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ABSTRACTS

COMMUNICATIONS

Deposition and Characterization of Crystalline Conductive RuO_2 Thin Films
Q.X. Jia, S.G. Song, S.R. Foltyn, X.D. Wu
(Los Alamos National Laboratory)

Highly conductive metal-oxide RuO_2 thin films have been successfully grown on yttria-stabilized zirconia (YSZ) substrates by pulsed laser deposition. Epitaxial growth of RuO_2 thin films on YSZ and the atomically sharp interface between the RuO_2 and the YSZ substrate are clearly evident from cross-sectional transmission electron microscopy. A diagonal-type epitaxy of RuO_2 on YSZ is confirmed from x-ray diffraction measurements. The crystalline RuO_2 thin films, deposited at temperatures in the range of 500°C to 700°C, have a room-temperature resistivity of 35±2 μΩ-cm, and the residual resistance ratio ($R_{300K}/R_{4.2K}$) is around 5 for the crystalline RuO_2 thin films.
Order No.: JA510-001

A Novel Route for the Synthesis of $Sr_{1-x}Ba_xNb_2O_6$ Thin Films
A. Kasbani, M.S. Tomar, E. Dayalan
(The University of Tulsa)

Stoichiometric $Sr_{1-x}Ba_xNb_2O_6$ (SBN) powder and thin films were prepared by a chemical method. The starting materials were niobium ethoxide and the hydroxides of strontium and barium. Powders were obtained by evaporation of the precursor solution, and thin films were deposited by spin coating. Annealing temperature required to obtain complete conversion to the crystalline material was about 700°C. Stoichiometric polycrystalline films of $Sr_{1-x}Ba_xNb_2O_6$ were deposited on quartz and silicon substrates. Leakage current-voltage and the capacitance-voltage measurements on a metal/SBN/n-silicon structure show a diode type characteristic.
Order No.: JA510-002

The Debye Temperature of Nanocrystalline β-Sn Measured by X-ray Diffraction
L.B. Hong, C.C. Ahn, B. Fultz
(California Institute of Technology)

A nanocrystalline β-Sn film of 7 nm average grain size was prepared by inert gas condensation followed by ballistic consolidation, and was investigated by x-ray diffractometry at temperatures of 77 and 293 K. Although Sn normally undergoes a $\beta \rightarrow \alpha$ phase transformation at 286 K, this transformation was suppressed in the nanocrystalline film. Compared with large-grained β-Sn, a larger Debye-Waller factor and a lower Debye temperature were measured for nanocrystalline β-Sn; Θ_D = 133 K for nanocrystalline material while Θ_D = 161 K for large grained material. The lower Debye temperature of the nanocrystalline β-Sn indicates that its vibrational entropy is increased by 0.6 k_B/atom with respect to large-grained material.
Order No.: JA510-003

Conventional and Microwave Sintering of Condensed Silica Fume
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Condensed silica fume, a by-product from the production of silicon alloys, was sintered by (i) conventional heating in a dilatometric furnace, both at constant heating rate and isothermal heating, and (ii) by the microwave heating. The dense products, with relative density up to 95% of theoretical can be obtained only by short runs, at high heating rates, preferentially accomplished by the microwave treatment. Prolonged heating leads to the devitrification of the original glassy phase to cristobalite, accompanied by an arrest of densification.
Order No.: JA510-004

SiC-Al-Si Composites by Rapid Pressureless Infiltration in Air
X.F. Yang, X.M. Xi
(Auburn University)

A rapid, pressureless infiltration technique for fabricating SiC-reinforced Al-Si composites is described. The infiltration can be performed by simply dipping a ceramic preform into a molten alloy in an open air environment. This method makes it possible to produce composites that contain a high volume fraction (80%) of a variety of ceramic reinforcements, including particulates and fibers. This technique has potential for low-cost and versatile production of SiC-Al-Si composites for many industrial applications, such as automobile components and electronic packaging.
Order No.: JA510-005

Structure of Au Ultrafine Particles in Silica Glass by X-ray Absorption Fine Structure Spectroscopy
K. Fukumi, H. Kageyama, K. Kadono, A. Chayahara, N. Kamijo, M. Makihara, K. Fujii, J. Hayakawa, M. Satou
(Osaka National Research Institute)

Structure of ultrafine gold particles embedded in silica glass by ion implantation has been studied by x-ray absorption fine structure spectroscopy. It is found that the Au-Au interatomic distance in the particles is very similar to that in bulk gold within 0.01 Å. Mean-square relative displacement in the particles is 1.2–1.3 times larger than that in bulk gold.
Order No.: JA510-006

ARTICLES

Parametric Study of *In-Situ* Growth of NdCeCuO Thin Films by Laser Ablation
W-T. Lin, G-J. Chen
(National Cheng Kung University)

The stability fields of the NdCeCuO (NCCO) films and the $Ce_{0.5}Nd_{0.5}O_{1.75}$ (CNO) phase grown by laser ablation from the stoichiometric targets, $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$, are demonstrated, respectively. The superconducting NCCO films were grown in a specific field below and close to the CuO/Cu_2O transition line, i.e., at 700–725°C in 20–50 mTorr, without vacuum annealing. On post-deposition cooling in lower oxygen pressures (<10 mTorr) the superconductivity of the NCCO films was significantly improved without vacuum annealing. The CNO phase usually appeared in the NCCO films at temperatures above 680°C and the higher the temperature the more the CNO phase due to copper loss. Surplus of copper in the NCCO targets effectively suppressed the formation of the CNO phase and thereby improved the superconductivity of the NCCO films.
Order No.: JA510-007

Interface Between Gold and Superconducting $YBa_2Cu_3O_{7-x}$
S-W. Chan*, L. Zhao*, C. Chen*, Q. Li+, D.B. Fenner+
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Gold(Au) and silver are known to be important contact metals on $YBa_2Cu_3O_{7-x}$ (YBCO). Both metals have been used as additives in fabricating tapes of YBCO and $Bi_2Sr_2CaCu_2O_8$ (BSCCO) materials and have favorable results in improving not only the flexibility but also the weighted critical currents of the resulting composites. Previous results on superconductor/normal metal/superconductor junctions made using YBCO/Au/YBCO and YBCO/Au/Nb demonstrated that a supercurrent can be induced in the normal metal layers through the proximity effect. Our transmission electron microscopy study of the Au/YBCO interfaces shows well-bonded interface with no extraneous phases present. Lattice fringes of the (001) plane in YBCO terminated at the interface abruptly. This observation supports previous results of contact resistance and x-ray photoelectron spectroscopy (XPS). Both (001) integral steps and multiples of 1/3(001) steps were observed at the Au/YBCO interface. When the top gold layer was absent locally, surface degradation was observed as the (001) lattice fringes stopped short from the surface by 10 nm. Our results support that Au is a desirable contact metal and a dependable surface passivation material for YBCO.
Order No.: JA510-008

Fabrication and Characterization of Highly Textured $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ Superconducting Ceramics Using High Magnetic Field and Cold Isostatic Pressing
W. Lo*, R. Stevens+, R. Doyle*, A.M. Campbell*, W.Y. Liang*
*(*IRC in Superconductivity, +University of Bath)*

Highly textured $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ ceramics have been fabricated by aligning deflocculated flakes of $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ suspended in an organic medium by means of a high dc magnetic field (6T) at room temperature followed by cold isostatic pressing. The proportion of the $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ phase in the precursor powder was carefully controlled and the characteristics of the powder, such as size distribution and morphology, were determined. A high degree of grain alignment was found in the specimens after the magnetic alignment, although the bulk density of the materials was low. Cold isostatic pressing substantially increased the density of the magnetically pre-aligned specimens which also resulted in a slight decrease in the degree of grain alignment. This minor realignment was found to be due to the various kinds of processing defects that appeared in the specimens during compaction due to the grinding and cracking of the grains and their interlocking. The microstructural and superconducting properties of the sintered ceramic have been studied using texture goniometry, high resolution scanning electron microscopy, transmission electron microscopy, ac magnetic susceptometry and critical current measurements.
Order No.: JA510-009

Magnetic Torque of Oriented Bulk Bi2212 by Shock-Loading Method
S. Kawamata*, K. Okuda*, M. Kikuchi+, H. Hikosaka+, Y. Syono+
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The magnetic torque of oriented bulk specimen of $Bi_2Sr_2CaCu_2O_{8+\delta}$ by shock-loading method was measured under magnetic fields up to 8 kOe in the temperature range from 4.2 to 100 K. The torque amplitude of the shocked specimen was much larger than that of the as-grown-bulk specimen measured as a reference. The angular dependence of the torque was reproduced assuming the Gauss-type distribution for crystalline orientation. It became clear that the magnetic torque of shocked specimen showed the high degree of crystalline orientation in which the *c*-axis is perpendicular to the pellet disk plane and that the flux pinning perpendicular to the pellet disk plane was enhanced by shock-loading.
Order No.: JA510-010

Anomalous Electrical Behavior of Partially Carbonized Polyacrylonitrile Fibers
B.Z. Jang, L. Zhao
(Auburn University)

Partially carbonized polyacrylonitrile fibers were observed to undergo a resistivity change of 2 to 4 orders of magnitude at a transition temperature typically in the range of 98°C to 200°C. The current-voltage curves exhibited an initial supercurrent-like increase, followed by a rapid drop to a high resistance state, and then a rise in current again at a later stage. These phenomena cannot be interpreted by existing theories on switching in inorganic amorphous semiconductors. They are explainable if the microstructure of the pyrolyzed fiber is viewed as comprising nanometer scale superconducting phases interspersed with semiconducting phases, much like a large number of Josephson junctions connected in series.
Order No.: JA510-011

Preparation and Chemical Analysis of High Purity Iron-Zinc Alloys
R.G. Grant, P.S. Cook, D.C. Cook
(Old Dominion University)

We have produced and chemically analyzed a series of high purity iron-zinc alloys with iron concentrations in the range 4–27 wt.%. The technique involved slow diffusion of high purity zinc with small particle iron powder. We produced alloys within the four main iron-zinc phases, Zeta, Delta, Gamma-1 and Gamma to aid in the identification of the intermetallic phases formed in commercially produced galvanneal steel coatings. The diffusion technique produces iron-zinc alloys which are suitable as instrument calibration standards for galvanneal producers

and users. A chemical titration technique which measures iron concentration to within 0.5 wt.%, was also refined. In determining the bulk iron concentration of the samples, we have compared the accuracy of the wet chemical technique with Induction Coupled Plasma spectroscopy. Homogeneity was measured using an electron microprobe and a scanning transmission electron microscope. The data shows that samples are homogeneous to greater than 98% of the mean iron concentration.

Order No.: JA510-012

Microstructure and High Temperature Deformation of the C15 $NbCr_2$-Based Laves Intermetallics in Nb-Cr-V Alloy System

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The microstructure and the high temperature deformation behavior of the C15 $NbCr_2$ intermetallics alloyed with V, which consist of both the C15 monophase and the duplex structure with the bcc solid solution, are observed by some microscopic techniques and compression tests. The alloys consisting of the C15 monophase microstructure show moderately higher 0.2% yield stress than the unalloyed $NbCr_2$, and the same BDTT as that in the unalloyed $NbCr_2$, indicating a moderate improvement of the high temperature deformability. This result is attributed to the larger atomic free volumes between the atomic stacking layers along the {111} planes than those of the unalloyed $NbCr_2$. The alloys consisting of the duplex microstructure show lower 0.2% yield stress and considerably decreased BDTT than the unalloyed $NbCr_2$. The considerable deformation improvement in this group of alloys is attributed to the existence of the ductile bcc solid solution.

Order No.: JA510-013

The Influence of the Reactant Size on the Micropyretic Synthesis of NiAl Intermetallic Compounds

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(University of Cincinnati)

The effect of the nickel (Ni) and aluminum (Al) reactant particle size on the micropyretic synthesis of NiAl is studied in this article. A change in the low melting component (Al particle) size is noted to have a limited influence on the micropyretic synthesis conditions. However, a change in the high melting component (Ni particle) size not only influences the combustion temperature and propagation velocity, but also affects the final porosity and grain size of the synthesized products. The combustion mode is also noted to change from stable to unstable when the Ni particle size is increased. It is noted that a diffusion type control mechanism is dominant for the rapid reaction sequence in the NiAl system. An atomistic mechanism of the Ni-Al micropyretic reaction is also proposed in this article. Following this model, analytical expressions are developed to relate the variation of the Ni size and NiAl formation rate with the imposed processing conditions during the micropyretic synthesis. The mechanism of the final grain formation and the grain size changes with changes in the processing variables is also discussed.

Order No.: JA510-014

Silicon Carbide Fiber Reinforced Alumina Extrusion

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(The University of Birmingham)

Silicon carbide fiber reinforced alumina bodies have been produced by ram extrusion. The Al_2O_3 powder and SiC fiber were milled together to give a dry dispersion of up to 30 vol.% fiber which was subsequently mixed to a paste by high shear kneading using hydroxypropylmethylcellulose solutions as the binder phase. Extruded bodies with green densities ranging between 56 and 63% full theoretical density were achieved. The paste flow behavior was characterized using physically based equations which show that for any given moisture content the pressure drop and the constituent paste parameters are all systematically reduced as the fiber loading is increased. This observation can be explained almost completely by combining packing theory with the paste rheology data. Fiber interactions within the paste and the die system appear not to greatly influence the rheological character of the material. It is shown statistically that the fibers are homogeneously dispersed throughout the paste mass after extrusion. Image analysis has been used to aid in macro defect analysis and it is shown that the optimum concentration of fiber was 20 vol.% with a quantity of binder sufficient to give an initial yield stress of 2 MPa.

Order No.: JA510-015

Investigation of SiC-AlN: Part III. Static and Dynamic Fatigue

J-L. Huang, J-M. Jih
(National Cheng Kung University)

SiC/AlN composites with controlled interfacial solid solution were employed in this present work to investigate the effects of interfacial chemical composition and AlN polytypes on the fatigue properties.

The dynamic strength of SiC/AlN composite was found to initially decrease as the stressing rate decreased. However, the strength increased with a decreasing in stress rate at a low stress rate region of below 0.01 MPa/sec. Crack arrest could have occurred before exhibiting spontaneous failure at an intermediate stress rate of 0.01 MPa/sec.

It was found that both the interfacial bonding strength and testing technique had essential effects on the behaviors of slow crack growth.

Order No.: JA510-016

Processing of High-Strain-Rate Superplastic Si_3N_{4w}/Al-Mg-Si Composites

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Effects of reduction, reinforcements and dynamic precipitation on grain refinement by hot extrusion have been investigated in a Si_3N_{4w}/Al-Mg-Si (6061) composite by tensile tests and microstructural investigation. The grain refinement was attributed to an interaction of recrystallization and dynamic precipitation, which was enhanced by reinforcements. Not only fine-grained microstructure but also relaxing stress concentrations which are caused at matrix/reinforcement interfaces during superplastic flow are required to attain large elongations. Partial melting resulting from segregation plays an important role in relaxing the stress concentrations. DSC investigations revealed that the degree of segregation was increased by hot extrusion.

Order No.: JA510-017

Measurement of Surface Topography and Area-Specific Nanohardness in the Scanning Force Microscope

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A scanning force microscope (SFM) is employed to indent and image surfaces with sub-micron resolution. The SFM image shows the area and depth of each indentation as well as its location with respect to nearby topographic surface features. The image also reveals the surface roughness, which can set a lower limit on useful nanoindentation size. A cross section of a nitrided steel surface is measured to illustrate the method. The use of the SFM with separate tip-cantilever structures for indenting and imaging has significant advantages over other nanohardness methods for the study of samples with lateral inhomogeneities.

Order No.: JA510-018

Analysis of Pore Structure of Activated Carbon Fibers Using High Resolution Transmission Electron Microscopy and Image Processing

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Activated carbon fibers (ACFs), already used widely as absorbent materials, are now expected to be useful as new electrical and electronic materials, for their very large specific surface areas (SSA). Chemical adsorption as well as x-ray diffraction have been mainly used for characterizing the ACF structure. While TEM observations reveal the texture of ACFs, such observations have not yet yielded quantitative information.

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To promote the quantitative interpretation of the TEM images, computer image analysis is used in this work to clarify the pore structure of ACFs.

The microstructures of three samples, which are all isotropic pitch-based ACFs but with different SSA values, have been investigated. Operations such as noise reduction, low frequency cut-off filtering, and binary image formation are used to clarify the pore images of the ACFs. The distribution of the ACF porosity size is clearly shown by a frequency analysis of the 2-dimensional fast Fourier transform (FFT). The results suggest that TEM images include contributions from many different pore sizes. Pores in different size ranges are extracted by the inverse FFT (IFFT) operation by selecting the specific frequency range, and by this analysis the pore structure is shown to have fractal characteristics.

Order No.: JA510-019

Growth of Highly Oriented $La_{0.84}Sr_{0.16}MnO_3$ Perovskite Films

B.W. Chung, E.L. Brosha, F.H. Garzon, I.D. Raistrick, R.J. Houlton, M.E. Hawley
(Los Alamos National Laboratory)

We have grown thin films of $La_{0.84}Sr_{0.16}MnO_3$ on $SrTiO_3$ (100), MgO (100), CeO_2 (100)/Al_2O_3, and (100) oriented yttria-stabilized zirconia (YSZ) substrates by using a 90° off-axis RF magnetron sputtering deposition. X-ray diffraction analysis and ion beam channeling experiments reveal that the deposited films grow epitaxially on $SrTiO_3$, biaxially textured on MgO, and highly textured on YSZ. Scanning tunneling microscopy reveals that the thin films possess extremely smooth surfaces.

Order No.: JA510-020

Neutron Irradiation and Annealing of ^{10}B Doped Chemical Vapor Deposited Diamond Films

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^{10}B doped diamond films grown by hot filament chemical vapor deposition were neutron irradiated at moderately high fluence levels. The as-irradiated and annealed samples, along with an unirradiated sample, were analyzed using Raman spectroscopy and x-ray diffraction. It was found that a nondiamond amorphous phase was formed on irradiation. This phase transformed back to diamond on annealing. No graphite formation was observed. A comparison with nondiamond powder was made. A similarity between irradiated diamond films and nanocrystalline diamond powder is discussed.

Order No.: JA510-021

Diamond-Graphite Hybrids and the Nature of Amorphous Carbon and Diamond-Like Carbon

R. Sen, R. Sumathy, C.N.R. Rao
(Indian Institute of Science)

The nature of amorphous carbon has been explored by molecular mechanics by examining the structures of species such as $C_{84}H_x$ and $C_{150}H_x$, wherein the percentage of sp^3 carbons is progressively increased in a graphitic network. The nature of diamond-like carbon has similarly been investigated by examining the structures of $C_{84}H_x$ and $C_{102}H_x$ where the percentage of sp^2 carbons is varied in a sp^3 network. The dependence of the average coordination number as well as the sp^3/sp^2 atom ratio on the atom fraction of hydrogen has been investigated in the light of the random covalent network model.

Order No.: JA510-022

Autostoichiometric Vapor Deposition I: Theory

R. Xu
(University of Utah)

The possibility of an autostoichiometric vapor deposition is explored. Heterometalorganic complexes such as double alkoxides are potential candidate precursors for such deposition. Two reaction schemes, the hydrolysis assisted pyrolysis and the hydrolysis-polycondensation of double alkoxides, are identified to be autostoichiometric reactions. A simple low-pressure apparatus is suggested for autostoichiometric vapor deposition. Mass-flow analysis allows for the identification of a non-stoichiometry factor *K* which can be used as a quantitative measure of the precursor's autostoichiometric capability.

Order No.: JA510-023

Autostoichiometric Vapor Deposition II: Experiment

K-W. Chour, R. Xu
(University of Utah)

An autostoichiometric vapor deposition method is implemented to achieve deposition of stoichiometric $LiTaO_3$ using $LiTa(OBu^t)_6$ precursor. Precisely stoichiometric $LiTaO_3$ can be grown by this method. A simple low pressure reactor is used to facilitate the autostoichiometric vapor deposition through hydrolysis polycondensation of double alkoxides in the vapor phase. Typical deposition and annealing conditions are described. X-ray diffraction, SEM and compositional analyses are performed on the films grown on fused silica, Pt, (100) sapphire and $LiNbO_3$ (001). Rocking curve measurements indicated excellent epitaxial growth of $LiTaO_3$ on (100) sapphire. Compositional analysis by measurement of lattice parameters confirmed that the present method can produce high quality stoichiometric $LiTaO_3$. The non-stoichiometry factor for $LiTa(OBu^t)_6$ is negligible. Mass spectrometric study of the precursor compound $LiTa(OBu^t)_6$ suggests that the volatile species is $Li_2Ta_2(OBu^t)_{12}$.

Order No.: JA510-024

Infrared Optical Properties of Pulsed Laser Deposited Carbon Films with the Bonding and Properties of Diamond

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(University of Texas at Dallas)

Composed of packed nanophase nodules in which the carbon atoms are linked with the tetrahedral bonding of diamond, laser plasma films are deposited in vacuum onto almost any substrate by condensing carbon ions carrying keV energies. These multiply charged ions are obtained from the laser ablation of graphite at intensities in excess of 10^{11} W cm^{-2}. The high energy of condensation provides both for the chemical bonding of such films to a wide variety of substrates and for low values of residual compressive stress. Coatings of 2–5 μm thicknesses have extended lifetimes of important optical materials against the erosive wear from high-speed particles and droplets by factors of tens to thousands. In this work, the optical properties of these films at infrared (IR) wavelengths were studied. Transmission spectra of several freestanding films on silicon frames were measured. Using a model considering rough surface scattering and free carrier absorption, satisfactory fits to these transmission spectra were obtained and from them the optical parameters were extracted. The characterization studies performed in this work indicated a great potential for the laser deposited nanophase diamond films in optical applications.

Order No.: JA510-025

Oxidation of Diamond Windows

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Diamond windows were heated at 700–900°C in air for short times to better define the limits of application at high temperature. The infrared (IR) transmission of polished chemical vapor deposition (CVD) diamond windows was reduced after heating in a furnace at 800°C for 75 s, while heating at 700°C for 75 s produced little change. The 800°C heating caused increased visible light scatter, and increased scatter appears to be mainly responsible for the reduced IR transmission. The forward scatter at 10.6 μm for the CVD diamond samples was 0.8% before heating, 2.8% after heating at 800°C for 75 s, and 6.2% after heating at 800°C for 255 s. Single crystal (110) type IIa diamond exhibited little change in IR transmission when heated at 800°C in air for 255 s, while heating for 555 s at 800°C caused a significant drop in IR transmittance (6–12%). A slight drop in IR transmittance (1–5%) occurred for a type IIa diamond when heated at 900°C for 45 s. The etched surfaces were characterized by differential interference contrast optical microscopy, scanning electron microscopy, and Talystep surface profiles. Etched features are concentrated at grain-boundaries (for the CVD samples) and at sites with residual damage from mechanical polishing that could not be seen before the heat treatment. Deep etch pits formed at grain boundaries that extend into the bulk of samples.

Order No.: JA510-026

Microstructure Development of Sol-Gel Derived Epitaxial $LiNbO_3$ Thin Films

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Film growth and microstructural evolution were investigated for sol-gel derived $LiNbO_3$ thin films deposited on lattice-matched single crystal substrates. Epitaxial $LiNbO_3$ films of about 100 nm nominal thickness were prepared by spin coating a solution of the lithium niobium ethoxide on sapphire (0001) substrates and annealing at 400°C or 700°C in a humidified oxygen atmosphere. These films exhibited an epitaxial relationship with the substrate of the type $LiNbO_3$ (0001)// α-Al_2O_3 (0001) and $LiNbO_3$ [100]//α-Al_2O_3 [100] as determined by x-ray pole figure analysis. Transmission electron microscopy indicated the epitaxial films annealed at 400°C consisted of slightly misoriented ~5 nm subgrains and of numerous ~10 nm enclosed pores. The microstructure and orientation development of these films was consistent with a heteroepitaxial nucleation and growth mechanism, in which epitaxial nuclei form at the substrate surface and grow upward into an amorphous and porous intermediate film. Epitaxial films annealed at 700°C contained larger 150–200 nm subgrains and pin holes. Misorientations between adjacent subgrains appeared to be significantly smaller in films annealed at 700°C than those in films annealed at 400°C.

Hydrolysis of the alkoxide precursor solution prior to spin coating promoted the development of polycrystalline films on single crystal sapphire substrates. Infrared spectra and thermal analysis indicated that, independent of the degree of the solution hydrolysis, nucleation of $LiNbO_3$ was immediately preceded by decomposition of an amorphous carbonate intermediate phase.

Order No.: JA510-027

Oxygen Incorporation in Aluminum Nitride via Extended Defects: Part III. Re-evaluation of the Polytypoid Structure in the Aluminum Nitride–Aluminum Oxide Binary System

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This paper extends the concepts that were developed to explain the structural rearrangement of the wurtzite AlN lattice due to incorporation of small amounts of oxygen, and to directly use them to assist in understanding the polytypoid structures. Conventional and high-resolution transmission electron microscopy, specific electron diffraction experiments and atomistic computer simulations have been used to investigate the structural nature of the polytypoids. The experimental observations provide compelling evidence that polytypoid structures are not arrays of stacking faults, but are rather arrays of inversion domain boundaries (IDBs).

A new model for the polytypoid structure is proposed with the basic repeat structural unit consisting of a planar IDB-P and a corrugated IDB. This model shares common structural elements with the model proposed by Thompson, even though in his model the polytypoids were described as consisting of stacking faults.

Small additions (≈1000 ppm) of silicon were observed to have a dramatic effect on the polytypoid structure. First, it appears that addition of Si causes the creation of a new variant of the planar IDB (termed IDB-P′), different from the IDB-P defect observed in the AlN-Al_2O_3 polytypoids; second, the addition of Si influences the structure of the corrugated IDB, such that it appears to become planar.

Order No.: JA510-028

Nuclear Magnetic Resonance Investigations of the Structural Phase Transitions of $AlPO_4$ Tridymite

Y. Xiao, R.J. Kirkpatrick
(University of Illinois)

^{27}Al and ^{31}P NMR spectroscopic data are presented for the tridymite polymorph of $AlPO_4$ ($AlPO_4$-t) through its structural phase transition at about 80°C. The RT ^{27}Al and ^{31}P spectra of $AlPO_4$-t both contain doublets of broad peaks indicating two well-separated groups of sites in the RT structure with mean Al-O-P bond angles per tetrahedron of ~147.8° and 153.1° (±1). With increasing temperature, the doublets remain the same up to about 74°C, where the relative intensities of the two peaks start to change. The peak corresponding to smaller Al-O-P bond angles disappears, and above ~88°C the ^{27}Al and ^{31}P spectra contain single symmetrical peaks, corresponding to a mean Al-O-P bond angle of 153.4°. This bond angle increases gradually with increasing temperature to 153.7° at ~150°C and remains constant to about 500°C. ^{27}Al quadrupole echo experiments suggest that the ^{27}Al nuclear quadrupole coupling constant (QCC) is small and decreases with increasing temperature. QCC remains non-zero in the high temperature phase of $AlPO_4$-t, consistent with the previously proposed $3m$ local symmetry of Al in the high temperature structure.

Order No.: JA510-029

Zirconia-Mullite Ceramics Made from Composite Particles Coated with Amorphous Phase: II. Effects of Boria Additions to the Amorphous Phase

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(Tatung Institute of Technology)

Mullite and zirconia-mullite ceramics have been prepared by coating alumina/zirconia particles with an amorphous silica layer. Effect of composition change of the amorphous silica layer by adding B_2O_3 was investigated. For the zirconia-free compositions, the addition of B_2O_3 remarkably accelerates the kinetics of the crystallization of the amorphous coating layer, the viscous sintering, and the mullitization. For the zirconia-containing ceramics, the addition of B_2O_3 enhanced the viscous sintering kinetics and delayed the decomposition of the transient zircon phase and the subsequent t- to m-ZrO_2 transition, thus resulting in a higher ratio of t- to m-ZrO_2. The B_2O_3-containing zirconia-mullite composites exhibit the same level of fracture toughness (K_{tc}) as the B_2O_3-free zirconia-mullite composites.

Order No.: JA510-030

Synthesis of Titanium Boride (TiB_2) Nanocrystallites by Solution-Phase Processing

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(Washington University)

Nanocrystalline TiB_2 is prepared by reaction of $NaBH_4$ and $TiCl_4$. The initial solution-phase reaction affords an amorphous precursor powder from which 5–100 nm TiB_2 crystallites are obtained upon

annealing at 900–1100°C. Crystallite sizes depend on the annealing temperature and other processing parameters. Crystallite morphology is size dependent; crystallites smaller than 12–15 nm are cuboidal, whereas crystallites larger than 12–15 nm are hexagonal platelets. The procedure affords gram quantities of the smallest available TiB_2 nanocrystallites.
Order No.: JA510-031

Damage Accumulation and Cyclic Fatigue in Mg-PSZ at Hertzian Contacts
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Hertzian contact damage in as-fired, peak-aged and over-aged Mg-PSZ is studied, in single-cycle and multiple-cycle loading. Indentation stress-strain curves reveal a monotonically increasing quasi-plasticity component in the contact deformation with increasing aging time. A bonded-interface technique is used to obtain surface and subsurface views of the damage zones beneath the spherical indenter. Analytical techniques, including optical and scanning electron microscopy, acoustic emission, Raman spectroscopy, and thermal wave imaging, are used to characterize the damage. The damage patterns are fundamentally different in the three aging states: microfracture-dominated in as-fired; tetragonal-monoclinic phase-transformation-dominated in peak-aged; and monoclinic-phase twinning-dominated in over-aged. The damage accumulates with increasing number of cycles, most strongly in the as-fired state. It also increases with increasing test duration in the as-fired and over-aged states, but not perceptibly in the peak-aged. The results imply predominantly mechanical fatigue effects, augmented by a chemical component in the as-fired and over-aged states. Broader implications in relation to the susceptibilities of zirconia ceramics to fatigue degradation in concentrated stress configurations, with special relevance to the evolution of flaws at the microstructural level, are considered.
Order No.: JA510-032

Generation of Amorphous Ceramic Capacitor Coatings on Titanium Using a Continuous Sol-Gel Process
B.G. Dixon, M.A. Walsh III, P.G. Phillips, R.S. Morris
(Cape Cod Research Inc.)

Thin amorphous films of ceramic capacitor materials were successfully deposited using sol-gel chemistry onto titanium wire using a continuous, computer controlled process. By repeatedly depositing and calcining very thin layers of material, smooth and even coats can be produced. Surface analyses revealed the layered nature of these thin coats, as well as the amorphous nature of the ceramic. The electrical properties of the better coatings, all composed of niobium, bismuth, and zinc oxides, were then evaluated.
Order No.: JA510-033

Preferential Orientation and Dielectric/Pyroelectric Properties of Sol-Gel-Derived $Pb(Mg,Zn)_{1/3}Nb_{2/3}O_3$ Thin Films
M.K. Cho, K.S. Kim, H.M. Jang
(Pohang University of Science and Technology [POSTECH])

Thin films of $Pb(Mg,Zn)_{1/3}Nb_{2/3}O_3$ (PMZN) were fabricated by spin casting the partially hydrolyzed Pb-Mg Zn-Nb-O complex alkoxide sols on (111)Pt-coated MgO (100) planes. Analysis of the isothermal transformation data indicated that the growth of perovskite grains from the pyrochlore matrix is a diffusion-controlled process with an initial fast nucleation. The effect of water concentration in the sol on the preferential orientation of perovskite grains in the corresponding thin film was examined. A strong preferential orientation of (100) perovskite was obtained in the PMZN thin film derived from the sol having the molar ratio of H_2O to total metal alkoxides (R_w) of 2. Small-angle x-ray scattering (SAXS) experiment in the Porod region indicated that weakly branched precursor systems led to highly (100)-oriented perovskite grains after thin-film formation. The pyroelectric coefficient and the corresponding figure-of-merit were controlled by suitably varying the degree of preferential orientation during the fabrication of thin films.
Order No.: JA510-034

The Influence of an Electric Field on the Mechanism of Combustion Synthesis of Tungsten Silicides
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(University of California-Davis)

The synthesis of tungsten silicides by self-propagating combustion has been successfully accomplished under the influence of an electric field. Materials with starting compositions ranging from 6 to 30 wt.% Si were investigated by the method of field-activated combustion synthesis (FACS). A threshold field value was required to initiate a self-sustaining wave; the threshold value depended on composition. It was shown that the level of the applied field can influence the mechanism of silicide formation. The silicide W_5Si_3 could only be formed at relatively high field values while WSi_2 can be formed at any field. The effect of the field on the silicide formation is discussed in terms of its role in liquid phase formation.
Order No.: JA510-035

Cu Microcrystals in Sol-Gel Derived Glasses
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Cu^{2+}-containing SiO_2 and $Al_2O_3{\cdot}9SiO_2$ glasses were prepared by the sol-gel method and heated under reducing conditions to precipitate small-sized Cu and Cu_2O crystals. Cu^{2+} ions incorporated in SiO_2 glass were reduced by heating in N_2 to precipitate Cu_2O and in H_2 to precipitate Cu microcrystals with diameters of about 5 to 15 nm. Microcrystalline Cu-precipitated glass showed an optical absorption band at 560 nm and its third-order nonlinear susceptibility was 1.25×10^{-10} esu, which originated from the enhancement by the surface plasmon resonance of Cu-particles. In contrast, Cu^{2+} ions incorporated in $Al_2O_3{\cdot}9SiO_2$ glass remained unchanged after heating in the reducing gas atmospheres.
Order No.: JA510-036

Optical and Electrical Conductivity Investigations of Fe^{3+}-(acrylonitrile-butadiene-styrene) Terpolymer Complex Systems
N.A. Bakr, M.I. Abdel-Hamid
(Mansoura University)

Infrared (IR) and UV-spectra have been obtained for pure and Fe^{3+}-doped acrylonitrile-butadiene-styrene (ABS) terpolymer. IR study revealed the appearance of a new band at about 380 cm^{-1} and a change of the shape and the intensity of the band at 1450–1500 cm^{-1} and 2240 cm^{-1} indicates the structural change on blending due to π-complexes and the complexes of the functional groups with Fe^{3+} forming ion-coordination polymer. The UV spectrum confirms this result. The optical energy for pure and Fe^{3+}-doped ABS is found to be 3.75 eV and 3.55 eV, respectively. The dc and ac electrical conductivities have been investigated. The apparent conductivity of high Fe^{3+}-doped ABS at the ambient temperature is more by about six orders of magnitude greater than that of the undoped film. The activation energy drastically decreases as the wt.% of $FeCl_3$ increases. The enhanced conductivity in the complex film may be attributed to the capacity of the dopant to give additional charge carriers. Transport of charge carriers will occur by hopping of charge carriers between close neighbors across the intervening dielectric, probably via a thermally activated process.
Order No.: JA510-037

Synthesis and Characterization of Polysilane Precursors for Silicon Carbide Fibers
W.R.I. Cranstone, S.M. Bushnell-Watson, J.H. Sharp
(University of Sheffield)

A series of polysilanes was prepared by the alkali metal dechlorination of chlorosilane monomers, in which the overall functionality, F, of the reaction was varied. Starting monomers of functionality f=2.0 and 3.0 were reacted together in various proportions to achieve values of F

of 2.2, 2.35 and 2.5. In addition to varying the functionality of the reaction, three different difunctional monomers, dimethyldichlorosilane (DMDCS), diphenyldichlorosilane (DPDCS) and methylphenyldichlorosilane (MPDCS) and two trifunctional monomers, phenyltrichlorosilane (PTCS) and ethyltrichlorosilane (ETCS), were used. The effect of these changes on the yields of the polysilanes was determined and the products were investigated by the use of thermogravimetry (TG), gel permeation chromatography (GPC) and thermomechanical analysis (TMA). The ability to spin a polysilane fiber was also assessed.
Order No.: JA510-038

The Elastic Interaction Between a Moving Screw Dislocation and a Surface Crack
Y-Z. Tsai, C.T. Hu, S. Lee
(National Tsing Hua University)

A moving screw dislocation near a surface crack was investigated using dislocation modeling. Motion directions parallel (*x*-direction) and perpendicular (*y*-direction) to the crack surface were considered. Due to free surface, the net Burgers vector inside the crack is zero. After obtaining the dislocation distribution in the crack, we calculated the stress field in the medium. Relative to a static screw dislocation, the magnitude of σ_{yz} due to moving screw dislocation decreases with increasing velocity V_x. Generally, the effect of dislocation shielding on fracture is reduced if the velocity V_x increases. The magnitude of the image force of the dislocation also decreases with increasing velocity V_x. The effect of velocity along the *y* direction on the stress intensity factor and image force has the opposite trend to that along the *x* direction. The present result can reduce to a moving dislocation near a semi-infinite crack and a static dislocation near a surface crack.
Order No.: JA510-039

COMMENT AND REPLY

Comment on "Ion Beam Sputter Deposition of $YBa_2Cu_3O_{7-\delta}$ Thin Films" [J. Mat. Res., 8, 3032 (1993)]
A. Gauzzi, D. Pavuna
(Swiss Federal Institute of Technology at Lausanne [EPFL])

In this brief letter we want to comment on several statements published recently in the Journal of Materials Research by our former coworkers Kellett and James (KJ) in the paper entitled "Ion Beam Sputter Deposition of $YBa_2Cu_3O_{7-\delta}$ Thin Films".[1]
Order No.: JA510-040

Reply to Comments of Drs. A. Gauzzi and D. Pavuna
B.J. Kellett*, J.H. James+
*(*University of Cincinnati, +Oxford Instruments)*

We thank Dr. A. Gauzzi and Dr. D. Pavuna for bringing to our attention their comments on our article "Ion Beam Sputter Deposition of $YBa_2Cu_3O_{7-\delta}$ Thin Films," as it gives us the opportunity to clear up some discrepancies, make more explicit some of the results, and correct unintended misunderstandings.
Order No.: JA510-041

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