osaka University)

Surface reoxidized-type barrier layer (BL) capacitors were prepared by hot-pressing $BaTiO_3$/SiC powder mixture in argon atmosphere and subsequently oxidizing the semiconducting $BaTiO_3$-based composites with fine SiC particles. Dielectric properties, such as apparent relative dielectric constant, dielectric loss tangent and Curie temperature, were investigated as a function of SiC content and oxidation procedures. Incorporating SiC particles into $BaTiO_3$ matrix, a thin surface insulating layer was formed, which was getting thinner with an increase in the SiC content. BL capacitors showing higher capacitance than 3×10^2 nF/cm^2 could be successfully fabricated. The results were discussed on the base of a high resolution transmission electron microscope (HRTEM) study. Thin oxidized layer and the resulting high capacitance were associated with the depression of oxygen diffusion due to the presence of intergranular SiC particles. The thickness of the surface layer and some dielectric properties could be controlled by the SiC content as well as oxidation temperature and time.

Order No.: JA810-021 **© 1998 MRS**

Metal ceramic interface toughness I: Plasticity on multiple length scales

J.D. Kiely, D.A. Bonnell
(The University of Pennsylvania)

The fracture toughness of Ni-sapphire interfaces was measured as a function of interfacial embrittlement. Embrittlement was controlled by segregating sulfur to the interface, by limiting the presence of moist air in the test environment, and by altering the distribution of interfacial particulates. Fracture energies scaled with the degree of embrittlement and ranged from 8.5 to 34.2 J/m^2. Scanning probe microscopy revealed four distinct plasticity features, the heights of which ranged from 1 μm to 0.5 nm. Plasticity generation processes are determined based on the variation of feature height and position with fracture energy, allowing features associated with the interface decohesion process to be identified.

Order No.: JA810-022 **© 1998 MRS**

Metal ceramic interface toughness II: Mechanisms of fracture and energy dissipation

J.D. Kiely, D.A. Bonnell
(The University of Pennsylvania)

Based on observations of plasticity on fracture surfaces, we propose two fracture mechanisms for Ni-sapphire interfaces. A brittle-type mechanism is proposed for the decohesion of Ni from sapphire by which cracks advance in increments of 20 nm. When particulates that increase the interface strength are present, debonding occurs at the leading edge of the particulate and unsteady crack advance occurs. Additionally, toughening mechanisms are proposed for each type of plasticity feature observed, and the fracture energy of each mechanism is quantified. Comparison of energy dissipated by these mechanisms with measured fracture energies indicates how the fracture energy of the interface varies with sulfur segregation and environmental embrittlement.

Order No.: JA810-023 **© 1998 MRS**

Annealing of mesoporous silica loaded with silver nanoparticles within its pores from isothermal sorption

W. Cai, L. Zhang, H. Zhong, G. He
(Academia Sinica)

Influences of annealing on the structure of mesoporous silica loaded with silver (Ag) nanoparticles, and on the coarsening of Ag particles within pores of the host were investigated from isothermal sorption. Doping small amount of Ag nanoparticles into pores of silica and subsequent annealing decrease the measured values of specific surface area and pore volume of porous silica significantly. This is attributed to the presence and coarsening of Ag particles within pores or channels between pores, which result in more and more isolated and unmeasured free spaces. The measured value of specific surface area for the doped samples cannot represent the real value, which is, in fact, unable to be measured directly. During additional annealing, Ag particles within silica coarsen mainly according to the mechanism of formation of Ag adatoms on pore wall and diffusion of the adatoms along with pore walls. Only the larger particles located in the larger pores can continuously grow. The smaller particles and those located in the channels or pores with smaller dimension will disappear. The activation energy of the ripening process was estimated to be about 0.60 eV and the migration barrier of Ag adatom on the pore wall of silica is about 0.10 eV.

Order No.: JA810-024 **© 1998 MRS**

Melt infiltration casting of bulk metallic-glass matrix composites

R.B. Dandliker, R.D. Conner, W.L. Johnson
(California Institute of Technology)

The authors describe a technique for melt infiltration casting of composites with a metallic-glass matrix. We made rods 5 cm in length and 7 mm in diameter. The samples were reinforced by continuous metal wires, tungsten powder, or silicon carbide particulate preforms. The most easily processed composites were those reinforced with tungsten and carbon steel continuous wire reinforcement. The $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10.0}Be_{22.5}$ matrix was quenched to a glass after infiltrating the reinforcement. We analyzed the microstructure of the composites by x-ray diffraction and scanning electron microscopy. The measured porosity was less than 3% and the matrix was about 97% amorphous material.

Order No.: JA810-025 **© 1998 MRS**

Tensile testing low density multilayers: Aluminum/titanium

D. Josell*, D. van Heerden+, D. Read*, J. Bonevich*, D. Shechtman#
*(*National Institute of Standards and Technology, +Johns Hopkins University, #Technion)*

Yield stresses, ultimate tensile strengths, and specific strengths of aluminum/titanium multilayer thin films are determined from the results of uniaxial tensile tests. The plasticity in the stress-strain curves, the nature of the fracture surfaces, and the relationship of the yield stress and the bilayer thickness are discussed. Properties are compared with those of other multilayer materials published in the literature.

Order No.: JA810-026

A method for crystallographic texture investigations using standard x-ray equipment

M.D. Vaudin*, M.W. Rupich+, M. Jowett+, G.N. Riley, Jr.+, J.F. Bingert#
*(*National Institute for Standards and Technology, +American Superconductor Corp., #Los Alamos National Laboratory)*

A fast and accurate method has been developed for measuring crystalline texture in homogeneous materials. The method uses a conventional powder x-ray diffractometer capable of θ scans. Two scans are recorded from the sample: first, a high resolution θ-2θ scan is obtained of a Bragg peak whose diffracting planes are normal to the preferred orientation direction; second, a θ scan is obtained using this peak. The θ scan contains the required texture information but the intensities must be corrected for defocussing and absorption to obtain the texture profile. The θ-2θ scan of the Bragg peak is used to make the defocussing correction, and first principles calculations are used to correct for absorption. The theory behind these corrections is presented here. The validity of the technique has been verified by making measurements on untextured alumina. Data obtained from $Bi_2Sr_2Ca_2Cu_3O_{10}$ superconducting tape specimens with this technique are compared with texture data obtained with a four-circle diffractometer.

Order No.: JA810-027

Crystallography and microstructural studies of phase transformations in the Dy_2O_3 system

Y.J. Kim, W.M. Kriven
(University of Illinois at Urbana-Champaign)

The crystallography, microstructures and phase transformation mechanisms in dysprosia (Dy_2O_3) have been studied. The lattice parameters of B and C phases were refined by XRD. The modulated structures and decomposed structures in the CaO-doped samples were characterized by TEM. A new twin was observed in the modulated B phase. Contrary to the previous studies, the B to C transformation was induced by grinding. The A to B transformation was considered to be ferroelastic and the spontaneous strain was calculated. The major driving force for the B (monoclinic) to C (cubic) transformation is suggested to be the release of lattice strains and cation charge repulsions in the B structure, which is analogous to the β (monoclinic) to γ (orthorhombic) transformation in Ca_2SiO_4. This transformation can be displacive, if some conditions are provided to overcome the bonding energy of the interlayer oxygens in the B structure.

Order No.: JA810-028

Preparation and characterization of $SrBi_2Nb_2O_9$ thin films made by polymeric precursors

S.M. Zanetti*, E.R. Leite*, E. Longo*, J.A. Varela+
*(*UFSCar, +UNESP)*

A polymeric precursor solution was employed in preparing $SrBi_2Nb_2O_9$ (SBN) powder and thin films dip coated onto Si(100) substrate. XRD results show that the SBN perovskite phase forms at temperatures as low as 600°C through an intermediate fluorite phase. This fluorite phase is observed for samples heat treated at temperatures of 400 and 500°C. After heat treatment at temperatures ranging from 300 to 800°C, thin films showed to be crack free. Grazing incident angle XRD characterization also shows the occurrence of the fluorite intermediate phase for films. The thickness of films, measured by MEV, were in the order of 80–100 nm.

Order No.: JA810-029

Mechanical properties from instrumented indentation: Uncertainties due to tip-shape correction

L.E. Seitzman
(Naval Research Laboratory)

The quality of hardness H and indentation modulus E* measurements from instrumented indentation is investigated. Load-displacement data from glass and sapphire are obtained by Vickers indentation and converted to H and E* through a series of equations, including those for tip-shape correction. The quality of H and E* is determined by calculating the statistical uncertainty at each step and propagating the uncertainty to the next step. Conventional tip-shape corrections, assuming either constant hardness or constant modulus, introduce significant errors in H and E* when single, continuous correction functions are used. Piece-wise corrections functions are shown to improve the quality of H and E*. This investigation demonstrates the importance of calculating and propagating uncertainty at each step when converting instrumented indentation load-displacement data to mechanical properties.

Order No.: JA810-030

Effects of Nd_2O_3 on the microwave dielectric properties of $BiNbO_4$ ceramics

W. Choi*, K-Y. Kim*, M-R. Moon+, K-S. Bae+
*(*Korea Institute of Science and Technology, +The University of Suwon)*

Effects of Nd substitution with Bi on the microwave dielectric properties of $BiNbO_4$ were studied. $Bi_{1-x}Nd_xNbO_4$ ceramics sintered at 920~980°C consisted of orthorhombic and triclinic phases. The amount of triclinic phase increased with the increase in the Nd content, x, and sintering temperature. The apparent density and the dielectric constant decreased with the Nd content, but increased with sintering temperature, reached the peak values at 960°C and then rapidly decreased. The $Q \times f_0$ value was between 11,000~13,000 GHz over all sintering temperatures for $x < 0.05$, but for $x > 0.05$ it reached the peak value at 950°C and then rapidly decreased. The temperature coefficient of resonance frequency increased in the positive direction with the Nd content and showed the minimum value of -1.82 ppm/°C for $x = 0.025$ sintered at 940°C. However it rapidly increased in the negative direction for sintering temperature over 960°C.

Order No.: JA810-031

Synthesis and characterization of Y_2O_3: Eu^{3+} powder phosphor by a hydrolysis technique

Y.D. Jiang*, Z.L. Wang*, F. Zhang+, H.G. Paris+, C.J. Summers+
*(*Georgia Institute of Technology, +Georgia Tech Research Institute)*

A forced hydrolysis technique is used for preparing Y_2O_3:Eu^{3+} powders at low processing temperatures. The technique uses yttrium oxide, europium oxide, nitric acid and urea, and has the potential for large-scale production for industrial applications. Several experimental conditions have been examined to optimize the luminescence efficiency. The best result was found to be at 2 mol% Eu doping and a 2 hour firing of 1400°C. Microstructural information provided by x-ray diffraction, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) have been applied to interpret the observed luminescent properties.

Order No.: JA810-032

Changes in optical transmittance and surface morphology of AlN thin films exposed to atmosphere

Y. Watanabe, Y. Sakuragi, Y. Amamoto, Y. Nakamura
(National Defense Academy)

Aluminum nitride (AlN) thin films have been prepared by ion-beam assisted deposition (IBAD) method, and the influence of exposure to different atmosphere on optical transmittance and surface morphology has been studied. AlN films have been prepared with the nitrogen ion beam energy of 0.1, 0.2 or 1.5 keV. Synthesized films have been exposed to the following conditions, (1) laboratory air (RT and 40–60% RH), (2) saturated humidity air (RT and 80–90% RH), and (3) elevated temperature air (100 °C and 10–20% RH). Optical transmission spectrum in the wavelength region from 190 to 2200 nm has been measured by UV-visible spectrometer every week. Surface morphology of the films has been

observed with an optical microscope (OM) and phase identification has been performed by thin film x-ray diffraction (TFXRD). The optical transmittance has not changed drastically after exposure both to the laboratory air and the saturated humidity air for 60 weeks and after exposure to the elevated temperature air for 48 weeks. Observations by OM showed that round shaped substances were formed on the film surfaces after exposure to the atmosphere and the size of the substances on the film surface exposed to saturated humidity air is much larger than those on the surface exposed to other atmosphere. The results of TFXRD revealed that the AlN diffraction peaks have gradually decreased with exposure time but any new phase due to reaction products has not been detected for the samples exposed to the laboratory air, the saturated humidity air, nor the elevated temperature air. From the present results, it is concluded that the IBAD AlN films can be applied in low humidity air without losing high transparency up to 60 weeks and the films prepared with 1.5 keV ion beam show better durability than the films prepared with 0.1 or 0.2 keV ion beam for exposure to the saturated humidity air.

Order No.: JA810-033 **© 1998 MRS**

Textures of thin copper films

W-M. Kuschke*, A. Kretschmann*, R-M. Keller*, R.P. Vinci+, C. Kaufmann#, E. Arzt*

*(*Max-Planck Institut für Metallforschung, +Stanford University, #Zentrum für Mikrotechnologien)*

The textures of thin copper films were determined quantitatively by measuring (111) pole figures with x-ray diffraction. Measurements were performed on a variety of samples, differing in copper film thickness and deposition technique, diffusion barrier material and the presence or absence of a cap layer. Texture changes due to an annealing treatment were also recorded and correlated with stress measurements by the wafer-curvature technique. It is found that the deposition method (PVD vs. CVD) has a strong effect on texture, barrier layer effects range from negligible to significant depending on the barrier material, and the effect of a cap layer is insignificant.

Order No.: JA810-034 **© 1998 MRS**

Preparation and size evaluation of nanometer gadolinium powder

Y.Z. Shao*, C.H. Shek+, J.K.L. Lai+

*(*Zhongshan University, +City University of Hong Kong)*

Nanometer-size gadolinium powders (nm-Gd) have been prepared by means of evaporation-condensation of Gd atoms within inert gas atmosphere. Microscopic analyses, based on measurements of small angle x-ray scattering (SAXS), x-ray diffraction (XRD), Raman scattering spectrum (RSS), and observation with transmission electron microscope (TEM), have been carried out in order to evaluate the size and size distribution of the as-prepared nm-Gd powder. It turns out that the size distribution function of nm-Gd powder agrees very well with the distribution function of Rayleigh instead of logarithmic distribution. The mean size d of nm-Gd powders bears a linear relationship with the logarithm of the pressure p of the inert gas atmosphere as follows: $d = a + b \cdot \ln p$. A discussion concerning the influence of particle size of nm-Gd powder on nanostructured material parameters such as the size distribution, specific surface area and the percentage of interface atoms have been given in detail.

Order No.: JA810-035 **© 1998 MRS**

Monodisperse magnetic nanoparticles: Preparation and dispersion in water and oils

S. Lefebure, E. Dubois, V. Cabuil, S. Neveu, R. Massart

(Université Pierre et Marie Curie)

Nanometric maghemite and cobalt ferrite particles are chemically synthesized. The process produces particles polydisperse in size. The positive charges of their surface allow them to disperse in aqueous acidic solutions and to obtain dispersions stabilized through electrostatic repulsions. Increasing acid concentration (in the range 0.1 to 0.5 mol.L^{-1}), interparticles repulsions are screened, phase transitions are induced. Using this phenomenon, we describe a two-step size sorting process, in order to get significant amounts of nanometric monosized particles (with diameters monitored typically between 6 and 13 nm). As the surface of the latter is not modified by the size sorting process, usual procedures are used to disperse them in several aqueous or oily media.

Order No.: JA810-036 **© 1998 MRS**

Composition tunable memory and threshold switching in $Al_{20}As_xTe_{80-x}$ semiconducting glasses

S. Murugavel, S. Asokan

(Indian Institute of Science)

I-V studies indicate a composition dependent switching behavior (Memory or Threshold) in bulk $Al_{20}As_xTe_{80-x}$ glasses, which is determined by the coordination and composition of aluminum. Investigations on temperature and thickness dependence of switching and structural studies on switched samples suggest thermal and electronic mechanisms of switching for the memory and threshold samples respectively. The present results also show that these samples have a wider composition range of threshold behavior with lower threshold voltages compared to other threshold samples.

Order No.: JA810-037 **© 1998 MRS**

Properties of polyimide shells made using vapor phase deposition

E.L. Alfonso, S.H. Chen, R.Q. Gram, D.R. Harding

(University of Rochester)

Hollow polyimide shells, to be used in inertial confinement fusion experiments, were fabricated by co-depositing monomer precursors onto spherical mandrels. Polyimide shells with 700 μm to 950 μm diameters and 4 μm to 13 μm wall thicknesses were produced. The shell wall shrunk 20%–30% due to imidization. Burst and buckle pressure tests on these shells yielded estimated mechanical strength properties: ~15 GPa elastic modulus and ~300 MPa tensile strength. The permeability of D_2 through polyamic acid at 298 K was 7.4×10^{-17} mol·m/m^2·Pa·s and increased to 6.4×10^{-16} mol·m/m^2·Pa·s upon curing the shell to 150°C. The permeability of D_2 at 298 K through vapor-deposited polyimide flat films was 240 times greater than through polyamic acid.

Order No.: JA810-038 **© 1998 MRS**

MoS_2 deposited by ion beam assisted deposition: 2H or random layer structure?

D.N. Dunn, L.E. Seitzman, I.L. Singer

(Naval Research Laboratory)

The structure of MoS_2 films grown by ion beam assisted deposition is investigated using transmission electron microscopy. Films consist of stacks of S-Mo-S planes with an [001] texture; however, three-dimensional crystal symmetry is disrupted by a high density of planar defects. Selected area electron diffraction patterns show (hk0) and (001) reflections, features similar to a random layer structure, as well as diffuse (103) reflections. It is suggested that these films do not have a true random layer structure, but rather a two dimensional structure formed by non-random in-plane translations.

Order No.: JA810-039 **© 1998 MRS**