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ABSTRACTS

COMMUNICATION

Fourteen year old aging study of the effect of thickness on methanol transport in crosslinked poly(methyl methacrylate)

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The effect of thickness on methanol transport in fourteen year old crosslinked poly(methyl methacrylate) was investigated. The samples studied here are of the same primary source as those used by the study made fourteen years earlier. The sample was encapsulated by a plastic bag and maintained in a desiccator at room temperature. Four thicknesses, 0.6, 1.0, 1.5 and 1.9 mm, were examined. Methanol sorption data was fit to a model in which the mass sorption is a combination of case I, case II and anomalous sorption. The diffusion coefficient for case I transport increases with increasing thickness, but the velocity for case II transport has the trend opposite to the diffusion coefficient. The diffusion coefficient for case I transport and the velocity for case II transport exhibit the Arrhenius behavior. The activation energies for case II transport are 18.9, 16.3, 14.6, and 13.4 kcal/mole corresponding to the thicknesses 0.6, 1.0, 1.5, 1.9 mm, respectively. The activation energies for case I transport are 24.7, 24.2, 21.7, and 21.9 kcal/mole corresponding to the thicknesses 0.6, 1.0, 1.5, and 1.9 mm, respec-tively. For thickness 1.5 mm the activation energies for case I and case II transport are 21.7 and 14.6 kcal/mole for this study, and 24.9 and 17.3 kcal/mole.for the study fourteen years ago.

Order No.: JA610-001

ARTICLES

Structural and superconducting properties of melt grown Y-Ba-Cu-O superconductors

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$YBa_2Cu_3O_7$ (123) samples with varying Y_2BaCuO_5 (211) concentrations (0 mol%, 20 mol%, 28 mol%, and 50 mol%) were synthesized by melt growth process. Microstructural characterizations were done using x-ray diffraction (XRD), optical microscopy, scanning electron microscopy and transmission electron microscopy (TEM). It was found that 123 platelet width, crack width between the platelets and 211 particle size decreased systematically with increasing 211 concentration. TEM study showed that there is a critical radius of curvature (rc $\leq$ 0.2 μm–0.3 μm) of the 123/211 interface where defects/contrasts of strain field start to appear and these defects are believed to be responsible for pinning the magnetic flux. Microhardness measurements showed that Vickers hardness (VHN) increases with increasing 211 content. Critical current density (J_c) values obtained from magnetization measurements using a SQUID magnetometer were found to increase in melt grown samples by the addition of 211 content.

Order No.: JA610-002

High resolution transmission electron microscopy study of interface structures and growth defects in epitaxial $Bi_2Sr_2Ca_{n-1}Cu_nO_{4+2n+\delta}$ films on $SrTiO_3$ and $LaAlO_3$

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The defect structure of epitaxial, c-oriented $Bi_2Sr_2Ca_{n-1}$-$Cu_nO_{4+2n+\delta}$ (BSCCO) thin films grown by DC sputtering and layer-by-layer MBE on $SrTiO_3$ and $LaAlO_3$ single crystal substrates was investigated by high resolution transmission electron microscopy (HRTEM). Particular emphasis was put on the structure of the film/substrate interface. The films grown by DC-sputtering show a rather perfect structure involving a regular stacking of the unit cells. In spite of this regularity, there are many defects, such as twins, chemical stacking faults, and precipitates, as well as interfacial dislocations accommodating the film/substrate lattice misfit. The MBE-grown films contain twins and interfacial dislocations, but most prominent are precipitates of various size and rather high number density. Composition and structure of the precipitates were analyzed. Interfacial dislocations were found to be located in the films at a distance of up to 3 nm from the film/substrate interface. The experiments showed that the quality of the film/substrate interface in MBE-grown films is considerably higher with respect to smoothness, sharpness and regularity, if the layer-by-layer MBE process starts with a Sr-O layer instead of a Bi-O layer. This observation is in correspondence to the observed interface structure of the DC-sputtered films, where the first film layer was a Sr-O layer, not a Bi-O layer, in spite of the films being sputtered from a composite target. A structure model of the $Bi_2Sr_2Ca_{n-1}Cu_nO_{4+2n+\delta}$/ (100) $SrTiO_3$ interface is proposed. The prolonged MBE process was shown to imply a chemical interaction between the $SrTiO_3$ substrate and the growing film resulting in the formation of Sr-rich phases in the near-interface substrate regions.

Order No.: JA610-003

Bi-epitaxial grain boundaries in $YBa_2Cu_3O_{7-x}$ thin films prepared by pulsed laser deposition and pulsed organometallic beam epitaxy: Direct comparison of transport properties and grain boundary structure

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A set of 45° [001] bi-epitaxial $YBa_2Cu_3O_{7-x}$ thin film grain boundaries was studied to compare the effects of the microstructure on transport properties. The grain boundaries were made using two different deposition techniques: pulsed laser deposition (PLD) and pulsed organometallic beam epitaxy (POMBE). The transport properties were highly dependent on the specific growth conditions used, resulting in both fully resistive and superconducting grain boundaries. Subsequent microstructural analysis of the measured boundaries showed that both types (superconducting and resistive) meandered on the length scale of hundreds of nanometers. The major structural difference between the boundaries was at the atomic scale where the resistive boundary had a 1 nm wide disordered region. The direct correlation of microstructure to transport properties demonstrates the importance of the atomic scale structure in the resulting transport behavior.

Order No.: JA610-004

Control of meandering grain boundary configurations in $YBa_2Cu_3O_y$ bicrystal thin films based on deposition rate

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Changing the film deposition rate is shown to be one way to influence the meandering configurations of grain boundaries (GBs) formed in $YBa_2Cu_3O_y$ (YBCO) bicrystal thin films. The magnitude and wavelength of the meander in YBCO films deposited at two different rates have been characterized by transmission electron microscopy (TEM) and statistically quantified. It has been found that the meander becomes more uniform and considerably less rough in films deposited at lower rates compared to that observed in films deposited at higher rates. A mechanism for the formation of the meandering GBs is proposed based on heterogeneous nucleation and three-dimensional (3-D) island growth together with overgrowth of the YBCO films across the substrate grain boundary. The different island sizes and tendency for overgrowth induced by changing the film deposition rate are believed to play important roles in controlling the meandering GB configuration. The possible effect of meandering configurations on transport properties are discussed.

Order No.: JA610-005

Peritectic solidification model for Y-system superconductive oxides

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A peritectic solidification growth model considering both solute diffusion in the liquid and interface kinetics is developed for the growth of $YBa_2Cu_3O_{6+x}$ (123) superconductive oxide. The effect of interface kinetics on the growth rate and the rate control process is discussed using this model. The growth rate of the 123 crystal is evaluated as a function of the undercooling using the proper kinetic equation. The evaluated results by this model show good agreement with the experimental results. As a result of this evaluation, the growth of the 123 crystal is predicted to be limited by a mixed control process, in general.

Order No.: JA610-006

Growth and electronic properties of epitaxial TiN thin films on 3C-SiC(001) and 6H-SiC(0001) substrates by reactive magnetron sputtering

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Epitaxial TiN films were grown on cubic (3C) SiC(001) and hexagonal (6H) SiC(0001) substrates by ultra high vacuum reactive magnetron sputtering from a Ti target in a mixed Ar and N_2 discharge at a substrate temperature of 700°C. Cross-sectional transmission electron microscopy including high-resolution imaging showed orientational relationships TiN(001) || 3C-SiC(001); TiN[110] || 3C-SiC[110] and TiN(111) || 6H-SiC(0001); TiN[110],[101] || 6H-SiC[$1\bar{2}10$]. In the latter case, twin-related TiN domains formed as the result of nucleation on SiC terraces with inequivalent stacking sequence of Si and C. The TiN/SiC interface was locally atomically sharp for both SiC polytypes. Defects in the TiN layers consisted of threading double positioning domain boundaries in TiN(111) on 6H-SiC. Stacking faults in 3C-SiC did not propagate upon growth of TiN. Room temperature resistivity of TiN films was $\rho = 14\ \mu\Omega$ cm for 6H-SiC(0001) and $\rho = 17\ \mu\Omega$ cm for 3C-SiC(001) substrates. Specific contact resistance of TiN to 6H-SiC(0001) was $1.3 \times 10^{-3}\ \Omega\ cm^2$ for a 6H-SiC substrate with an n-type doping of $5 \times 10^{17}\ cm^{-3}$.

Order No.: JA610-007

Highly resistive sputtered ZnO films implanted with copper

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Polycrystalline (0001)-oriented thin films of ZnO (thickness 120 nm) were deposited by rf magnetron sputtering and post-deposition annealed at 500°C in oxygen (1 atm.). The films were sub-sequently implanted with copper at doses over the range 10^{16} to 10^{17} ions/cm^2. X-ray diffraction (XRD) indicates the compressive intrinsic film stress is largely relieved by the pre-implantation anneal, and does not change when implanted or when further annealed after implantation, suggesting that the dominant cause of intrinsic stress is the atomic packing density rather than the crystallographic defect density. Resistivity measurements indicate that annealing of pure ZnO films causes the perpendicular resistivity to increase from $1.3 \times 10^5\ \Omega$.cm to $5 \times 10^{10}\ \Omega$.cm. Copper implantation results in a lower resistivity of the order of $10^7\ \Omega$.cm, but subsequent annealing actually increases resistivity beyond that of annealed non-implanted ZnO to $3 \times 10^{12}\ \Omega$.cm. It is proposed that copper increases the resistivity of these annealed films by trapping free electrons with the Cu 3d hole state occurring in CuO (formed predominantly during annealing). In order to check this, the oxidation state of the implanted copper was studied before and after annealing by x-ray photoelectron spectroscopy (XPS) and extended x-ray absorption fine structure (EXAFS). Three oxidation states of copper (Cu^0, Cu^{1+}, Cu^{2+}) are detected in the implanted films, and post-implantation annealing results in oxidation of copper to the Cu^{2+} state, confirming that the presence of CuO in ZnO is associated with increased resistivity.

Order No.: JA610-008

Preparation of EuO films by sol-gel technique

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EuO thin films with the cubic NaCl structure were fabricated by a sol-gel technique. The composition and crystal structure of the films were investigated as a function of firing temperature. The preparation of the stock solution and the coating and firing of the films are described. The firing of the gel film and the reduction of the Eu_2O_3 phase are also discussed.

Order No.: JA610-009

A study of a low copper dental amalgam by analytical transmission electron microscopy

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Analytical transmission electron microscopy was used to study specimens of a low-Cu dental amalgam (Velvalloy), prepared using the "wedge technique." Analysis confirmed that the microstructure consists of a Ag_2Hg_3 (γ_1)/$HgSn_{7-9}$ (γ_2) matrix surrounding unreacted Ag_3Sn (γ) particles. In addition, a hitherto uncharacterized reaction layer of fine grains between Ag_3Sn (γ) and Ag_2Hg_3 (γ_1) is a mixture of Ag_3Sn (γ), Ag-Hg-Sn (β_1), Ag_2Hg_3 (γ_1) and occasionally Cu_6Sn_5 (η'). An Ag-Hg-Sn (β_1) phase was clearly identified for the first time. Since Velvalloy is a simple commercial dental amalgam, it is a reasonable starting point for characterizing more complex dental amalgam microstructures.

Order No.: JA610-010

Circumferential permeability in non-magnetostrictive amorphous wires

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Real and imaginary components of the impedance response on $Co_{68.1}Fe_{4.4}B_{15}Si_{12.5}$ amorphous as cast wires, were measured in the 100Hz-100kHz frequency range and 0.05-30 mA (RMS) current amplitude, at axial dc fields of 0 and 4800 A/m. From these data, plots of circumferential complex permeability as a function of circular field, as well as magnetization curves were derived. Results are analyzed in terms of equivalent circuits, which allows a resolution of domain wall and rotational contributions to the circumferential magnetization processes.

Order No.: JA610-011

Synthesis and characterization of a novel layered titanium phosphate

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A novel metastable layered titanium phosphate has been synthesized by the treatment of layered titanates ($Na_2Ti_3O_7$ and $Na_4Ti_9O_{20}$) with 1–2 M phosphoric acid solution at 120–150°C. Based on the data of ^{31}P MAS NMR and IR spectroscopy, x-ray powder diffraction, thermal and elemental analysis the formula $Ti_2O_3(H_2PO_4)_2{\cdot}2H_2O$ was assigned to the novel compound. The layered nature of the compound was confirmed from n-alkylamine intercalation and the ion exchange behavior towards alkali, alkaline earth and some transition metal ions.

Order No.: JA610-012

Magnetoelastic anisotropy distribution in glass-coated microwires

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Amorphous microwires obtained by the glass-coated melt-spinning method having diameters in the range of micrometers can exhibit perfectly square (single and large Barkhausen jump) or quasi-anhysteretic hysteresis loops depending on the easy magnetization direction determined by the intrinsic magnetoelastic anisotropy. The thermoelastic internal stresses frozen-in during the fabrication that model the domain structure are calculated here by considering the classical theory of elasticity. A complex stress distribution is obtained having the order of magnitude of 10^3 MPa. Circular stresses turn out to be predominant, which arises from the composite nature of the microwire (metallic nucleus and insulating glass coating having different mechanical and thermal properties).

Order No.: JA610-013

Microstructure and magnetic properties in percolating $(Ni\text{-}Fe)_x(SiO_2)_{1-x}$ granular films

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We studied composition, structure, microstructure and magnetic properties of percolating $(Ni\ Fe)_x(SiO_2)_{1-x}$ granular thin films. We found that the magnetic susceptibility increases and the coercivity decreases when increasing x towards x_t, the critical metallic volume fraction for the metal-insulator transition. While the susceptibility decreases and the coercivity increases when increasing annealing temperature for x just below x_t. The comparison of the microstructure and the magnetic properties suggests that the enhanced magnetic susceptibility for x just below x_t is probably associated with the labyrinthine structure of the granular magnetic particles where there is an enhanced surface to volume ratio of the magnetic particles.

Order No.: JA610-014

Sintering and properties of $Li_2O{\cdot}Al_2O_3{\cdot}4SiO_2$-borosilicate glass composites

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Sintering, glass crystallization, microstructure, and the resulting properties of spodumene-borosilicate glass composites were investigated. Densities > 90% could be obtained for samples sintered below 1000°C. Samples that contained more low-viscosity glass densified at lower temperatures, and significant density reduction was observed at higher temperatures. Samples that contained high-viscosity glass densified only at higher temperatures. When glasses of different viscosity were added, the densification of the composites was improved. There was interdiffusion between spodumene and glass. Cristobalite crystallized from the pure glasses. Addition of spodumene changed the major phase crystallized to anorthite or α-quartz as a result of dissolution of spodumene into glass. Composites that contained glass of higher silica content showed more crystallization. As sintering temperatures increased, the amount of crystallization decreased, due to dissolution of glass into spodumene. The spodumene particles showed angular shape with smooth and straight sides. The spodumene-glass composites showed a dielectric constant in the range of 5–6, a loss tangent generally < 1%, and a CTE value in the range of 2–5.6 x 10^{-6} K^{-1}. Co-doping of glasses with different viscosity effectively improved the densification and properties of the composites.

Order No.: JA610-015

Fracture behavior of Al_2O_3/SiC-platelet composites

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Mechanical behavior of hot pressed SiC platelet reinforced alumina composites has been analyzed as a function of SiC platelet content for two different alumina matrix powders. Fracture toughness and flexural strength at temperatures ranging from 25 to 1200°C, R-curve behavior and thermal shock resistance have been determined. Small differences in the impurity content of the starting Al_2O_3 powders strongly determine the microstructure and the mechanical behavior of Al_2O_3/SiC-pl composites. Low alkali content alumina led to composites with large matrix grain size which presented spontaneous microcracking. At high temperature, high viscosity liquid phase is formed that shields cracks enhancing mechanical properties and R-curve behavior. Small amount of impurities reduced Al_2O_3 matrix grain size and avoided spontaneous microcracking. Enhanced fracture toughness (up to 30%) at room temperature, R-curve behavior and thermal shock resistance were achieved for these materials.

Order No.: JA610-016

Mechanical properties and oxidation resistivity of carbon fiber/ceramic composites prepared from borosiloxane

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Diphenylborosiloxane (PBS), an organometallic compound with Si-O-B bonds, was compounded with carbon fiber (CF) to form CF/ceramic composite. Three types of PBS with different molar ratios (Si/B) in the materials were used. On the PBS obtained, specific gravity, Si content, molecular weight, melting point, and infrared absorption spectrum were

measured. On the basis of their results, structures of PBS were examined, which clarified that the PBS thus synthesized consisted of several components with different molecular weights. Mechanical properties and oxidation resistivity of the CF/ceramic composites obtained differed with the Si/B ratios of PBS. Mechanical strength of the CF/ceramic composites increased with increase of Si content of PBS. It was also found that, when PBS with high boron content was used, compact vitreous film consisting mainly of B_2O_3 was formed over the composite. Due to the formation of this film, oxidation resistivity of the composite was improved.

Order No.: JA610-017

Dynamic convection driven-thermal gradient chemical vapor infiltration

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The operation of the process of chemical vapor infiltration using a combination of pressure pulsing and thermal gradients is theoretically investigated in this study. Past studies had shown that pulsing of the pressure in the gas phase can lead to a dramatic reduction of the density gradients in the densifying structure, in comparison to those seen in isobaric diffusion-driven infiltration, with significant gradients present only in the vicinity of the external surface of the preforms. Using a detailed model for chemical vapor infiltration under unsteady nonisothermal conditions, we show that temperature gradients, created in our study through microwave heating, can, in conjunction with pressure pulsing, eliminate the density gradients in the final product. Moreover, appropriate tuning of the operational parameters can lead to a situation where densification proceeds from the interior of the preform towards the external surface.

Order No.: JA610-018

Processing and characterization of $Pb(Zr,Ti)O_3$ films, up to 10 μm thick, produced from a diol sol-gel route

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A recently developed diol sol-gel route has been modified in order to produce multilayer $PbZr_{0.53}Ti_{0.47}O_3$ films on platinized sapphire substrates. Up to 20 depositions of a 1.1M sol were carried out leading to a final film thickness of 10 μm. A similar thickness could be achieved from 12 coatings of a more concentrated 1.6M sol.

Decomposition and crystallization of the multi-layer coatings were performed using a two stage pre-firing sequence, at 350°C and 600°C, followed by a final firing step at 700°C. Ferroelectric remanent polarization increased with increasing film thickness to a value of 40 μC cm^{-2} for a 10 μm film, with a corresponding coercive field of 30 kV cm^{-1}; the relative permittivity of this film was ~1000 and the dissipation factor 0.04. The thickness dependence of relative permittivity could be modeled on a simple series capacitor circuit representing the ferroelectric PZT film and low-permittivity interface layers; but other possible contributory factors are also discussed.

Order No.: JA610-019

Chemical vapor deposition synthesis and characterization of co-deposited silicon-nitrogen-boron materials

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Si-N-B films have been deposited by LPCVD from $SiH_4/B_2H_6/NH_3$ gas mixtures. The influence of the temperature and the composition of the gas mixture on the deposition process and film properties have been investigated. At 1000°C, for the highest ammonia flow rate (SiH_4:B_2H_6:NH_3, 10:25:500), a mixture of turbostratic boron nitride and silicon nitride was deposited. For decreasing ammonia flow rates, the Si-N-B ternary system was formed (1260 cm^{-1} band in the infrared spectra), which co-exists with the unstable turbostratic boron nitride structure. Finally, for a low NH_3 flow rate of 100 sccm, stable amorphous films are obtained. On the other hand, at 800°C, stable films with a high content in the ternary Si-N-B compound were obtained for a wide range of ammonia concentrations (100–500 sccm). At this temperature (800°C), the composition of the films, as measured by Auger and photoelectron spectroscopies, strongly depends on the $[SiH_4]/[B_2H_6]$ ratio in the gas mixture. The improvement in the mechanical and chemical properties of the samples has been associated with the increase in the content of Si-N bonds in the Si-N-B films.

Order No.: JA610-020

Metastable δ-$Bi_{12}SiO_{20}$ and its effect on the quality of grown single crystals of γ-$Bi_{12}SiO_{20}$

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The main results are as follows: (1) Metastable δ-$Bi_{12}SiO_{20}$ may crystallize from the overheated $Bi_{12}SiO_{20}$ melt and transform into stable γ-$Bi_{12}SiO_{20}$ at about 569.5°C during the subsequent slow cooling process. (2) The transition "δ-$Bi_{12}SiO_{20}$ → γ-$Bi_{12}SiO_{20}$" is irreversible and the γ-$Bi_{12}SiO_{20}$ is stable up to the melting temperature. (3) By quenching the $Bi_{12}SiO_{20}$ melt, pure δ-$Bi_{12}SiO_{20}$ can be obtained at room temperature. The quenched δ-$Bi_{12}SiO_{20}$ crystal is nontransparent and has a space group of *Fm3m (225)* and a lattice constant of 5.5417 Å at 20°C. (4) The quenched metastable δ-$Bi_{12}SiO_{20}$ can transform into pure γ-$Bi_{12}SiO_{20}$ at 382.5~386.1°C with an exothermic heat of 31.68~32.38 J/g. The transition-produced γ-$Bi_{12}SiO_{20}$ crystal is still nontransparent and has a large lattice distortion. (5) The transition "δ-$Bi_{12}SiO_{20}$ → γ-$Bi_{12}SiO_{20}$" causes about 6% volume contraction, which may result in cracking of the grown crystal. By controlling the growth parameters, this transition can be effectively avoided.

Order No.: JA610-021

Preparation of zirconia thin films by metalorganic chemical vapor deposition using ultrasonic nebulization

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Zirconia (ZrO_2) thin films were prepared by MOCVD using ultrasonic nebulization with new source materials, $Zr(OBu)_4$, $Zr(OBu)_3(acac)$, $Zr(OBu)_2(acac)_2$, and $Zr(OBu)(acac)_3$. This process is a simple and economic method to prepare oxide thin films. $Zr(OBu)_4$ was successfully reacted with acetylacetone at a molar ratio of 1:3. Polycrystalline thin films were deposited at a substrate temperature range from 300°C to 550°C. The substitution of alkoxy radicals by acetylacetone made the deposition rate higher and insensitive to substrate temperature. The films deposited below 450°C mostly had a monoclinic structure and those deposited above 450°C had a tetragonal structure. The measured optical energy band gap of zirconia film was 5.32 eV.

Order No.: JA610-022

Microstructure and ferroelectric properties of fine-grained $Ba_xSr_{1-x}TiO_3$ thin films prepared by metalorganic decomposition

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Thin films of $Ba_xSr_{1-x}TiO_3$ (x = 0.7, 0.8, 0.9 and 1.0) were prepared by metalorganic decomposition (MOD). The relative permittivity, dissipation, polarization, resistivity, and grain size of these films were studied as a function of composition and temperature. Ferroelectric hysteresis loops were observed for all values of x; and were found to be independent of measurement temperature though strongly dependent upon grain size.

Order No.: JA610-023

Microstructural observation of the ZrC/ZrO_2 interface formed by oxidation of ZrC

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The oxidation of ZrC single crystals having (100) faces was performed at a temperature of 600°C at an oxygen pressure of 2.6 kPa. A polished cross section of oxide scale was observed by backscattered electron imaging (BEI) in a scanning electron microscope (SEM). The oxide scale was observed to consist of two sub scales, zones 1 and 2; zone 1 is adjacent to the ZrC. The thickness of zone 1 was kept constant (about 2 μm) independent of oxidation time. The interfacial microstructure between ZrC and cubic ZrO_2 (c-ZrO_2) phase in zone 1 was observed by high resolution transmission electron microscopy, by using an extremely thin foil of an oxidized crystal. The c-ZrO_2 crystallites of 2 to 10 nm in size showing the (111), (200) and (220) lattice fringes were aggregated and distributed in an area about 10 to 20 nm away from the interface, with an amorphous layer observed adjacent to the ZrC. Electron dispersive x-ray analysis (EDX) indicated that carbon is concentrated at the interface; a decreasing oxygen concentration gradient in the oxide phase and away from the interface in the ZrC suggests the formation of oxygen deficient ZrO_{2-x} and oxycarbide on the respective sides of the interface. A black coating layer appeared, resulting from detachment or dissolution of zone 2, when a crystal oxidized for 1 h was treated in a HF solution. The layer was shown by the Raman spectrum to be amorphous carbon.

Order No.: JA610-024

Numerical analysis of powder behavior in argon-hydrogen and argon-nitrogen radio frequency plasmas: Part I. Thermal plasma treatment of titanium carbide powders

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Modeling of TiC powder behavior in Ar-H_2 and Ar-N_2 RF thermal plasmas has been performed as well as the numerical analysis of the plasma fields to investigate the plasma/powders interaction. The heat transfer rate from the plasma to the powders decreases while increasing the powder feed rate, and with decreasing the plasma pressure, because of the significant local cooling owing to the presence of the injected powders. These results agreed fairly well with the experimental results of our paper. The present modeling would give the guidance for the rational design of new material processing using thermal plasmas.

Order No.: JA610-025

Phase transformation and characterization of TiO_2 and ZrO_2 addition in the Li_2O-Al_2O_3-SiO_2 gels

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Glass-ceramic powders with a composition of $Li_2O{\cdot}Al_2O_3{\cdot}4SiO_2$ (LAS) have been synthesized by the sol-gel technique using $LiOCH_3$, $Al(OC_2H_5)_3$, $Si(OC_2H_5)_4$, $Ti(OC_2H_5)_4$ and $Zr(OC_2H_5)_4$ as starting mate-rials and investigated the phase transformation behavior during cal-cination. Differential thermal analysis (DTA), x-ray diffraction (XRD), and scanning electron microscopy (SEM) were utilized to determine the thermal behavior of the gels. Considering the LAS gels with TiO_2 6.0 wt% and various wt% ZrO_2 content, the peak position of β-spodumene phase formation in DTA curves was shifted to a higher temperature when the ZrO_2 content was increased. The activation energy of β-spodumene crystallization was 283.8 kcal/mol for LAS gels with TiO_2 6.0 wt% and 2.0 wt% ZrO_2. Unlike foregoing studies for LAS gels, during calcination of the dried LASTZ gels from 800°C to 1200°C neither β-eucryptite, nor γ-spodumene was noted to be present. The crystallized phases comprised of β-spodumenees as the major phase and rutile (TiO_2) together with zirconia (ZrO_2) are precipitated as minor phases.

Order No.: JA610-026

Crystal growth and electrical properties of $Li_2B_4O_7$

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Growth of $Li_2B_4O_7$ crystals has been carried out under hydrothermal conditions at relatively low temperature and pressure conditions (T = 250°C, P = 100 bars). A systematic study of electrical measurements have been carried out within a wide range of internal frequency and temperature. The corresponding impedance, Arrhenius and Bode plots are given.

Order No.: JA610-027

Synthesis of cubic diamond in the graphite-magnesium carbonate and graphite-$K_2Mg(CO_3)_2$ systems at high pressure of 9–10 GPa region

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Cubic diamond was synthesized with two systems: (1) graphite with pure magnesium carbonate (magnesite), and (2) graphite with mixed potassium and magnesium carbonate at pressures and temperatures above 9.5 GPa, 1600°C and 9 GPa, 1650°C, respectively. At these conditions: (1) the pure magnesite is solid, whereas (2) the mixed carbonate exists as a melt. In this pressure range, graphite seems to be partially transformed into hexagonal diamond. Measured carbon isotope $\delta^{13}C$ values for all the materials suggest that the origin of the carbon source to form cubic diamond was the initial graphite powder, and not the carbonates.

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Optical fiber drawing temperature of fluorogallate glasses

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The thermal properties and the crystallization behavior of fluorogallate based glasses were analyzed. The kinetic nature of the glass transition was used to determine the temperature dependence of the viscosity and from it an estimation of the appropriate drawing temperature for an optical fiber was established. The crystallization kinetics were studied by using both isothermal and continuous heating regimes. The temperature range for nucleation was evaluated and for samples previously nucleated the activation energy of the growth process was found. The results were used to estimate the empirical nucleation and crystal growth rates from which the time-temperature-transformation curves and the temperature-heating rate-transformation diagrams were constructed. The results obtained agree with experimental data and are discussed in the light of minimizing the volume of crystals formed during fiber drawing.

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Characterization of CeO_2-Al_2O_3-SiO_2 glasses by infrared and x-ray absorption near edge structure spectroscopies

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The structural characteristics of CeO_2-Al_2O_3-SiO_2 glasses have been studied by infrared and XANES spectroscopies. The Kramers-Kronig analysis of IR reflection data are performed. Results of IR spectra show that ceria is the network modifier and favors the formation of [SiO_4] tetrahedron with non-bridging oxygens. The high frequency Si-O-Si stretching vibrational peak near 1100 cm^{-1} shifts toward the lower frequency as ceria content increases and alumina content is constant. The XANES data of Cerium LIII edge show that the majorities of cerium ions in cerium aluminosilicate glasses are Ce^{3+} ions. The ratio of Ce^{4+}/Ce^{3+} increases with increasing the ratio of Ce/(Al+Si). However, the ratio of Ce^{4+}/Ce^{3+} decreases with increasing the ratio of Al/(Al+Si).

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The relationship between glass structure and poling-induced optical second harmonic intensity for ZnO-TeO_2 glasses

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Local arrangement around Te and Zn atoms of ZnO-TeO_2 glass systems are investigated by Te K and Zn K EXAFS using synchrotron radiation. Tellurium K EXAFS results suggest the coordination states of tellurium atoms are changed from TeO_4 trigonal bipyramids to TeO_{3+1} polyhedra or TeO_3 trigonal pyramids with increasing ZnO. Zinc K EXAFS results indicate that Zn atom has the second oxygen shell which locates at the distance of 0.222-0.288 nm. The second harmonic intensity of the poled glass samples was also measured. The compositional dependence of the second harmonic intensity could be explained in terms of the structural change of ZnO-TeO_2 glass with the addition of ZnO.

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Study of electrical and optical properties of ion-implanted polymers in relation to carbon structure

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The relationship between electrical conductivity, optical properties and carbon structure of ion-implanted polymers has been studied by optical transmission and Raman spectroscopy. The electrical conductivity, which depends not only on the ion dose but also on the dose rate in a complicated manner, was found to have a simple relation to the optical absorbance. By Raman spectroscopic analyses, the difference in the electrical conductivity was explained in terms of the difference in carbon structure.

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Effects of sequential He^+ and Ar^+ implantation on surface properties of polymers

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Three important polymers: polystyrene (PS), poly ether ether ketone (PEEK) and polyimide Kapton, were irradiated separately with 1 MeV He^+, 1 MeV Ar^+, and 1 MeV He^+ followed by 1 MeV Ar^+ sequentially, to a fluence of 3×10^{19} ions/m^2 for each ion. The specimens were characterized for changes in surface hardness using a nanoindentation technique, and wear resistance using a reciprocating sliding wear apparatus with a steel ball counterface. Results indicated that while all polymers showed higher hardness values after ion irradiation, the dual irradiation resulted in the largest hardness increase, greater than for the single ion irradiated specimens. Wear test results also indicated that the dual $He^+ + Ar^+$ irradiation resulted in the best improvement in wear resistance of the polymers. These improvements in properties are a consequence of crosslinking of the polymer material caused by the ion irradiation. Linear energy transfer considerations showed that the dual $He^+ + Ar^+$ implantation was better because it combined a deeper implant, in the form of He, along with Ar irradiation which resulted in a shallower but more highly crosslinked layer at the near-surface. Thus a deeper and graded crosslinked surface region was formed. The study shows that there is greater flexibility for tailoring surface properties of polymers by using a judicious combination of ion species, ion energies and fluences.

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