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ABSTRACTS

RAPID COMMUNICATIONS

Epitaxial growth of semiconducting LaVO₃ thin filmsW. Choi,¹ T. Sands,¹ K.-Y. Kim²*(¹University of California, ²LG Electronics)*

Epitaxial thin films of LaVO₃ were grown on (001) LaAlO₃ substrates by pulsed laser deposition from a LaVO₄ target in a vacuum ambient at substrate temperatures ≥ 500 °C. X-ray diffraction studies showed that epitaxial LaVO₃ films consist of mixed domains of [110] and [001] orientations. Thermoprobe and four-probe conductivity measurements demonstrated the *p*-type semiconducting behavior of the epitaxial LaVO₃ films. The temperature dependence of the conductivity is consistent with a thermally activated hopping mechanism with an activation barrier of 0.16 eV.

Order No.: JA001-001

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Phase selection during solidification of undercooled bulk Bi₉₅Sb₅ melts

Z. Zhou, J. Zhao, W. Wang

(Chinese Academy of Sciences)

Relationships between melt heat treatment and undercooling of alloy melts were clarified with Bi₉₅Sb₅ through four-factor, three-level orthogonal experiments. The results show that the cooling rate plays the most important role in the undercooling of Bi₉₅Sb₅ alloy melts. Undercooling as large as 121 K was obtained in bulk Bi₉₅Sb₅ alloy melt. It is presently the highest undercooling of this alloy system. Metastable phase with tetragonal structure was found in Bi₉₅Sb₅ alloy with undercooling of 121 K.

Order No.: JA001-002

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Hydrogen-enhanced dislocation velocities in Ni₃Al single crystals

C.B. Jiang, S. Patu, Q.Z. Lei, C.X. Shi

(Chinese Academy of Sciences)

The average dislocation velocity in hydrogenated Ni₃Al single crystals was directly measured as a function of resolved shear stress (RSS) at room temperature (293 K) by the etch-pit technique. It was found that the dislocation velocity with hydrogen is about 5–25 times faster than that without hydrogen for the same RSS, and hydrogen decreases activation energy for dislocation motion in Ni₃Al single crystals. The reason hydrogen can enhance dislocation velocity in this compound is briefly discussed. These preliminary results quantitatively provide first evidence of hydrogen enhancing dislocation mobility in Ni₃Al material.

Order No.: JA001-003

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Fabrication of cellular NiTi intermetallic compoundsB.-Y. Li,¹ L.-J. Rong,¹ Y.-Y. Li,¹ V.E. Gjunter²*(¹Chinese Academy of Sciences, ²V.D. Kuznetsov Siberian Physical Technical Institute)*

Self-propagating high-temperature synthesis (SHS) has been successfully developed for the fabrication of cellular NiTi intermetallic compounds, which have an open cellular structure with about 60 vol% porosity and more than 95% open-porosity ratio. The SHS reactions lead to the formation of TiNi, Ti₂Ni, Ni₃Ti, and Ni₄Ti₃ intermetallics. The SHS process can be controlled by regulating the preheating temperature, which has effects on the phase amount and the shape as well as macrodistribution of pores in the products.

Order No.: JA001-004

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Significant reduction in thermal expansion of a low-carbon steel sheet by depositing a Fe–Ni alloy thin film

S.S. Kim, Y.B. Park

(Sunchon National University)

A 3- μ m-thick Fe–Ni alloy thin film was deposited on a 0.2-mm-thick low-carbon steel substrate by radio-frequency magnetron sputtering, and the properties of the film were investigated. The film exhibits a columnar growth structure and a fiber texture with the relationship of (110)_{film}//(111)_{substrate}. A thermal cycle test showed good adhesion between the film and the substrate. Most importantly, the Fe–Ni alloy deposition results in a significant reduction in thermal expansion of the whole specimen. These results suggest that a properly deposited Fe–Ni alloy film could be applied to a low-carbon steel sheet to significantly reduce its thermal expansion. Such reduced thermal expansion would enhance the image resolution of a shadow mask made of a low-carbon steel sheet.

Order No.: JA001-005

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Fracture origins in LiNbO₃ wafers due to postprocessing micro-repolarization

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In the process of developing electro-optic devices from ferroelectric z-cut LiNbO₃ wafers, a repolarization throughout the wafer thickness occurs due to a localization of electric charges on the wafer. The repolarization not only generates microdomains causing light to scatter but also large defects in the crystal that become the origin of wafer fracture. The size of such defects is comparable to the wafer thickness (0.5 mm), and an anomaly in the chemical and crystalline structures can be found in them. X-ray diffractometry and x-ray photoelectron spectroscopy confirm that a chemical reduction in the defective region occurs.

Order No.: JA001-006

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Optical and electrical properties of transparent conducting In₂O₃–ZrO₂ filmsS.B. Qadri,¹ H. Kim,² H.R. Khan,³ A. Piqué,¹ J.S. Horwitz,¹ D. Chrisey,¹E.F. Skelton¹*(¹U.S. Naval research Laboratory, ²George Washington University,**³Forschungsinstitut für Edelmetalle und Metallchemie)*

The optical transparencies and electrical conductivities of thin films of In₂O₃ mixed with ZrO₂ have been investigated. These films were deposited on glass substrates at room temperature using pulsed-laser deposition. Indium–zirconium oxide films with a ZrO₂ content up to 15 wt% were conducting and more than 80% transparent from 450 to 700 nm. As the ZrO₂ content increased from 0 to 15 wt%, the electrical resistivities increased from 1.28×10^{-3} to 6.48×10^{-2} Ω cm; the carrier densities were decreased from 2.14×10^{20} to 1.0×10^{18} /cm³; and the Hall mobilities decreased from 21 to 5 cm² V⁻¹ s⁻¹, all monotonically.

Order No.: JA001-007

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Diamond synthesis by high-velocity thermal spray:**The laboratory analogue of meteorite impact**

R. Goswami, H. Herman, S. Sampath, J.B. Parise

(State University of New York at Stony Brook)

Nanocrystalline-diamond particles were produced in the form of a coating by depositing Ni-clad graphite powder in a high-velocity thermal spray experiment. Particles were accelerated to impact and form a thick film (>20 μ m)

on a steel substrate, with the high-velocity impact generating a shock wave, which propagates through the particle and the underlying deposits. Transmission electron microscopy revealed that this deposit contains cubic diamond nanocrystals having a size range of 5 to 10 nm in graphite. In addition to diamond, it was observed that a portion of the deposit contains "closed-curved graphite."

Order No.: JA001-008

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Synthesis of crack-free thick diamond wafer by step-down control of deposition temperature

J.-K. Lee,¹ Y.-J. Park,¹ K.-Y. Eun,¹ Y.-J. Baik,¹ J.-W. Park²¹Korea Institute of Science and Technology, ²Hanyang University

Due to growth tensile stress, which evolves in diamond films during deposition, thick diamond films are easily cracked. In this study we successfully prevented growth cracks by introducing thermal compressive stress with step-down control of deposition temperatures during growth. Three deposition temperature drops of 10 °C each during deposition enabled us to successfully synthesize crack-free four-inch diamond wafers several hundred micrometers in thickness. This method is very simple and may be applicable to coating of films of various materials different from those of substrates.

Order No.: JA001-009

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ARTICLES

Fabrication and characterization of large Nd–Ba–Cu–O grains prepared under low oxygen pressure

W. Lo, N. Hari Babu, D.A. Cardwell, Y. Shi, D.M. Astill

(University of Cambridge)

A large, single-grain Nd–Ba–Cu–O (NdBCO) composite consisting of superconducting NdBa₂Cu₃O_{7- δ} containing nonsuperconducting Nd₄Ba₂Cu₂O₁₀ phase inclusions was fabricated up to 2 cm in diameter using a top-seeded melt-textured growth technique. A MgO single-crystal seed was used to provide a heterogeneous nucleation site at the center of a presintered pellet heated above its peritectic temperature and cooled continuously in a conventional tube furnace in reduced oxygen partial pressure. This process produces individual grains with the *c* axis oriented at $\approx 10^\circ$ to the seed surface which, from vibrating sample magnetization measurements, exhibit a pronounced peak effect in their magnetic moment over a wide temperature range (50–90 K) when the supercurrent flows in the *a*-*b* planes. A very high irreversibility field (>9 T at 77 K) is also observed in these grains for field applied both perpendicular and parallel to the crystallographic *c* axis, which is significantly greater than that observed in good-quality melt-processed Y–Ba–Cu–O. These results underline the potential of NdBCO for high field engineering applications.

Order No.: JA001-010

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Lattice parameters and thermal expansion of GaN

R.R. Reeber, K. Wang

(North Carolina State University)

Neutron powder diffraction methods with Rietveld analysis are utilized to determine GaN lattice parameters from 15 to 298.1 K. Using these measurements and literature data, we calculated the thermal expansion of gallium nitride (GaN) and predicted its higher temperature thermal expansion. The results are compared with available experimental data and earlier work.

Order No.: JA001-011

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X-ray diffraction and transmission electron microscopy analysis of ordering and structure in Al_{1-x}In_xAs thin films

R.L. Forrest, J. Kulik, T.D. Golding, S.C. Moss

(University of Houston)

This paper presents an x-ray diffraction and transmission electron microscopy analysis of Al_{1-x}In_xAs grown by molecular beam epitaxy. Two samples grown on (001) InP at temperatures of 370 and 400 °C are characterized. The first, which contains a high density of twin lamellae, exhibits triple-period short-range ordering with a rather short correlation range normal to the (111) planes. Within these (individual) planes, the concentration, however, is uniform over a considerably greater distance, leading to a highly anisotropic scattering. This is the first observation of triple-period short-range ordering in a sample that exhibits 2 × 1 surface reconstruction. The second sample exhibits CuPt-type short-range ordering with scattering that is significantly streaked, suggestive of lamellar-shaped ordered domains. Both samples contain high densities of stacking faults leading to additional sharp streaking along symmetry-allowed (111) directions.

Order No.: JA001-012

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Icosahedral nanophases in the Al–Mn–Ce system

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(National Institute for Physics of Materials)

Nano-icosahedral phases were prepared by melt-spinning in the AlMnCe system (Al ≥ 89 at.%). Ce-induced structure alterations are reported by x-ray diffraction for the icosahedral (i) AlMnCe phase. They comprise apparition of superstructure diffraction lines, due to chemical ordering in the 6-dimensional icosahedral lattice, which gradually changes from a simple to a face-centered (fci) type. Also, strong anomalies of diffracted intensities, with reference to model fci phases, support Ce preferential location on a special set of sites.

Microstructure investigations show a nanodispersion of i-AlMnCe in an Al-rich matrix, suggesting a low nucleation barrier of this metastable phase in the melt.

Order No.: JA001-013

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Microalloyed synthesis of NiAl containing Ti and B

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The effect of alloying additions of Ti and B on the process of microalloyed synthesis of NiAl and on the microstructure of the synthesized alloy was examined. It was observed that the combustibility of the quaternary alloy is good despite the presence of the alloying elements because of an additional combustion reaction between Ti and B. The microstructure of the quaternary alloy was found to consist primarily of the NiAl and Ti boride phases. The effect of preheating of the specimen prior to synthesis on the process of synthesis was also examined. It was observed that preheating not only can change the morphology of the phases but also influence the nature of the phases present in the alloy. The mechanism of the formation of the two-phase microstructure during the synthesis from the elemental powders was established by stopping the combustion front and by carrying out a detailed microstructural characterization of regions around the stopped combustion front.

Order No.: JA001-014

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Nondestructive evaluation of cavitation in an Al–Mg material deformed under creep conditions

E.M. Taleff,¹ T. Leon-Salamanca,² R. Ketcham,¹ R. Reyes,¹ W.D. Carlson¹¹The University of Texas at Austin, ²Reinhart and Associates, Inc.)

Cavitation was examined in an Al–Mg solid-solution alloy deformed in tension at 400 °C under conditions providing solute-drag creep, which can produce tensile ductilities from 100% to over 300%. Two nondestructive evaluation techniques were employed to measure the extent of cavitation: ultra-high-resolution x-ray computed tomography and pulse-echo ultrasonic evaluation. Subsequent to nondestructive evaluation, the sample was sectioned for examination by standard metallographic techniques. Metallographic examination confirmed that both nondestructive techniques accurately indicated the extent of cavitation. Ultrasonic testing provided a practical means of distinguishing material with cavities from that without cavities. Ultra-high-resolution x-ray computed tomography provided an accurate three-dimensional image of internal cavitation.

Order No.: JA001-015

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The effect of microstructure on the thermal properties of an aluminum casting alloy

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The photoacoustic technique was used to determine the thermal conductivity of some representative sample obtained from an aluminum casting alloy. This material was solidified with an imposed unidirectional thermal gradient to obtain samples with different microstructures characterized by the secondary dendrite arm spacing, which increases as the solidification rate decreases. It was found that the thermal conductivity of the alloy decreases with an increase in the secondary dendrite arm spacing and a decrease in the integral dendrite perimeter.

Order No.: JA001-016

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Pyrocarbon anisotropy as measured by electron diffraction and polarized light

X. Bourrat,¹ B. Trouvat,¹ G. Limousin,¹ G. Vignoles,¹ F. Doux²¹Laboratoire des Composites Thermostructuraux, ²SNECMA)

This work deals with the measurement of pyrocarbon anisotropy on very thin fiber coatings used to control the interfacial behavior in carbon/carbon composites. Differentiation of the various pyrocarbons was performed through computerized image analysis of the electron diffraction patterns by measuring

the azimuth opening of the carbon 002 diffraction arcs. This orientation angle decreases when the texture switches from rough- to smooth-laminar. Relationship with a polarized light measurement technique at a lower resolution is discussed.

Order No.: JA001-017

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Influence of reduction mechanism on the morphology of cobalt nanoparticles in silica-gel matrix

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(¹Bengal Engineering College, ²Jadavpur University)

Cobalt-chloride- and dextrose-containing silica gels were reduced *in situ* under nitrogen atmosphere in the temperature range of 600 to 950 °C. Analysis of kinetic data on the *in situ* reduction shows that, in the temperature range of 600 to 750 °C, contracting geometry type and, in the temperature range of 800 to 950 °C, nucleation and growth type of mechanism remain operative. The shape and size of the reduced cobalt nanoparticles in the silica matrix was studied by examining the transmission electron micrographs of the reduced Co/SiO₂ samples. The morphology of the reduced metallic particles was found to be influenced by the change in reduction mechanism.

Order No.: JA001-018

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Fracture mode of alumina/silicon carbide nanocomposites

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Computer simulations have been designed to elucidate the evolution of microcracking in a nanocomposite using appropriate material values for alumina and silicon carbide. These are compared to a single-phase material using elastic and thermal expansion coefficients for alumina. It is found that the region and the fracture mode where microcracking ensues are determined by the intensity and the length scale of the residual stress fields, which interact. Of specific interest are the region, fracture mode, and length of ensuing microcracks for materials with different inclusion locations (at the grain boundary or within the grain) and with different grain size to inclusion size ratios.

Order No.: JA001-019

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Domain structure and electrical properties of highly textured PbZr_xTi_{1-x}O₃ thin films grown on LaNiO₃ electrode-buffered Si by metalorganic chemical vapor deposition

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(¹University of Illinois at Urbana-Champaign, ²National Tsing Hua University)

Thin films of highly (100) textured fine-grain (lateral grain size \approx 0.1 to 0.15 μ m) PbZr_xTi_{1-x}O₃ (PZT) ($x = 0$ to 0.7) were grown on conductive perovskite LaNiO₃-buffered platinumized Si substrates by metalorganic chemical vapor deposition. Domain configuration and crystalline orientation were studied using x-ray diffraction and transmission electron microscopy. The predominant domain boundaries of Ti-rich tetragonal-phase PZT and Zr-rich rhombohedral-phase PZT were found to be on the (110) planes and (100) planes, respectively. The equilibrium domain widths were observed and estimated numerically based on transformation strain, grain size, and domain boundary energy. The peak value of the dielectric constant was 790 near the morphotropic boundary. Hysteresis behavior of these PZT thin films was demonstrated. A decrease in coercive field with the increment of Zr content was found; this variation was attributed to domain density and the multiplicity of polarization axes. Furthermore, the low leakage current ($J \leq 5 \times 10^{-7}$ A/cm² at $V = 4$ V) was observed for all samples, and the involvement of several possible conduction mechanisms was suggested.

Order No.: JA001-020

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Dielectric ceramics in BaO-Sm₂O₃-TiO₂-Ta₂O₅ quaternary system

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(Zhejiang University)

Dielectric ceramics in the BaO-Sm₂O₃-TiO₂-Ta₂O₅ quaternary system were prepared and characterized for five typical compositions: BaSm₅Ti₇Ta₃O₃₀, Ba₂Sm₄Ti₆Ta₄O₃₀, Ba₃Sm₃Ti₅Ta₅O₃₀, Ba₄Sm₂Ti₄Ta₆O₃₀, and Ba₅SmTi₃Ta₇O₃₀. The latter three compositions tended to form the tungsten-bronze phase, and the ceramics based on these compositions had a high dielectric constant (134–175) and a low dielectric loss (on the order of 10⁻³) but a larger temperature coefficient of the dielectric constant. Meanwhile, the former two compositions generally had a more complex phase constitution, containing the tungsten-bronze phase combined with some unknown phase, and a relatively small temperature coefficient of the dielectric constant in the temperature range of 20 to 85 °C could be achieved in these two compositions.

Order No.: JA001-021

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Orientation-dependent properties of silicon nitride with aligned reinforcing grains

D-S. Park, M-J. Choi, T-W. Roh, H-D. Kim, B-D. Han

(Korea Institute of Machinery and Materials)

Silicon nitride with the aligned reinforcing grains was prepared by tape casting with addition of the silicon nitride whiskers and gas-pressure sintering at 2148 K. The microstructure and the mechanical properties of the sintered sample, including the fracture toughness and the three-point flexural strength, were highly anisotropic. Both the fracture toughness and the flexural strength were the highest when the crack-propagation direction was normal to the alignment direction. This result was interpreted with respect to the laminate composite materials. Although the large elongated grains were as long as 44.4 \pm 12 μ m and as wide as 5.1 \pm 0.67 μ m, they were not the fracture origins.

Order No.: JA001-022

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Joining of silicon nitride ceramics for high-temperature applications

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²Chinese Academy of Sciences)

A refractory silicon nitride joint, which contains β -Si₃N₄ grains and grain boundary amorphous phase in the joined layer, was developed with the aid of a ceramic adhesive based on the system Si₃N₄-Y₂O₃-SiO₂-Al₂O₃. The similarity in chemistry and microstructure between the parent ceramic and the joint zone indicates that the joining mechanism is the same as that involved in the sintering of Si₃N₄. The resultant joint exhibits a high bond strength of 550 MPa at 25 °C and retains a strength of 332 MPa at 1000 °C. Post-joining hot-isostatic pressing was applied to strengthen the joint, resulting in increased strengths of 668 MPa at room temperature and 464 MPa at 1000 °C.

Order No.: JA001-023

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Slow crack-growth behavior of alumina ceramics

M.E. Ebrahimi, J. Chevalier, G. Fantozzi

(GEMPPM)

The fracture behavior of high-purity alumina ceramics with grain sizes ranging from 2 to 13 μ m is studied by means of the double torsion method. Crack-propagation tests conducted in air, water, and silicon oil, for crack velocities from 10⁻⁷ to 10⁻² m/s, show that slow crack growth is due to stress corrosion by water molecules. An increase of the grain size leads to enhanced crack resistance, which is indicated by a shift of the V-K_I (crack velocity versus applied stress intensity factor) plot towards high values of K_I. Moreover, the slope of the curve is apparently higher for coarse grain alumina. However, if the R-curve effect is subtracted from the experimental results, a unique V-K_{I,tip} (crack velocity versus stress intensity factor at the crack tip) law is obtained for all alumina ceramics, independent of the grain size. This means that the crack-growth mechanism (stress corrosion by water molecules) is the same and that the apparent change of the V-K_I law with grain size is a direct effect of crack bridging.

Order No.: JA001-024

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Nanoindentation and adhesion of sol-gel-derived hard coatings on polyester

C.M. Chan, G.Z. Cao, H. Fong, M. Sarikaya, T. Robinson, L. Nelson

(University of Washington, Korry Electronics, Co.)

We investigated sol-gel-derived silica-based hard coatings on modified polyester substrates. The silica network was modified by incorporating an organic component and adding transition metal oxides. These modifications resulted in tailored thermal, optical, and mechanical properties of the coatings. Various low-temperature densification techniques were studied including sol-preparation procedure, enhanced solvent evaporation, ultraviolet irradiation, and low-temperature heating (below 150 °C). Oxygen plasma etching was applied to improve the adhesion of the sol-gel coatings on the plastic surface. Nanoindentation analysis revealed that the coatings have a surface hardness up to 2.5 \pm 0.27 GPa and an elastic modulus up to 13.6 \pm 0.4 GPa, approximately an order of magnitude higher than that of the plastic surface.

Order No.: JA001-025

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Water-based Si₃N₄ suspensions: Part I. Effect of processing routes on the surface chemistry and particle interactions

F. Bertoni,¹ C. Galassi,¹ S. Ardizzone,² C.L. Bianchi²

(¹IRTEC-CNR, ²University of Milan)

Si₃N₄ powders manufactured by two different preparative routes were characterized for the solid-liquid interfacial reactivity and surface composition. Three mixing processes were tried to investigate the modifications of silicon

nitride particle surface in aqueous suspensions. The surfaces of the starting powders and the dried mixed powders were investigated by x-ray photoelectron spectroscopy to determine the nature and ratios of surface groups. Electroacoustic measurements show that no change occurs in the isoelectric point for the mixed Si_3N_4 powders while the milling/mixing process has a great influence on the zeta potential magnitude and particle size distribution.

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Water-based Si_3N_4 suspensions: Part II. Effect of wet mixing/milling processes on the addition of the sintering aids

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Three mixing processes were used to introduce lanthanum oxide and yttrium oxide in silicon nitride suspensions. X-ray photoelectron spectroscopy (XPS) analysis was used to identify the surface composition and investigate the surface coverage by the added oxides. XPS results evidenced the protective effect against hydrolysis and oxidation of the coated layer. Electroacoustic measurements showed that the milling/mixing process and solids loading contents had a great influence on the isoelectric point (IEP) and on the dispersing degree for suspensions with sintering aids. With the change of solids load content and mixing energy, the IEP of mixed powders suspensions reflected a surface behavior dominated by the additives oxides. The attrition mixing and the ultrasonication resulted in the more efficient processing routes to distribute the sintering aids on the starting Si_3N_4 powders, especially at high solids volume fraction.

Order No.: JA001-027 © 1999 MRS

Ultrafine MnFe_2O_4 powder preparation by combusting the coprecipitate with and without Mg^{2+} or Zn^{2+} additives

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Ultrafine MnFe_2O_4 powder with its crystallites less than 100 nm was prepared using a combustion process. The coprecipitates containing the stoichiometric amount of Mn^{2+} and Fe^{3+} to form MnFe_2O_4 were prepared by dissolving the required metallic nitrates in de-ionized water and adding NH_4OH to adjust the pH of the solutions to 9. The collected dried precipitates were then heated up to predetermined temperatures and then quickly contacted with the acetone spray. Upon contacting with the heated precipitates, the acetone spray was ignited. The combustion of acetone caused the precipitates to form crystalline MnFe_2O_4 without chemical segregation. The crystallinity of MnFe_2O_4 powder so obtained depended on the ignition temperature of acetone spray. MnFe_2O_4 powder obtained at acetone ignition temperature of 773 K had higher crystallinity than that obtained at acetone ignition temperature of 523 K. The presence of a small amount of Mg^{2+} or Zn^{2+} in the composition of the coprecipitates promoted the mobility of constituent ions of the combusted powder and resulted in bigger MnFe_2O_4 crystallites at a lower acetone ignition temperature.

Order No.: JA001-028 © 1999 MRS

Role of organic solvents and surface-active agents in the sol-emulsion-gel synthesis of spherical alumina powders

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Spherical alumina particles were prepared following the sol-emulsion-gel method by systematic variation of (i) the concentration of the surfactant Span 80 above or below the critical micelle concentration (CMC) in different organic solvents and (ii) hydrophilic-lipophilic balance (HLB) of different surfactants in cyclohexane. The experimentally determined CMC of Span 80 was found to increase with increasing dielectric constant of the organic solvent, influencing the sol droplet and alumina particle size. With an increase in the HLB value of the surfactants, the tendency of monodispersed sphere formation among the particles increased with a decrease in the size distribution and average particle size (d_{50}).

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Calculation of metastable immiscibility region in the Al_2O_3 - SiO_2 system using molecular dynamics simulation

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The metastable immiscibility region in the Al_2O_3 - SiO_2 system was calculated by conventional thermodynamic equations using thermodynamic parameters obtained from molecular dynamics simulation. The calculated

miscibility gap has a consolute temperature of around 1500 °C at the critical composition of about 20 mol% Al_2O_3 and spreads more widely toward the Al_2O_3 -rich composition side than the SiO_2 -rich side. The calculated miscibility gap in this study showed a fair agreement with that reported by Ban *et al.* [T. Ban, S. Hayashi, A. Yasumori, and K. Okada, *J. Mater. Res.* **11**, 1421 (1996)] calculated by a regular solution model, but the present calculated region is somewhat narrower in the Al_2O_3 -rich composition side than that reported by Ban *et al.*

Order No.: JA001-030 © 1999 MRS

Optimization of Ta-Si-N thin films for use as oxidation-resistant diffusion barriers

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We have demonstrated that the optimum Ta-Si-N compositions for use as oxygen diffusion barriers in stacked-capacitor dynamic random-access memory structures with perovskite dielectrics are in the range Ta(20–25 at.%)–Si(20–45 at.%)–N(35–60 at.%). Twenty-two different Ta-Si-N compositions were evaluated, starting from six sputter-deposited Ta-Si alloys of which four were reactively deposited in 2–8% nitrogen in an argon plasma. The barriers were evaluated after an aggressive 650 °C/30 min oxygen anneal to determine if they remained electrically conductive, prevented oxygen diffusion and formation of low dielectric constant oxides, and had minimal interaction with the Pt electrode and underlying Si plug. Rutherford backscattering spectroscopy, four-point probe sheet resistance, through-film-resistance, and x-ray diffraction analysis techniques were used in the evaluation.

Order No.: JA001-031 © 1999 MRS

Microstructural and electrical property studies of $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ films grown on p-InP (100) substrates by a radio-frequency magnetron sputtering technique at low temperature

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$\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ thin films were grown on p-InP (100) substrates by using radio-frequency magnetron sputtering at a relatively low temperature (~450 °C). X-ray diffraction measurements showed that the $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ film layers grown on the InP substrates were polycrystalline, and Auger electron spectroscopy measurements indicated that the compositions of the as-grown films consisted of lead, zirconium, titanium, and oxygen. Transmission electron microscopy measurements showed that the grown $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ was a polycrystalline layer with small domains and that the $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3/\text{InP}$ (100) heterointerface had no significant interdiffusion problem. Room-temperature current-voltage and capacitance-voltage (C-V) measurements clearly revealed a metal-insulator-semiconductor behavior for the $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ insulator gates, and the interface state densities at the $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3/\text{p-InP}$ interfaces, as determined from the C-V measurements, were approximately 10^{11} eV⁻¹cm⁻² at an energy of about 0.6 eV below the conduction-band edge. The dielectric constant of the $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ thin film, as determined from the C-V measurements, was as large as 907.2. These results indicate that the $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ layers grown on p-InP (100) substrates at low temperatures hold promise for potential high-density nonvolatile memories and high-speed infrared sensors based on InP substrates.

Order No.: JA001-032 © 1999 MRS

Adhesion and reliability of copper interconnects with Ta and TaN barrier layers

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With the advent of copper metallization in interconnect structures, new barrier layers are required to prevent copper diffusion into adjacent dielectrics and the underlying silicon. In addition, the barrier must also provide adequate adhesion to both the dielectric and copper. While Ta and TaN barrier layers have been incorporated for these purposes in copper metallization schemes, little quantitative data exists on their adhesive properties. In this study, the critical interface fracture energy and the subcritical debonding behavior of ion-metal-plasma sputtered Ta and TaN barrier layers in Cu interconnect structures were investigated. Specifically, the effects of interfacial chemistry, Cu layer thickness, and oxide type were examined. Behavior is rationalized in terms of relevant reactions at the barrier/dielectric interface and plasticity in adjacent metal layers.

Order No.: JA001-033 © 1999 MRS

Electron field emission from polycrystalline diamond films

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The influence of the carbon network structure of polycrystalline diamond films that were prepared from a mixture of H_2 , CH_4 , and N_2 using microwave-enhanced plasma chemical vapor deposition on electron field emission has been systematically investigated. With increasing nitrogen gas flow ratio of $[N_2]/[H_2 + CH_4 + N_2]$, the film hardness and surface roughness of the as-grown films decreased, and the concentration ratio of amorphous sp^2 -bonded carbon clusters and mixed sp^2 - sp^3 carbon structures to tetrahedrally bonded amorphous carbon phases increased. Correspondingly, the turn-on voltage for electron emission decreased. After the surface post-treatment by pure hydrogen plasma exposure, the concentration ratio was clearly found to have increased dramatically and the turn-on voltage significantly decreased for films produced at small nitrogen flow ratio. Our results suggest that the influence of the concentration ratio on electron field emission is much more significant than that of the surface roughness of the polycrystalline diamond films studied in this paper.

Order No.: JA001-034

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Structural and physical properties of Fe_2O_3 -doped lead vanadate glass

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The role of Fe_2O_3 in the structural and physical properties of ternary lead vanadium iron glass system has been studied in comparison with the binary lead vanadate glasses. X-ray diffraction, scanning electron microscopy, and differential thermal analysis show that homogeneous glasses of composition $10Fe_2O_3 \cdot xV_2O_5 \cdot (90-x)PbO$ can be obtained for $x = 50$ to 80 mol%. Observation from the infrared spectroscopy shows that the basic building blocks of these glasses are same as those of crystalline V_2O_5 , while differential thermal analysis and electrical conduction of these glasses suggest that there is a strong role of iron, both in the glass network and in the conduction mechanism for the glasses containing a low percentage of vanadium.

Order No.: JA001-035

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The effect of crystallization on microwave dielectric properties of stoichiometric cordierite glasses containing B_2O_3 and P_2O_5

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The effect of crystallization on microwave dielectric properties of cordierite glasses containing B_2O_3 and P_2O_5 additions was investigated. Two glasses containing 5 wt% B_2O_3 /5 wt% P_2O_5 and 7.5 wt% B_2O_3 /7.5 wt% P_2O_5 were studied. Both glasses were sintered to nearly full density at temperatures as low as 860 °C. The frequency constant and quality factor of α cordierite are, respectively, about five times and two times larger than those of glassy phase and μ cordierite. The temperature coefficients of resonant frequency are estimated to be about -13, -55, and -15 ppm/°C for glassy phase, μ cordierite, and α cordierite, respectively. As a result of the microwave dielectric properties of the individual phase, cordierite glasses containing α cordierite possess best microwave properties.

Order No.: JA001-036

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Effects of plasma power on the properties of low-k polymerlike organic thin films deposited by plasma-enhanced chemical vapor deposition using the toluene precursor

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Effects of plasma power on the properties of polymerlike organic thin films deposited by plasma-enhanced chemical vapor deposition using the toluene precursor were studied. As the plasma power was increased from 5 to 60 W, the relative dielectric constant increased from 2.53 to 2.85. The film deposited at higher plasma power showed higher thermal stability. The film deposited at 60 W was stable up to 400 °C. All the films were insulating under applied field ≤ 1 MV/cm.

Order No.: JA001-037

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Mechanical properties of biocompatible protein polymer thin films

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(University of Michigan)

A silk-like protein with fibronectin functionality (SLPF) (ProNectin F[®], Protein Polymer Technologies, Inc.) is a genetically engineered protein polymer containing structural and biofunctional segments. The mechanical properties and deformation mechanism of electrostatically deposited SLPF thin films were examined by scratch testing, tensile testing, and nanoindentation. Scanning electron microscopy and scanned probe microscopy revealed that the macroscopic properties were a sensitive function of microstructure. The SLPF films were relatively brittle in tension, with typical elongation-to-break values of 3%. Nanoindentation data were fit to a power law relationship.

Order No.: JA001-038

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Effect of recrystallization on the oxidative stability of poly(butylene terephthalate)

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The oxidative stability of poly(butylene terephthalate) is investigated over the range 175 to 220 °C by isothermal differential calorimetry. It is shown that this property is affected by a fast recrystallization process, which takes place between 200 and 220 °C, with a maximum around 208 °C. In fact, recrystallization produces an increase of the crystallinity degree of the polymer, which enhances its resistance to oxidation, as proven by the longer oxidation times detected.

Order No.: JA001-039

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The effect of NbC porosity on reaction-layer microstructure in NbC/Si diffusion couples

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Further experimental observations have allowed us to refine and confirm some aspects of our recently proposed mechanism for reactive diffusion between Si single crystal and NbC powder compact, particularly regarding the prediction of Si as the dominant diffusing species and the nature of the dependence of SiC particle morphology on the presence of voids in the NbC end member. In Si/NbC diffusion couples annealed at either 1300 or 1350 °C, a two-phase NbSi₂ + SiC reaction layer formed. Although NbSi₂ was the matrix in all of the reaction layers, the SiC phase morphology depended upon NbC porosity: when high-porosity NbC was used, SiC was present as discontinuous particles greater than 1 μ m across, while when low-porosity or void-free NbC was used, SiC grew cooperatively with NbSi₂ in the form of lamellae less than 0.5 μ m thick. We propose that this difference arises from the effect of voids both as nucleation sites for SiC particles and as channels for unrestricted SiC growth. Marker experiments conclusively show that Si is the dominant diffusing species in the reaction layer.

Order No.: JA001-040

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Deformation of catalytically grown carbon nanotubes induced by annealing under high pressure

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A study of the structural transformation of catalytically grown carbon nanotubes induced by annealing under high pressure is presented in this paper, in which the atomic details of the microstructural transformations have been monitored mainly with electron microscopy. The microstructural change from the multi-walled carbon nanotubes into quasi-spherical onion becomes obvious just at 770 °C under 5.5 Gpa. The nanotubes deform and almost transform into nanographite ribbons directly when the annealing temperature is above 950 °C under 5.5 Gpa. It is suggested that the pressure and temperature play an important role in the structural transformations of multi-walled carbon nanotubes described here.

Order No.: JA001-041

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