JMR Abstracts

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

Abstracts for January 1997 Journal of Materials Research *Volume 12, Number 1*

TABLE OF CONTENTS

ABSTRACTS

COMMUNICATIONS

The intercalation of Ar into C_{60} films
G.E. Gadd, P.J. Evans, S. Moricca, M. James
(Australian Nuclear Science and Technology Organisation)

In this letter we communicate how unimplanted and implanted films (Y and Au implanted) of C_{60}, when hot isostatically pressed (HIPed) at a pressure of 170 MPa (1.7 kbar) of Ar and temperatures of 300 or 400°C, show substantial uptake of Ar into the film. Rutherford backscattering (RBS) provides an effective method for elucidating film composition, showing the films to have ~1.5 at.% of Ar, consistent with a stoichiometry close to Ar_1C_{60}. The Ar was found to diffuse from the films when these were held in a vacuum at 300°C. It could subsequently be reincorporated into the film by re-HIPing, with uptake of a similar amount of Ar. IR spectroscopy showed that the C_{60} IR absorptions remain unchanged throughout uptake of the Ar and its subsequent loss during heating at 300°C. It appears that the Ar is trapped interstitially in the films outside the C_{60} cages.

Order No.: JA701-001

Martensite transition in rapidly solidified Ti_3Al-2Nb alloy
R. Xu, Y.Y. Cui, D.M. Xu, D. Li, Q.C. Li, Z.Q. Hu
(Academia Sinica)

The microstructure of rapidly solidified Ti_3Al-2Nb alloy consists of an ordered orthorhombic α''_0 phase. In the foil samples for transmission electron microscopy prepared by chemical thinning, the $\alpha''_0 \rightarrow \beta_0$ transformation took place due to the charging of hydrogen during thinning. The $\beta_0 \rightarrow \alpha''_0$ transition in the rapidly solidified alloy can be explained by a shape deformation mechanism and the β_0/α''_0 interface (habit) plane is near to $\{\bar{3}34\}_{\beta_0}$. The orientation relationships between the β_0 and α''_0 phases are $[\bar{1}10]_{\beta_0} \parallel [001]_{\alpha''_0}$, and $(1\bar{1}0)_{\beta_0} \wedge (010)_{\alpha''_0} = (001)_{\beta_0} \wedge (100)_{\alpha''_0} = 4.6°$.

Order No.: JA701-002

The cross-over of preferred orientation in TiN film growth: A real time x-ray scattering study
J.H. Je*+, D.Y. Noh*#, H.K. Kim§, K.S. Liang*
*(*Exxon Corporate Research, +Pohang University of Science & Technology, #Kwangju Institute of Science & Technology, §Pusan University)*

The orientational cross-over phenomena in a rf sputtering growth of TiN films were studied in a real-time synchrotron x-ray scattering experiment. Following the initial random nucleation and growth stage, the growth was dominated by the grains with the (002) planes aligned with the substrate surface. Surprisingly, at later stages, the grains with the (002) growth front tilted away from the surface by about 60° became dominant. The tilting of the growth front resulted in a faceted surface topology that was confirmed by an *ex situ* AFM study. Our x-ray results suggest that the cross-over was driven by the competition between the surface and the strain energy.

Order No.: JA701-003

Stabilization of lead lithium iron tungstate with adding barium titanate
C-H. Lu, B-K. Fang
(National Taiwan University)

The kinetics of formation, phase stabilities and dielectric constants of $Pb(Li_{1/4}Fe_{1/4}W_{1/2})O_3$ and $BaTiO_3$-added $Pb(Li_{1/4}Fe_{1/4}W_{1/2})O_3$ have been compared. The addition of 2 mol% $BaTiO_3$ in $Pb(Li_{1/4}Fe_{1/4}W_{1/2})O_3$ was confirmed to promote the complete formation of the perovskite phase at 700°C. Also the thermal stability of the perovskite phase was significantly enhanced, which resulted in an increase of the dielectric permittivity.

Order No.: JA701-004

Electroluminescence from amorphous-silicon-based switching devices
V.A. Kuznetsov, D. Haneman
(University of New South Wales)

Information about the nature of formed switching structures in amorphous hydrogenated silicon has been found from features of the electroluminescence spectrum. Several small peaks have been detected in the region of 1.8 to 2.4 eV, on the shoulder of the usual a-Si:H peak at 1.32 eV. The intensity of the latter is found to depend on the voltage across the film rather than the current. The EL fine-structure can be explained in terms of a model of the switching where there are small metal-rich inclusions in the film. The apparent dependence of EL intensity on voltage is explained in terms of this model with parallel current paths in the structure.

Order No.: JA701-005

ARTICLES

Influence of powder characteristics on the properties of green compacts of Bi-2212 powders
D.W. Yuan, M.D. Aesoph, J. Kajuch
(Concurrent Technologies Corporation)

With the growing potential for use of Bi-2212 powders in high temperature superconducting applications, it is important to understand the processing characteristics of the material. To meet this need, the present work established the relationship between confining pressure and green

density for powders of different particle sizes and morphologies. Mechanical properties, including elastic and plastic behavior, of the resulting green compacts were also measured as a function of relative density. Particle size and size distribution are shown to have a significant impact on the properties of interest. The implication of such findings are discussed with respect to the powder-in-tube process for making high temperature superconducting wire and tape.

Order No.: JA701-006

X-ray diffraction line broadening effects in $MBa_2Cu_3O_{7-\delta}$ (M = Y, Gd) thin films

P. Scardi*, F.C. Matacotta+, V.I. Dediu#, L. Correra§
*(*Università di Trento, +CNR-ISM, #HTS Laboratory-ICTP, §CNR-LAMEL)*

X-ray diffraction Line Profile Analysis (LPA) has been carried out on a set of superconducting thin films of $MBa_2Cu_3O_{7-\delta}$ (MBCO, M = Y, Gd), deposited by pulsed and continuous PVD techniques on different single-crystal substrates. The choice of appropriate deposition conditions, substrates and buffer layers, promoted a high degree of [00*l*] preferred orientation, leading to a well-defined columnar grain morphology in the MBCO films. Under such conditions, the LPA of diffraction patterns collected with the widely spread Bragg-Brentano geometry, gives a detailed information on the distributions of coherent scattering domain (crystallite) size and microstrain along the [00*l*] growth direction; considering the particular MBCO film microstructure, the mean crystallite size ($\overline{M}$) can be regarded as the mean distance between extended planar defects parallel to the film surface.

The significance of $\overline{M}$ goes beyond a merely statistical value. As long as the morphology of the films is similar, $\overline{M}$ is found to be strictly connected with the average microstrain by a simple proportionality relation. Moreover, the correlation extends to important superconducting transport parameters, like the transition width ΔT_c. These regular behaviors are irrespective of deposition techniques, substrate and film materials, and are a clear indication of some fundamental relation between the defects and the overall properties of the films.

Order No.: JA701-007

Precipitate size refinement by CeO_2 and Y_2BaCuO_5 additions in directionally solidified $YBa_2Cu_3O_7$

N. Vilalta, F. Sandiumenge, S. Piñol, X. Obradors
(Institut de Ciència de Materials de Barcelona-C.S.I.C.)

Directional solidification of $YBa_2Cu_3O_7$ has been carried out through a Bridgman technique and the influence of Y_2BaCuO_5 and CeO_2 additives on the size of Y_2BaCuO_5 precipitates has been investigated. It is demonstrated in this work that the most efficient procedure to reduce the size of the Y_2BaCuO_5 precipitates is to increase the concentration of nucleation centers present in the peritectic decomposition of $YBa_2Cu_3O_{7-x}$. A small concentration (0.3-1 wt.%) of CeO_2 has a strong influence on the solidification process and on the size of Y_2BaCuO_5 precipitates. It is shown that when CeO_2 is added, further refinement of the size of precipitates results from the formation of nanometric Y_2O_3 particles which further enhance the multinucleation effect. We have also observed that coarsening effects are avoided with CeO_2 additives.

Order No.: JA701-008

Oxygen content and inhomogeneity effects on the electrical properties of $YBa_2Cu_3O_y$ thin films

R. Aguiar, F. Sánchez, M. Varela
(Universitat de Barcelona)

Oxygen content is a very important factor influencing the electrical properties of $YBa_2Cu_3O_y$. In this work the electrical properties of laser deposited $YBa_2Cu_3O_y$ thin films on $LaAlO_3$(100), in the whole range $6 \le y \le 7$, are studied. An electrical network model, which randomly assigns oxygen contents and R(T) characteristics to the different elements in the circuit according to an arbitrary distribution, is used to analyze several features in the measured R(T) characteristics as a function of oxygen homogeneity. The model takes into account both short-range and long-range oxygen inhomogeneities. Good agreement between estimated oxygen contents from x-ray diffraction data in our samples and the average oxygen contents used to reproduce their R(T) characteristics is found. The model points out that oxygen homogeneity is very important in order to get the best and reproducible properties, and for conduction and superconductivity analysis through the shape or derivatives of R(T) characteristics.

Order No.: JA701-009

Micromechanical and tribological characterization of doped single-crystal silicon and polysilicon films for microelectromechanical systems devices

B. Bhushan, X. Li
(The Ohio State University)

Microelectromechanical systems (MEMS) devices are made of doped single-crystal silicon, LPCVD polysilicon films and other ceramic films. Very little is understood about tribology and mechanical characterization of these materials on micro- to nanoscales. Micromechanical and tribological characterization of p-type (lightly boron-doped) single-crystal silicon (referred to as "undoped"), p^+-type (boron doped) single-crystal silicon, polysilicon bulk and n^+-type (phosphorous doped) LPCVD polysilicon films have been carried out. Hardness, elastic modulus and scratch resistance of these materials were measured by nanoindentation and microscratching using a nanoindenter. Friction and wear properties were measured using an accelerated ball-on-flat tribometer. It is found that the undoped silicon and polysilicon bulk as well as n^+-type polysilicon film exhibit higher hardness and elastic modulus than the p^+-type silicon. The polysilicon bulk and n^+-type polysilicon film exhibit the lowest friction and highest resistance to scratch and wear followed by the undoped silicon and with the poorest behavior of the p^+-type silicon. During scratching, the p^+-type silicon deforms like a ductile metal.

Order No.: JA701-010

Low-load indentation behavior of HfN thin films deposited by reactive rf sputtering

R. Nowak, C.L. Li, S. Maruno
(Nagoya Institute of Technology)

Deformation of HfN thin films deposited by reactive sputtering method on silicon and alumina substrates has been investigated using depth-sensing indentation. The experiments performed in a low load range (2–50 mN) revealed that even extremely shallow indentations were affected by elastic/plastic response of the substrate. The analysis of the shape of the indentation load-depth hysteresis loops and of conventional hardness data was supplemented by considerations based on the recently proposed energy principle of indentation.

Order No.: JA701-011

Comparison of C_2F_6 and FASi-4 as fluorine dopant sources in plasma enhanced chemical vapor deposition fluorinated silica glass films

W.S. Yoo, R. Swope, B. Sparks, D. Mordo
(Novellus Systems, Inc.)

Fluorine doping of silicon dioxide films in tetraethylorthosilicate (TEOS)-based plasma enhanced chemical vapor deposition (PECVD) processes was investigated using two fluorine dopant sources, C_2F_6 and 1,2 bis[methyldifluorosilyl]ethane (FASi-4). As much as TEOS-based undoped silica glass (USG) films display improved step coverage over silane-based USG films, it was suspected that fluorinated silica glass (FSG) films deposited using the relatively new TEOS-based fluorine source FASi-4 might have improved gap fill capabilities as compared to FSG films deposited using gas-based C_2F_6 fluorine sources. The physical properties and intermetal gap filling capabilities of FSG films deposited using FASi-4 as a fluorine dopant source were compared with the properties of FSG films deposited using C_2F_6 as a fluorine source. Fluorine dopant levels in the films were found to be linear functions of C_2F_6/TEOS and FASi-4/TEOS ratios. The RI, film stress and gap fill capability were found to be strongly

dependent on the Si-F content in the film regardless of dopant source reagents. Improved gap fill characteristics were observed in films doped with FASi-4 at a given Si-F/Si-O% as compared to C_2F_6-based FSG films. Dopant source dependence of doping characteristics, physical properties and gap filling capability of FSG films is reported.
Order No.: JA701-012

Surface short-range ordering of Cu_3Au above T_c in the topmost 80 A of an (001) face
M. Kimura*, J.B. Cohen*, S. Chandavarkar+, K. Liang#
*(*Northwestern University, +University of Tennessee, #Exxon Corporate Research Laboratory)*

The short-range order in the near surface region of the Cu_3Au (001) face was investigated above the critical temperature by glancing-incidence x-ray diffraction, measuring the diffuse intensity throughout a two-dimensional region of reciprocal space. This intensity was analyzed quantitatively to obtain the two-dimensional Cowley-Warren short-range-order parameters and atomic displacements. Monte-Carlo simulation based on these values has revealed that the atomic configurations in the surface consist of ordered domains and clusters in a disordered matrix. There is a large number of {10} anti-phase domain boundaries (APDB).
Order No.: JA701-013

Fractal morphologies from decomposition of Fe-Ni-Invar alloys
Q. Li, A. Wiedenmann, H. Wollenberger
(Hahn-Meitner-Institut Berlin)

Small angle neutron scattering investigations performed on Fe_{1-x}-Ni_x alloys with $0.26 \leq x \leq 0.45$ revealed a thermodynamically driven decomposition below 800°C. The miscibility gap extends at least from 30 at.% Ni to 45 at.% Ni. The diffusion controlled decomposition produces fractal morphologies during the early stages of the process. During annealing the structure of the precipitated phase densifies continuously from mass fractals with small dimensionality ($1 \leq d_m \leq 3$) to surface fractals with $2 \leq d_s \leq 3$. The results are compared with recent simulations of aggregation and growth as well as with a new analysis of spinodal decomposition, both predicting fractal morphologies.
Order No.: JA701-014

Embedded atom calculations of unstable stacking fault energies and surface energies in intermetallics
D. Farkas*, S.J. Zhou+, C. Vailhé*, B. Mutasa*, J. Panova*
*(*Virginia Polytechnic Institute and State University, +Los Alamos National Laboratory)*

We performed Embedded Atom Method calculations of surface energies and unstable stacking fault energies for a series of intermetallics for which interatomic potentials of the embedded atom type have recently been developed. These results were analyzed and applied to the prediction of relative ductility of these materials using the various current theories. Series of alloys with the B2 ordered structure were studied and the results were compared to those in pure b.c.c. Fe. Ordered compounds with $L1_2$ and $L1_0$ structures based on the f.c.c. lattice were also studied. It was found that there is a correlation between the values of the Anti-Phase Boundary (APB) energies in B2 alloys and their unstable stacking fault energies. Materials with APB energies tend to have higher unstable stacking fault energies, leading to an increased tendency to brittle fracture.
Order No.: JA701-015

X-ray photoelectron spectroscopy studies of silicon suboxides obtained by the sol-gel method
S. Santucci*, E. Cordeschi*, L. Lozzi*, M. Passacantando*, P. Picozzi*, L. Mancinelli degli Esposti+
*(*Università dell'Aquila, +ISRIM)*

Silicon suboxide thin films obtained by sol-gel and dip-coating methods, starting from a sol containing different percentages of TEOS (tetraethoxysilane) and MTEOS (methyltriethoxysilane), were grown onto silicon substrates. The samples were annealed at 100, 300 and 500°C and the electronic and compositional properties of the surface were studied by XPS detecting the Si "Auger parameter" and the valence band. The effects produced by an ion-sputtering treatment of the samples were also studied.
Order No.: JA701-016

Fatigue in hydrazone-based xerographic photoreceptors—Effect of ultraviolet irradiation
C.K.H. Wong, Y.C. Chan, J. Pfleger, Y.W. Lam, K.M. Leung, D.S. Chiu
(City University of Hong Kong)

The effect of ultraviolet irradiation on the xerographic sensitometry of organic photoreceptors was studied. Absorbed ultraviolet light decreased both the dark decay and the photoinduced discharge rates, and an increased buildup of the residual potential was observed. Above a threshold dose of ultraviolet irradiation, the residual potential was seen to decrease, and at the same time a slight increase of the hardness of the photoreceptor surface was detected. These behaviors originate from a decrease in the density of charge transport sites which is caused by the photochemical changes in the Charge Transport Layer system.
Order No.: JA701-017

Copper-boron nitride interaction in hot-pressed ceramics
M. Hubáček*, T. Sato*, M. Ueki+
*(*National Institute for Research in Inorganic Materials, +Nippon Steel Corporation)*

Ceramics were prepared by hot pressing of chemical mixtures of turbostratic boron nitride with copper. A positive effect of copper on boron nitride crystallization and densification has been found. Preferred orientation of boron nitride grains has been revealed in such ceramics, being opposite to general experience: The c-axis of boron nitride crystallites was arranged preferably within a plane perpendicular to the direction of uniaxial pressure. Such an arrangement is interpreted as a result of the uniaxial compression of the boron nitride grains extensively growing within the c crystallographic axis.
Order No.: JA701-018

Epitaxial dependence of the melting behavior of In nanoparticles embedded in Al matrices
H.W. Sheng, G. Ren, L.M. Peng, Z.Q. Hu, K. Lu
(Chinese Academy of Sciences)

Nanometer-sized In particles (5~45 nm) embedded in the Al matrix were prepared by using melt-spinning and ball-milling techniques. Different crystallographic orientationships between In nanoparticles and the Al matrix were constructed by these two approaches. Melting behavior of the In particles was investigated by means of differential scanning calorimetry (DSC). It was found that the epitaxially oriented In nanoparticles (with the Al matrix) in the melt-spun sample were superheated about 0~38°C; whereas the randomly oriented In particles in the ball milled sample melted below its equilibrium melting point by about 0~22°C. We suggest that the melting temperature of In nanoparticles can be either enhanced or depressed, depending on the epitaxy between In and the Al matrix.
Order No.: JA701-019

Finite-size corrections for the Johnson-Mehl-Avrami-Kolmogorov equation
L.E. Levine, K. Lakshmi Narayan, K.F. Kelton
(Washington University)

The Johnson-Mehl-Avrami-Kolmogorov (JMAK) equation is frequently used to describe phase transformations involving nucleation and growth. The assumptions used in the derivation of this equation, however, are frequently violated when making experimental measurements; use of the JMAK equation for analyzing such data can often produce invalid results. Finite-size effects are among the most serious of these problems. We present modified analytic JMAK equations that correct for the finite-size

effects and are roughly independent of both the sample shape and the shape of the growing nuclei. A comparison with computer simulations shows that these modified JMAK equations accurately reproduce the growth behavior over a wide range of conditions.
Order No.: JA701-020

Reactive phase formation in sputter-deposited Ni/Al multilayer thin films
K. Barmak*, C. Michaelsen+, G. Lucadamo*
*(*Lehigh University, +GKSS Research Center)*

We have investigated reactive phase formation in magnetron sputter-deposited Ni/Al multilayer films with a 1:3 molar ratio and various periodicities, Λ, ranging from 320 nm down to a codeposited film with zero effective periodicity. The films were studied by x-ray diffraction, differential scanning calorimetry, electrical resistance measurements, and transmission electron microscopy. We find that Ni and Al have reacted during deposition to form the B2 NiAl phase and an amorphous phase. The formation of these phases substantially reduces the driving force for subsequent reactions and explains why nucleation kinetics become important for these reactions. Depending on the periodicity, these reactions result in the formation of $NiAl_3$ or Ni_2Al_9 followed by $NiAl_3$. Detailed calorimetric analysis reveals differences in the nucleation and growth behavior of $NiAl_3$ compared with other studies.
Order No.: JA701-021

Response of silicon carbide to high-intensity laser irradiation in a high-pressure inert gas atmosphere
L.Y. Sadler, M. Shamsuzzoha
(The University of Alabama)

α-SiC (hexagonal) pressed powder targets were heated with a highly-focused beam of CO_2 laser radiation to temperatures in excess of 3200°C in an oxygen- and water-free argon atmosphere. The argon pressure was maintained at greater than 100 atm to prevent vaporization of any liquid formed. This pressure was estimated to be several times greater than the total vapor pressure above a liquid solution of carbon and silicon. After a 30 s period at temperature, the heating was abruptly terminated. The 1 mm diameter region on the surface of the SiC targets most affected consisted of a central crater from which all SiC had been vaporized. The crater walls consisted of an amorphous carbon phase which acted as a matrix for fibrous graphite and granular silicon. Beyond the crater wall, the microstructure changed to that of various polytypes of hexagonal SiC, with the grains being equiaxed and smaller than those of the starting SiC powder, an indication that melting, followed by formation of a finer-grain structure on abrupt cooling may have occurred. Still further from the crater, the microstructure was characterized by β-silicon (cubic) particles of spherical morphology and of larger size than the starting SiC powder, a response typical of solid state sintering. Even further from the crater, the structure was α-SiC, and the grain size was nearly the same as that of the starting material. The particles were lightly bonded together at points of contact between them, indicating only light sintering.
Order No.: JA701-022

Structural properties of molecular beam epitaxy-grown Ni/Pt-superlattices
W. Staiger, A. Michel, V. Pierron-Bohnes, N. Hermann, M.C. Cadeville
(CNRS-Université Louis Pasteur)

We find that the $[Ni_{3.2nm}Pt_{1.6nm}]$ x 15 and $[Ni_{3.2nm}Pt_{0.8nm}]$ x 15 multilayers are semicoherent and display a columnar morphology. From both the period of the moiré fringes and the positions of the diffraction peaks in electronic (plan view and cross-section geometries) and x-ray diffraction patterns, one deduces that the nickel is relaxed (at least in the error bars of all our measurements) whereas the platinum remains slightly strained (≈-1%). The interfaces are sharp: no intermixing takes place giving rise to neat contrasts in TEM and to high intensities of the superlattice peaks in the growth direction in both diffraction techniques. The relaxation of the interfacial misfit occurs partially through misfit dislocations, partially through the strain of platinum. A quasi periodic twinning occurs at the interfaces, the stacking fault which forms the twin being the most often located at the interface Pt/Ni, i.e., when a Pt layer begins to grow on the Ni layer. The simulation of the θ/2θ superlattice peak intensities takes into account the columnar microstructure. It shows that the roughness is predominantly at medium scale with a fluctuation of about 12.5 % for Ni layers and negligible for Pt layers.
Order No.: JA701-023

Epitaxial growth of aluminum nitride layers on Si(111) at high temperature and for different thicknesses
F. Malengreau*, M. Vermeersch*, S. Hagège+, R. Sporken*, M.D. Lange*, R. Caudano*
*(*Facultés Universitaires Notre-Dame de la Paix, +CNRS)*

We present the epitaxial growth by rf reactive sputtering of aluminum nitride on Si(111) at high temperature. The grain size of the obtained films was sufficient to obtain a good Low Energy Electron Diffraction pattern (LEED) from which we determined a lattice parameter of 3.1 Å, indicative of fully relaxed films. The surface of the film was examined *in situ* by Auger electron spectroscopy (AES); and no contamination was detected, with the exception of low levels of oxygen. The film and its interface were studied by High Resolution Electron Energy Loss Spectroscopy (HREELS), X-ray Photoelectron Spectroscopy (XPS) depth profiling, and Transmission Electron Microscopy (TEM). Again, a low concentration of oxygen and no carbon contamination were detected by XPS. Three different growth methods were applied to the deposition of aluminum nitride at high temperature. The obtained films were studied in order to determine the influence of the methods on the interface, on the "bulk structure" of the film and on its surface. Each has been shown to have particular characteristics. The first one, performed at a temperature of 1000°C, and including a cleaning of the surface by exposure to Al flux, was characterized by an interfacial layer with no long range order and increasing the interaction between the film and the substrate. The second growth consisting of deposition at the same high temperature has shown a good surface quality for very thin layers (<50 Å) and the absence of an interfacial layer. The last method, based on a first step of growth at low temperature (700°C) resulted in good quality, thick layers which allowed us to determine the infrared dielectric constants of aluminum nitride by HREELS.
Order No.: JA701-024

Low temperature synthesis of lead titanate by a hydrothermal method
J. Moon*, T. Li*, C.A. Randall+, J.H. Adair*
*(*University of Florida, +The Pennsylvania State University)*

Alkoxide-based hydrothermal powder synthesis of lead titanate was investigated. The objective of this work was to lower the synthesis temperature. By modifying titanium isopropoxide with acetylacetone during solution mixing, the phase-pure lead titanate with perovskite structure was synthesized at temperatures as low as 150°C. It was determined that the pH of the hydrothermal reaction medium and the initial Pb/Ti ratio are critical factors in forming stoichiometric $PbTiO_3$. When the pH of the initial feedstock is above 14 and the Pb/Ti ratio is greater than 1.5, a phase-pure $PbTiO_3$ can be obtained. The modification of titanium alkoxide gave rise to the formation of a stable complex against hydrolysis and eventually reduced the synthesis temperature significantly. A possible formation mechanism for $PbTiO_3$ is the dissolution recrystallization from an amorphous precursor to a well-crystalline product as originally proposed by Rossetti, et al. Hall-Williamson analysis was also performed on the hydrothermally derived $PbTiO_3$ to interpret the systematic peak broadening and asymmetry for {001} reflections unlike the commercial $PbTiO_3$. It was observed that the strain in the c-axis direction is much higher than that in a-axis direction while the domain sizes for both directions are similar. This strain anisotropicity exerted in the particles may indicate a unique domain structure in the hydrothermally synthesized particles in which either only 180° domains exist or possibly only a single domain.
Order No.: JA701-025

Sintering of the ultrahigh pressure densified hydroxyapatite monolithic xerogels

J. Majling*, P. Znáik*, A. Palová*, S. Svetík*, S. Kovalík*, D.K. Agrawal+, R. Roy+
(*Slovak Technical University, +The Pennsylvania State University)

Dense and translucent ceramics were prepared by sintering of cylindrical preforms of hydroxyapatite extruded from xerogels. Extruded specimens were dried as monoliths and then consolidated by applying cold isostatic pressure, ranging from 500 to 1500 MPa. Upon heating the samples began to densify at 610°C and the densification/sintering was completed at 870°C as was evidenced by the dilatometry plot indicating no further shrinkage. The sintered specimens thus formed were translucent in appearance. Further heating of the samples up to 1200°C resulted in their "bloating" or creation of pores in the originally dense matrix. Pore creation within the structure is reproducible; it proceeds from the surface to the interior of the sample, and its spreading can be thermally controlled. Pore evolution within the single phase dense polycrystalline material is not related to the frequently occurring phenomenon of microcracking in ceramics during cooling.

Order No.: JA701-026

High temperature strength of silicon nitride ceramics with ytterbium silicon oxynitride

T. Nishimura*, M. Mitomo*, H. Suematsu+
(*National Institute for Research in Inorganic Materials, +Tokyo Institute of Technology)

Silicon nitride ceramics with ytterbium silicon oxynitride ($Yb_4Si_2O_7N_2$) as secondary phase were fabricated by hot-pressing the powder mixtures including 50.0 to 97.0 mol% of silicon nitride with a mixture of Yb_2O_3 and SiO_2 ($Yb_2O_3/SiO_2 = 4$). Sinterability of the materials with Yb_2O_3 was higher than that with Yb_2O_3 in the same composition of raw powder mixtures. High density materials were obtained under the condition of 50.0 to 89.1 mol% of silicon nitride in raw powder mixtures. Mechanical properties of silicon nitride containing 97.6 mol% of Si_3N_4 and 2.4 mol% of $Yb_4Si_2O_7N_2$ were measured. Fracture toughness measured by indentation technique was 5.9 $MPam^{1/2}$. Bending strength at room temperature and at 1500°C was 977 MPa and 484 MPa, respectively. The silicon nitride grains consisted of highly elongated rodlike grains and thin needlelike grains. The $Yb_4Si_2O_7N_2$ grains were crystallized at multigrain junctions and bonded close to Si_3N_4 grains. High strength at high temperature is supposed to be based on the presence of crystalline $Yb_4Si_2O_7N_2$ having a high melting point.

Order No.: JA701-027

Residual surface stress by localized contact-creep

S. Widjaja, K. Jakus, R. Atri, J.E. Ritter, S. Bhattacharya
(University of Massachusetts)

When a ceramic material creeps under a localized stress and then cools under load, a portion of the creep flow stress is retained as a residual compressive stress due to elastic rebound being constrained by the creep zone. Localized contact-creep was used to generate residual compressive surface stress in soda-lime glass and two sintered aluminas. The Vickers indentation technique was used to measure the residual stress within the contact-creep area. Alumina with a higher elastic modulus than glass retained higher residual compressive surface stress. The results were in reasonable agreement with the predicted stress distribution given by Finite Element Analysis.

Order No.: JA701-028

Excimer laser ablation of aluminum nitride

J.K. Lumpp*, S.D. Allen+
(*University of Kentucky, +Tulane University)

Excimer laser wavelengths ablate aluminum nitride at rates up to 0.2 μm/pulse where the rate increases with decreasing background pressure and increasing fluence. The ablation threshold for AlN at 248 nm is approximately 2 J/cm^2. Blind vias are produced with flat bottoms, straight walls and a decomposed metallic layer remaining on the surface. Ablation rate dependence on fluence saturates at high fluences due to absorption by the ablation plume. The influence of processing variables on ablation rate and ablation mechanisms are discussed. Statistical design of experiment techniques are used to compare data sets.

Order No.: JA701-029

Change of the weak field properties of $Pb(ZrTi)O_3$ piezoceramics with compressive uniaxial stresses and its links to the effect of dopants on the stability of the polarizations in the materials

Q.M. Zhang, J. Zhao, K. Uchino, J. Zheng
(The Pennsylvania State University)

The properties of several $Pb(ZrTi)O_3$ (PZT) piezoceramics under compressive uniaxial stresses were characterized. It was observed that uniaxial stresses have a marked effect on the soft PZT materials, including reducing the piezoelectric coefficients and depoling the samples at relatively low stress levels. The effect of the uniaxial stresses on the properties of hard PZTs is more complicated because the domain structure of the materials can be changed substantially without depoling the samples. Therefore, under a compressive stress along the poling direction, the piezoelectric and electromechanical coupling factor can be increased markedly due to both the increased non-180° domain boundary motions and the deaging effect. In addition, the experimental results support the notion that the difference between a hard PZT and a soft PZT lies in the types of defects introduced by dopants. Immobile defects create frustrations in the lattice and result in a soft behavior and mobile defects stabilize the polarization and produce a hard behavior.

Order No.: JA701-030

Computer simulations of interactions between ultrafine alumina particles produced by an arc discharge

M.H. Teng, L.D. Marks, D.L. Johnson
(Northwestern University)

We wrote two computer programs, *3D* and *BUMP*, to interpret transmission electron microscope (TEM) micrographs made during a study of the initial stage sintering of ultrafine alumina particles (UFPs, 20–50 nm in diameter). The first simulated the 3-D geometric relationships of particles, from which we concluded that surface diffusion was the predominant sintering mechanism because no shrinkage occurred. *BUMP* simulated random contact of two particles and showed that the particle chains that formed before sintering were not formed purely by chance. Instead the particles experienced a rearrangement process (rotation and sliding) which reduced the total surface energy.

Order No.: JA701-031

Balance of graphite deposition and multi-shell carbon nanotube growth in the carbon arc-discharge

P.M. Ajayan, Ph. Redlich, M. Rühle
(Max-Planck-Institut für Metallforschung)

Except for atomistic models of individual carbon nanotube growth, there is still no precise understanding of the large scale deposition of carbon during the arc-discharge. We study the microstructure of cathode deposits in detail using scanning electron microscopy focusing on two distinct regimes found in the deposits, having different large scale morphology. The shell grown circumferentially consists of extended graphite layers with preferred orientation and the structure reveals close similarity to pyrolitic graphite. The core region is a porous assembly of nanotubes and nanoparticles. We conclude that closed nanostructures self-assemble from a dense carbon vapor whereas pyro-graphitic shell grows by continuous deposition on exposed substrates.

Order No.: JA701-032

The characterization of strain, impurity content and crush strength of synthetic diamond crystals

T.L. McCormick*, W.E. Jackson+, R.J. Nemanich*
*(*North Carolina State University, +GE Superabrasives)*

This study addresses the correlation of the macroscopic and microscopic characteristics of synthetic diamond crystals produced by high pressure – high temperature conditions. Microscopic properties were characterized using Raman spectroscopy, birefringence, and photoluminescence (PL). Macroscopic properties characterized included inclusion content and crush force. Raman measurements detected measurable stress shifts in only two samples. The PL measurements indicated an increased presence of the H3 center in areas of high strain. The absence of the H3 center and the presence of the N-V PL center were correlated to lower average crush force. A hierarchy has been developed that relates microscopic properties to average crush force.

Order No.: JA701-033

The structural characterization of amorphous thin films and coatings in their *as-deposited* state using x-rays at shallow angles of incidence

J.S. Rigden*, R.J. Newport*, G. Bushnell-Wye+
*(*The University-Canterbury, +CLRC)*

We demonstrate the method of x-ray diffraction at shallow angles of incidence, using the intrinsically highly collimated x-ray beam generated by a synchrotron source, to study the atomic scale structure of amorphous thin films and coatings in their *as-deposited* (i.e., on-substrate) state. As the incident angle is decreased, scattering from the film/coating can be isolated as contributions from the substrate are reduced. Systems studied include CVD diamond films deposited onto both silicon and steel substrates, where evidence of an interfacial region between the film and silicon wafer has been observed, but we focus on a range of amorphous films/coatings (mixed TiO_2:SiO_2 sol-gel spun films, hydrogenated carbon films and "glassy" carbon coatings, silicon:germanium semiconducting films and alumina coatings). The data are used both to comment upon the systems studied and to elucidate the potential, and the limitations, of the experimental method.

Order No.: JA701-034

Effect of oxygen gas on polycarbonate surface in keV energy Ar^+ ion irradiation

J-S. Cho*, W-K. Choi*, K.H. Yoon+, H-J. Jung*, S-K. Koh*
*(*Korea Institute of Science and Technology, +Yonsei University)*

Ar^+ ion irradiation on polycarbonate (PC) surface was carried out in oxygen environment in order to investigate effects of surface chemical reaction, surface morphology, and surface energy on wettability of PC. Doses of Ar^+ ion were changed from 5×10^{14} to 5×10^{16} at 1 keV ion beam energy by a broad ion beam source. Contact angle of PC was not much reduced by Ar^+ ion irradiation without flowing oxygen gas, but decreased significantly as Ar^+ ion was irradiated with flowing 4 sccm (ml/min.) oxygen gas and showed a minimum value of 12° to water and 5° to formamide. A newly formed polar group was observed on modified PC surface by Ar^+ ion irradiation with flowing oxygen gas, and it made PC surface energy increase. On a basis of x-ray photoelectron spectroscopy analysis, the formed polar group was identified as a hydrophilic C=O bond(carbonyl group). In AFM study, the root mean square of surface roughness was changed from 14 Å to 22~27 Å by Ar^+ ion irradiation without flowing oxygen gas and 26~30 Å by Ar^+ ion irradiation with flowing 4 sccm oxygen gas. It was found that wettability of modified PC surface was not greatly dependent on the surface morphology but on an amount of hydrophilic group formed on surface in the ion beam process.

Order No.: JA701-035

COMMENTARIES AND REVIEWS

Laser-assisted formation of metallic oxide microtubes

L. Nánai*, T.F. George+
*(*József Attila University, +University of Wisconsin-Stevens Point)*

The fabrication of metallic oxide microtubes is possible directly, without any support structure, by CW infrared laser-assisted oxidation of the metal in air. The particular case presented is the growth of tubelike vanadium pentoxide microcrystals grown in our laboratories.

Order No.: JA701-036

Please use the convenient postcard located in the back of the *MRS Bulletin* to order *JMR* reprints. When ordering single article reprints please note they are not available until the issue is published. See *JMR* Abstracts on the MRS Homepage at http://www.mrs.org/publications/jmr/jmra/