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TABLE OF CONTENTS

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ABSTRACTS

COMMUNICATIONS

Materials in nanopipes of undoped GaN
J. Kang*+, T. Ogawa+
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The nanopipes in undoped GaN epilayers grown on sapphire substrates were investigated by field emission high-resolution electron microscopy (HREM) and energy-dispersive x-ray spectrometry (EDS). In the HREM images, the cores of the nanopipes appeared disordered in the thin regions and more ordered in the thicker regions, indicating the amorphous layer on the surface has a significant influence on the visible image of the nanopipe in the thin regions. The EDS spectra showed that composition of the materials in nanopipes was mainly oxygen, carbon, and gallium elements. The results suggest that the nanopipes are related to impurities.
Order No.: JA901-001 **© 1998 MRS**

Preparation of conducting film composed of polyaniline and metal oxide by sol-gel method
T. Hori, N. Kuramoto, H. Tagaya, M. Karasu, J. Kadokawa, K. Chiba
(Yamagata University)

Conducting thin films were prepared by entrapping water suspended polyaniline into a silica matrix by a sol-gel route. Without metal oxide, the conductivity of the film decreased after heat treatment. However, in the presence of metal oxides such as TiO_2 and Al_2O_3 the conductivity increased after heat treatment at 85 °C and reached 17 Scm^{-1}. The conductivity of the film depended on the kinds and amounts of metal oxides and the temperature of heat treatment.
Order No.: JA901-002 **© 1998 MRS**

A high specific strength, deformation processed scandium-titanium composite
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#Kulicke & Soffa Industries, Inc.)

A 59% Sc–4l % Ti deformation processed metal metal composite was produced by rolling to a true strain of 2.3 at 873 K followed by cold rolling to a total true strain of 3.6. Rolling reduced the original eutectoid microstructure to lamellae of α–Sc and α–Ti with average lamellar thicknesses of l50 nm (Sc) and 120 nm (Ti). The cold rolled material had an ultimate tensile strength of 942 MPa and a specific strength of 259 J/g. The Sc matrix was oriented with the <0001> tilted 22° from the sheet normal direction toward the rolling direction, an unusual texture for an HCP metal with a low c/a ratio, which suggests Sc may deform primarily by basal slip.
Order No.: JA901-003 **© 1998 MRS**

Expedient route to volatile zirconium metal-organic chemical vapor depostion precursors using amide synthons and implementation in yttria-stabilized zirconia film growth
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This communication reports rapid, efficient syntheses of the zirconium-organic MOCVD precursors $Zr(acac)_4$ and $Zr(dpm)_4$ (acac = acetylacetonate; dpm = dipivaloylmethanate) as well as a new, highly volatile, air- and moisture-stable Zr precursor based on a tetradentate Schiff-base ligand, $Zr(tfacen)_2$ (tfacen = bis-trifluoroacetylacetone-

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ethylenediiminate). The improved one-step synthetic routes employ tetrakis(dimethylamido)zirconium as a common intermediate and represent a major advance over previous methods employing $ZrCl_4$ or diketonate metathesis. Furthermore, $Zr(tfacen)_2$ is shown to be an effective metal-organic precursor for the MOCVD-mediated growth of (l00) oriented yttria-stabilized zirconia thin films.

Order No.: JA901-004 **© 1998 MRS**

ARTICLES

Phase relations and superconducting properties of the Y–Ni–B–C system

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The influence of composition and high-temperature heat treatment on phase content and superconducting properties of the YNi_2B_2C phase was investigated. Phase relations in those parts of the Y–Ni–B–C quaternary phase diagram, which are relevant for the YNi_2B_2C intermetallic phase formation were revealed by x-ray diffraction, optical and scanning electron microscopy, and high-temperature differential thermoanalysis. A widespread interval of superconducting transition temperatures T_c = 10.4–15.2 K and small transition width < 0.3 K were determined from samples of different nominal compositions after high-temperature annealing. The different intrinsic properties are ascribed to composition variations of the YNi_2B_2C phase and related to structure parameters, residual resistance ratios, and element concentrations determined by the electron probe microanalysis.

Order No.: JA901-005 **© 1998 MRS**

Microstructural investigation of low temperature chemical vapor deposited 3C–SiC/Si thin films using single-source precursors

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Transmission electron microscopy (TEM) was utilized to investigate microstructures of heteroepitaxial SiC/Si films, grown from single-source precursors such as tetramethylsilane [TMS, $Si(CH_3)_4$], hexamethyldisilane [HMDS, $Si_2(CH_3)_6$], and 1,3-disilabutane [1,3-DSB, $H_3SiCH_2SiH_2CH_3$]. In the case of TMS/H_2 and HMDS/H_2 samples, SiC/Si films grown at relatively high precursor concentration and/or low temperatures showed columnar grains with a high degree of epitaxial relationship with the Si substrate. Higher quality films with larger grains were observed in the case of high temperature and/or low precursor concentration samples, although high density of interfacial voids were observed. Samples grown from pure 1,3-DSB at a low pressure showed high quality single crystalline films with few interfacial voids. It was suggested that the microstructural behavior of these films closely resembles that of the SiC films formed during the carbonization of Si surfaces by the pyrolysis of hydrocarbons, in which the nucleation rate of the film at the initial stage plays a key role. The improvement achieved during the 1,3-DSB growth is proposed to be due to the low growth pressure and the 1:1 ratio of Si and C associated with this precursor.

Order No.: JA901-006 **© 1998 MRS**

Preparation of boron and phosphorus-doped SiC:H films using electron cyclotron resonance chemical vapor deposition: Some effects of microwave power

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Hydrogenated silicon carbide films (SiC:H) were deposited using the electron cyclotron resonance chemical vapor deposition (ECR-CVD) technique from a mixture of methane, silane, and hydrogen, and using diborane and phosphine as doping gases. The effects of changes in the microwave power on the deposition rate and optical bandgap were investigated, and variations in the photo- and dark-conductivities and activation energy were studied in conjunction with film analysis using the Raman scattering technique. In the case of boron-doped samples, the conductivity increased rapidly to a maximum followed by rapid reduction at high microwave powers. The ratio of the photo- to dark-conductivity (σ_{ph}/σ_d) peaked at microwave power of ~600 W. Under conditions of high microwave power, Raman scattering analysis showed evidence of the formation and increase in the silicon microcrystalline and diamondlike phases in the films, the former of which could account for the rapid increase and the latter the subsequent decrease in the conductivity. In the case of phosphorus-doped SiC:H samples, it was found that increase in the microwave power has the effect of enhancing the formation of the silicon microcrystalline phase in the films which occurred in correspondence to a rapid increase in the conductivity and reduction in the activation energy. The conductivity increase stabilized in samples deposited at microwave powers exceeding 500 W probably as a result of dopant saturation. Results from Raman scattering measurements also showed that phosphorus doping has the effect of enhancing the formation of the silicon microcrystals in the film whereas the presence of boron has the effect of preserving the amorphous structure.

Order No.: JA901-007 **© 1998 MRS**

Investigation of crystal growth on (111) InSb thin films to produce high performance Hall elements

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The crystal growth of InSb thin films on mica substrates was investigated by conventional three temperature vacuum evaporation with varied Sb/In flux ratios and temperature programming for the substrate. The Sb/In flux ratio was varied from higher than 1.0 (about 2.0 is optimum), to less than 1.0 (about 0.7 is optimum), to again much higher than 1.0 during the stages of evaporation. The electromagnetic characteristics were investigated, and x-ray analysis of the films at various stages was undertaken. The films obtained contained no excess In and they were (111) highly oriented in x-ray analysis, showing high electron mobility. These films were used to prepare high performance Hall elements.

Order No.: JA901-008 **© 1998 MRS**

Thermal and thermomechanical effects on defect evolution in an Al–Li superplastic alloy

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A commercial Al–Li alloy, in which the superplastic microstructure is developed by "strain-assisted continuous recrystallization" in the early stages of flow, was studied using positron annihilation lifetime spectroscopy. Results revealed that exposing the material to a temperature of 525 °C (optimal temperature of superplastic deformation for this alloy) led to agglomeration of single vacancies into vacancy clusters of size of approximately four vacancies. Evidence for superplastic strain-induced cavitation was not found at 450 °C up to an elongation of 432% and at 525 °C up to 341% the initial strain rate of deformation in both cases being 1.0×10^{-3} s^{-1}. These results have practical significance.

Order No.: JA901-009 **© 1998 MRS**

Matrix laminate composites: Realizable approximations for the effective moduli of piezoelectric dispersions

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(Princeton University)

This paper is concerned with the effective piezoelectric moduli of a special class of dispersions called matrix laminate composites that are known to possess extremal elastic and dielectric moduli. It is assumed that the matrix material is an isotropic dielectric, and the inclusions and composites are transversely isotropic piezoelectrics that share the same axis of symmetry. The exact expressions for the effective coefficients of such structures are obtained. They can be used to approximate the effective

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properties of any transversely isotropic dispersion. The advantages of our approximations are that they are (i) realizable, i.e. correspond to specific microstructures; (ii) analytical and easy to compute even in non-degenerate cases; (iii) valid for the entire range of phase volume fractions; and (iv) characterized by two free parameters that allow one to "tune" the approximation and describe a variety of microstructures. The new approximations are compared with known ones.

Order No.: JA901-010 **© 1998 MRS**

Abrasion of Al_2O_3–SiC–(Al,Si) composites made by melt oxidation

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(Indian Institute of Science)

We report here the remarkable abrasion wear resistance (normal pressure: 2.92 to 5.42 MPa, sliding speed: 0.25 to 3 m/s) of a soft (relative to other structural ceramics) melt oxidized ceramic matrix composite Al_2O_3–SiC–(Al,Si). Inspite of being half as hard and more porous than zirconia toughened alumina (ZTA), a common cutting tool material, this composite fabricated at l l00 °C shows wear resistance comparable to that of ZTA at sliding speeds more than 2 m/s. The ability of this composite to generate a metal rich tribofilm when rubbed and its high thermal shock resistance are factors which may be responsible for this unique performance.

Order No.: JA901-011 **© 1998 MRS**

Effects of Si content on the microstructure and tensile strength of an *in situ* Al/Mg_2Si composite

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(Chinese Academy Sciences)

Al/Mg_2Si composites were *in situ* fabricated by the usual die-casting technique, and effects of the Si contents in the composites on microstructures and tensile strengths were investigated. Experimental results show that extra Si contents in Al/Mg_2Si composites induce a ductile matrix and a uniform distribution of *in situ* particles. The refined microstructures lead to an obvious increase in both strength and ductility of the MMCs. The effects of extra Si on both solidification process and fracture characteristics of the Al/Mg_2Si composites were analyzed.

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Microstructure and mechanical properties of NiAl/Al_2O_3 composites

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(National Cheng-Kung University)

To improve mechanical properties of NiAl, a method for making NiAl matrix composites containing oxide ceramics is introduced. The method involves oxidation of NiAl powder in air to form a thin and uniform oxide scale, mainly Al_2O_3, on the NiAl particles. The Al_2O_3 contents increase with increasing oxidation temperature. The NiAl/Al_2O_3 composites are then formed by hot-pressing the oxidized NiAl powder under vacuum atmosphere. Al_2O_3 inhibits the grain growth of NiAl during the hot-pressing. The residual stress and the Ni-rich NiAl composition exist in the hot-pressed NiAl/Al_2O_3 composites. Strength and toughness data on NiAl/Al_2O_3 composites indicate that the use of oxidation of NiAl powder is a viable technique for improving these properties over that of monolithic NiAl.

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Phase transitions of $(1-x)PbZrO_3 + x(Na_{1/2}Bi_{1/2})TiO_3$ $(0.01 \le x \le 0.15)$ solid solutions

J-K. Lee, H-J. Youn, K.S. Hong
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Morphotropic phase boundaries and temperature dependent phase transitions of $(1-x)PbZrO_3 + x(Na_{1/2}Bi_{1/2})TiO_3$ $(0.01 \le x \le 0.15)$ solid solutions were investigated by x-ray diffraction, differential scanning calorimetry (DSC), and dielectric property analysis. Two morphotropic phase transitions at room temperature were found at $x = 0.1$ and 0.13, which were from anti-ferroelectric orthorhombic (with 4 x 4 x 2 superlattice [orthorhombic(I)]) to anti-ferroelectric orthorhombic (with 2 x 2 x 2 superlattice [orthorhombic(II)]) and from orthorhombic(II) to ferroelectric rhombohedral, respectively. With increasing temperature, the samples with $0.01 \le x \le 0.1$ showed two phase transitions, i.e. from orthorhombic(I) to orthorhombic(II) and from orthorhombic(II) to cubic. The other samples had only one phase transition with increasing temperature. Phase transition temperatures of all the samples were measured using DSC and a phase diagram for the solid solutions was constructed. A model illustrating the anti-parallel shift of Pb ions in the orthorhombic(II) phase was also proposed.

Order No.: JA901-014 **© 1998 MRS**

Annealing effects on phase transformation and powder microstructure of nanocrystalline zirconia polymorphs

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Nanocrystalline zirconia powders in pure form and doped with yttria and calcia were prepared by precipitation method. In the as-prepared condition, all the doped samples show only monoclinic phase, independent of the dopants and dopant concentration. On annealing the powders at 400 °C and above, in the case of 3 and 6 mol% Y_2O_3 stabilized ZrO_2 (3YSZ & 6YSZ) the monoclinic phase transforms to tetragonal and cubic phases respectively whereas in 3 and 6 mol% CaO stabilized ZrO_2 (3CSZ & 6CSZ), the volume percentage of the monoclinic phase gradually decreases up to the annealing temperature of about 1000 °C and then increases for higher annealing temperatures. The presence of monoclinic phase in the as-prepared samples of doped zirconia has been attributed to the lattice strain effect which results in the less symmetric lattice. For the annealing temperatures below 1000 °C, the phenomenon of partial stabilization of the tetragonal phase in 3CSZ and 6CSZ can be explained in terms of the grain size effect. High resolution transmission electron microscopy (HRTEM) observations reveal the lattice strain structure in the as-prepared materials. The particles are found to be a tightly bound aggregate of small crystallites with average size of l0 nm. The morphology of the particles is observed to be dependent on the dopants and dopant concentration.

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The effect of TiO_2 addition on the thermal behavior of sol-gel derived β–spodumene powders

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The effect of TiO_2 addition on the crystallization and phase transformation process in $Li_2O \cdot Al_2O_3 \cdot 4SiO_2$ gels with various TiO_2 contents was investigated using differential thermal analysis, x-ray diffraction, and transmission electron microscopy. The activation energy increased from 98.2 to 184.6 kcal/mol as the TiO_2 content rose from 2.0 to 8.0 wt%. The crystallization sequence and phase transformation were similar in LAS gels with various wt% of TiO_2 additions, except in the case of a 2.0 wt% TiO_2 content. During calcination from 800 to 1200 °C, crystallization of the β–spodumene phase progressed with increasing temperature, and a minor crystalline phase, rutile, also appeared.

Order No.: JA901-016 **© 1998 MRS**

Electromechanical coupling and piezoelectric coefficients of La-modified $(Pb(Mg_{1/3}Nb_{2/3})O_3)_{0.65}$–$(PbTiO_3)_{0.35}$

S.M. Gupta, J.F. Li, D. Viehland
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The electromechanical and piezoelectric properties of La-modified $(0 < y < 0.10)$ $(Pb(Mg_{1/3}Nb_{2/3})O_3)_{0.65}$–$(PbTiO_3)_{0.35}$ (PLMN–PT l00*y*/65/35) were studied for use as transducer materials. The optimum values of the planar coupling coefficient ($K_p = 0.66$) and transverse electromechanical coefficient ($K_{31} = 0.37$) for this crystalline solution were observed for the composition 2/65/35. With increasing y for $y > 0.02$, K_p and K_{31} decreased significantly, as the sample could not sustain a remanent state on removal of an electric field. However, for $y > 0.04$, under external DC bias field, the values of the electromechanical coefficients increased and approached the

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values for 2/65/35. Temperature dependent studies under DC bias for 5/65/35 revealed that the electromechanical coefficients decreased sharply on heating above the Vogel-Fulcher freezing temperature T_f.
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Microwave dielectric properties and applications of rare-earth aluminates
S-Y. Cho*, I-T. Kim+, K.S. Hong*
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Rare-earth aluminates, $LnAlO_3$ (Ln = Dy, Er, Gd, La, Nd, Pr, Sm, and Y) were prepared using the mixed oxide method and their microwave dielectric properties were examined at X-band. Most rare-earth aluminates have suitable permittivities and quality factors for applications as dielectric resonators but a modification of τ_f is necessary due to the coefficient's large negative value. When considering dielectric properties and lattice matching, $YAlO_3$, rather than $LaAlO_3$, was suggested as a promising substrate material for microstrip antennas utilizing high-temperature superconductor thin films. Rare-earth aluminates with a rhombohedral structure exhibited larger permittivities than those with an orthorhombic structure. This difference was attributed to the difference in ionic size and coordination number. It was demonstrated that a non-zero magnetic susceptibility of rare-earth aluminates has an adverse effect on their quality factor. An abrupt variation in the temperature coefficient of permittivity was discussed in terms of oxygen octahedra tilting.
Order No.: JA901-018 © 1998 MRS

Grain boundary effects in NTC–PTC composite thermistor materials
D.J. Wang, J. Qiu, Y.C. Guo, Z.L. Gui, L.T. Li
(Tsinghua University)

Yttrium doped $(Sr_{0.45}Pb_{0.55})TiO_3$ ceramics have been studied by complex impedance analysis. As a sort of NTC–PTC composite thermistor, it exhibited a significantly large negative temperature coefficient of resistivity below T_c in addition to the ordinary PTC characteristics above T_c. It is found that the NTC effect in NTC–PTC materials was not originated from the deep energy level of donor (bulk behavior), but from the electrical behavior of the grain boundary. Therefore, the NTC–PTC composite effect was assumed to be a grain boundary effect and yttrium was a donor at shallow energy level. The NTC–PTC ceramics were grain boundary controlled materials.
Order No.: JA901-019 © 1998 MRS

Preparation and characterization of compounds in the $BaBiO_3$–$Ba(Ce_{1-x}Gd_x)O_{3-x/2}$ system
R. Mukundan, P.K. Davies, W.L. Worrell
(University of Pennsylvania)

The structure, non-stoichiometry, and electrical conductivity of compositions in the $BaBiO_3$–$Ba(Ce_{1-x}Gd_x)O_{3-x/2}$ system have been investigated in an attempt to prepare new mixed (ionic-electronic) conducting oxides. The substitution of Bi into $Ba(Ce_{1-x}Gd_x)O_{3-x/2}$ decreases the concentration of oxygen-ion vacancies, and the effective negative charge of the Gd dopant is compensated by the mixed valence of Bi (3+, 5+). For low Bi contents a decrease in ionic conductivity decreases the overall conductivity; however, higher levels of Bi introduce significant electronic conductivity, and for $Ba(Bi_{0.5}Ce_{0.5})O_3$, $\sigma_{total} \approx 1$ S/cm at 800 °C in air. Compositions in the $Ba(Bi_{0.5}Ce_{0.5-x}Gd_x)O_3$ pseudobinary system undergo a B-cation order-disorder transformation at 1300–1350 °C for $x = 0.5$ and at ≈1250 °C for $x = 0.4$; all other compositions retain a disordered B-site arrangement. While these disordered perovskites exhibit oxygen non-stoichiometry under reducing conditions at elevated temperatures, with the extent of reduction decreasing with increasing Gd content, their ordered counterparts remain close to stoichiometry. The electronic conductivities of this pseudobinary could be fitted to a "band-type" model, and, despite the presence of oxygen vacancies for the lower values of x, no significant ionic conductivity was observed.
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Determination of domain structure and abundance of epitaxial $Pb(Zr,Ti)O_3$ thin films grown on MgO(001) by rf magnetron sputtering
K.S. Lee, Y.M. Kang, S. Baik
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Epitaxial $Pb(Zr_xTi_{1-x})O_3$ ($x = 0.0 \sim 0.32$) ferroelectric thin films of 500 nm thickness were grown on MgO(001) single crystal substrates by *in situ* rf magnetron sputtering, and evolution of their domain structures are characterized by employing various x-ray diffraction techniques. X-ray θ-2θ scan showed the films were grown highly *c* axis oriented with a tetragonal perovskite structure. 90° domain configuration was investigated using the x-ray rocking curve analysis for PZT *100* peaks in two different φ angles. The rocking curve analysis showed that the degree of *c* axis orientation and the crystalline quality of the films were improved continuously with increasing Zr concentration. The *c* domain abundance as a function of Zr concentration was quantified using the x-ray rocking curves of PZT 001 and 100 taking account of structural factors and Lorentz-polarization factors. High temperature x-ray technique was also employed to quantify the domain structure as a function of temperature during cooling after reheating the samples to 650 °C. During the cooling process, *c* domain abundance was found to increase continuously while the crystalline quality of the films was deteriorated below the Curie temperature. The results led us to conclude that the transformation strain of the film at and below the Curie temperature plays a significant role in the final domain structure and abundance of epitaxial PZT thin films.
Order No.: JA901-021 © 1998 MRS

Effect of the species of substituted ion on ferroelastic domain switching of rare earth ion doped ZrO_2 pseudo-single crystals
T. Kiguchi, A. Saiki, K. Shinozaki, N. Mizutani
(Tokyo Institute of Technology)

The differences between the domain switching amounts of 3 mol% R_2O_3–ZrO_2 (R = Yb, Y, Dy, Gd, Eu, Sm) pseudo-single crystals with additive cation species were investigated from the microstructural aspect. The switching amounts of Yb, Y, Dy, and Ed substituted ZrO_2 were three times higher than those of Eu and Sm. The amounts were corresponded to the volume fraction of t'-phase, and they indicated that phase separation was proceeded especially in Eu and Sm substituted ZrO_2.
Order No.: JA901-022 © 1998 MRS

Determination of creep behavior of thermal barrier coatings under laser imposed high thermal and stress gradient conditions
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A laser sintering/creep technique has been established to determine the creep behavior of thermal barrier coatings under steady-state high heat flux conditions. For a plasma sprayed zirconia 8 wt.% yttria coating, a significant primary creep strain and a low apparent creep activation energy were observed. Possible creep mechanisms involved include stress induced mechanical sliding and temperature and stress enhanced cation diffusion through the splat and grain boundaries. The elastic modulus evolution, stress response, and total accumulated creep strain variation across the ceramic coating are simulated using a finite difference approach. The modeled creep response is consistent with experimental observations.
Order No.: JA901-023 © 1998 MRS

Sintering of cubic boron nitride without additives at 7.7 GPa and above 2000 °C
T. Taniguchi, M. Akaishi, S. Yamaoka
(National Institute for Research in Inorganic Materials)

The sintering behavior of cBN powder with various particle sizes from 0.5 to 12 μm was investigated when sintered at temperatures from 1500 to 2500 °C and pressure of 7.7 GPa without additives. Above 2000 °C, translucent sintered bodies were obtained. Microstructure observation indicated that the optimum sintering temperature was near 2350 °C for fine powders of 0.5 to 1.2 μm and 2 to 4 μm and slightly higher than

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2350 °C for powders from 8 to 12μm. The fracture toughness of the well-sintered bodies decreased with grain growth above the optimum sintering temperature.

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Nucleation control for hot-working of silicon oxynitride based ceramics

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An attempt was made to develop an engineering ceramic plastically-deformable at high temperatures with low flow stress at high strain rates and without strain hardening. Dense ceramic preforms were fabricated by pressureless sintering Si_3N_4 + SiO_2 mixed powders with an addition of $MgAl_2O_4$ at 1500 °C. A transient liquid, which occurs during the reaction sintering of Si_2N_2O, was utilized for subsequent net-shape forming. The ceramic (6 ϕ x 6 mm column) was deformed without any cracks and cavities in compression tests at high strain rates (10^{-2} ~ $10^{-3}s^{-1}$) at 1500 °C, but this was not achieved in a test at lower strain rates for a long time, because of the growth of elongated Si_2N_2O grains during the test. Potassium fluoride (KF) was used as a dopant for retardation of nucleation of Si_2N_2O during sintering and hot-working. The KF-doped preforms were successfully plastically deformed even in the test for a long time.

Order No.: JA901-025 **© 1998 MRS**

Effect of β–Si_3N_4 seed crystal on the microstructure and mechanical properties of sintered reaction-bonded silicon nitride

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β–Si_3N_4 seed crystal has been synthesized from α–Si_3N_4 powder. Reaction-bonded Si_3N_4/SiC composite has been fabricated with β-Si_3N_4 seed crystals. The nitridation behavior and the changes in mechanical properties resulting from the addition of seed crystals were studied before and after gas pressure sintering. Addition of seeds showed a considerable improvement in the nitridation, resulting in an increase in fracture strength of the composite. Highly nitrided RBSN as a result of the addition of seed crystals gave rise to high strength of composite after post sintering. Fracture toughness of the seeded Si_3N_4 was also improved up to 35% compared to the baseline Si_3N_4. Micrographs showed that the seeded Si_3N_4 developed a bimodal microstructure which resulted in an improvement in fracture toughness.

Order No.: JA901-026 **© 1998 MRS**

Selective gas sensing properties of surface ruthenated tin oxide

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(National Chemical Laboratory)

Gas sensing properties of a novel surface functionalized tin oxide material have been studied to demonstrate the possibility of selectivity control by surface state formation. Covalent anchoring of ruthenium oxide on tin oxide surface (ruthenated tin oxide) is found to give considerable enhancement in sensitivity (320) as well as selectivity to 1000 ppm of LPG at 300 °C compared to the sensitivity (4) of pure tin oxide samples. The amount and distribution of grafted ruthenium oxide on the surface of tin oxide seems to be the most important parameter controlling the change in electrical transport with LPG gas adsorption.

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Processing of silicon carbide ceramics using chemically modified polycarbosilanes

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Chemically modified polycarbosilane (PC) which contains Si–Al–C–O component, PCOAl, was synthesized using PC and aluminum triisopropoxide. Ceramic yield was greatly improved through the modification of PC with a metal alkoxide. The phase transformation behavior and microstructure development of silicon carbide (SiC) were studied on β–SiC powders coated with chemically modified PC. The β–α phase transformation of SiC was enhanced by the coating of chemically modified PC on β–SiC powder. A unique microstructure with submicron-sized plate-like grains was developed, since the fine α phase produced at low temperature served as a nucleation site for β–α phase transformation of SiC.

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Effect of TiO_2 addition on the preparation of β–spodumene powders by sol-gel process

M-C. Wang*, M-H. Lin*, H-S. Liu+
*(*National Kaohsiung Institute of Technology, +National Cheng-Kung University)*

This study has shown the possibility of achieving two primary considerations for the advanced fabrication of spodumene with a composition of $Li_2O \cdot Al_2O_3 \cdot 4SiO_2 \cdot nTiO_2$ (LAST) glass-ceramics by a sol-gel process, namely: an enormous reduction of sintering temperature from 1600 to 1200 °C together with the appreance of simples phases of β–spodumene/rutile as opposed to products via the conventional melting-crystallization process. Fine glass-ceramic powders with a composition of $Li_2O \cdot Al_2O_3 \cdot 4SiO_2$ (LAS) have been synthesized by the sol-gel process using $Si(OC_2H_5)_4$, $Al(OC_2H_5)_3$, $LiOCH_3$, and $Ti(OC_2H_5)_4$ as the starting materials. The process included well controlled hydrolysis polycondensation of the raw alkoxides. X-ray diffraction (XRD), Fourier transform infrared spectrocopy (FT-IR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and electron diffraction (ED) analysis were utilized to study the effect of TiO_2 addition on the preparation of β-spodumene powders by sol-gel process. The gelation time of the LAST solution increases at longer time as the TiO_2 content increased. For the low (< 3) or high (>11) pH value, the gelation time was shorten. At pH = 5, regardless of the TiO_2 content, the gel has the longest time of gelation. When the dried gels of the LAST system are heated from 800 to 1200 °C, the crystallized samples are composed of the major phase of β–spodumene and a minor phase of rutile (TiO_2).

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Phase formation and composition of Mn–Zn ferrite powders prepared by hydrothermal method

W-H. Lin, S-K. Jang Jean, C-S. Hwang
(National Cheng Kung University)

Mn–Zn ferrite powders were prepared by hydrothermally aging the coprecipitates of compositional metal ions using ammonium hydroxide as a precipitant. R value (alkalinity) = (moles of added OH^-)/[(moles of added Zn^{2+}) x 2 + (moles of added Mn^{2+}) x 2 + (moles of added Fe^{3+}) x 3] was introduced to adjust the amount of added ammonia. The results show that the R value of starting suspension and hydrothermal time have similar and dominant effects on the composition, spinel ratio, and crystallite size of synthesized powders. From the analyses of XRD and ICP, it notes that no α–Fe_2O_3 peak in the XRD patterns of powders synthesized at R = 2 ~ 3, 150 °C x 2 h, may be due to lower degree of crystallinity and less amount of α–Fe_2O_3 existing in these powders. Both the increase of hydrothermal time and of R value can promote the crystallinity of powders and also cause a significant loss of zinc, hinting that in the hydrothermal process, the loss of zinc may play a crucial role in the crystallinity of hydrothermally synthesized powders.

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Thermoelectric properties of PbTe thin films prepared by gas evaporation method

M. Ito, W-S. Seo, K. Koumoto
(Nagoya University)

PbTe thin films with fine grains were successfully fabricated by the gas evaporation method. Thermoelectric properties, i.e. Seebeck coefficient and electrical conductivity, both decreased with decreasing grain size. This was attributed to the decrease in carrier mobility exceeding the increase in carrier concentration with decreasing grain size. It was clarified that the

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effects of grain boundaries and of oxidation on carrier mobility are considerably large.
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Formation of $TiSi_2$ on nitrogen ion implanted (001)Si
S.L. Cheng*, L.J. Chen*, B.Y. Tsui+
*(*National Tsing Hua University,*
+Industrial Technology and Research Institute)

Formation of $TiSi_2$ on nitrogen ion implanted (001)Si has been investigated. Nitrogen ion implantation was found to suppress the B and As diffusion in silicon. For Ti on 30 keV BF_2^+ – 20 keV N_2^+ and 30 keV As^+ – 20 keV N_2^+ implanted samples, a continuous low-resistivity $TiSi_2$ layer was found to form in all samples annealed at 700–900 °C. For Ti on 1 x $10^{15}/cm^2$ N_2^+- and As^+-implanted samples, end-of-range defects were completely eliminated in all samples annealed at 700–900 °C. The results indicated that with appropriate control, N^+-implantation can be successfully implemented in forming low-resistivity $TiSi_2$ contacts on shallow junctions in deep submicron devices.
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Ultraviolet patterning of $KTiOPO_4$ (KTP) thin films through metallo-organics
K. Noda, W. Sakamoto, T. Yogo, S. Hirano
(Nagoya University)

Patterned $KTiOPO_4$ (KTP) films were successfully synthesized through metallo-organics using ultraviolet (UV) patterning. A homogeneous precursor solution was prepared by the reaction control of $(^nBuO)_2P(O)(OH)$, $Ti(OEt)_4$ and KOEt in ethanol. The solubility of the KTP precursor films in ethanol changed with UV irradiation because of the polymerization of KTP precursor. The patterned KTP precursor films crystallized to single-phase KTP after heat treatment at 600 °C for 2 h. The patterned films were found to be exactly stoichiometric KTP.
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Ion beam enhanced thermal depolymerization of poly(methyl methacrylate)
M.E. Fragalà, G. Compagnini, O. Puglisi
(Università di Catania)

Ion beam enhanced thermal depolymerization of poly(methyl methacrylate) thin films, 1–2 μm thick, has been studied in the temperature range 100–400 °C using a 300 keV He^+ beam at very low fluence (5 x 11^{10} – 5 x10^{11} *ions* cm^{-2}). A relevant monomer evolution (mass signal m/z = 100) at temperature (150 °C) well below the conventional degradation temperature (360 °C) has been detected during irradiation. The observed phenomenon is discussed in terms of activation energies and diffusion processes within the investigated films. The possibility offered by this phenomenon of performing a microlithography process in only one step is discussed.
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Synthesis of silicon-based polymerized films by excimer laser ablation deposition of hexaphenyldisilane
X. Zeng, F. Rossignol, S. Konno, H. Nagai, Y. Nakata, T. Okutani, M. Suzuki
(Hokkaido National Industrial Research Institute)

A new method of synthesizing silicon-based polymer films by excimer laser ablation of hexaphenyldisilane (HPDS) has been studied. The polymerized films were formed on a substrate by laser ablation deposition of HPDS at 248 nm. The structure of the polymerized films depended strongly on the laser fluence and repetition rates. The thermal stability and hardness of the deposited films were estimated by thermogravimetry and a Vickers microhardness meter. The films showed good thermal stability, depending on the laser processing parameters.
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Vapor deposition of parylene-F using hydrogen as carrier gas
D. Mathur, G-R. Yang, T-M. Lu
(Rensselaer Polytechnic Institute)

A new method for depositing parylene-F (PA-F) thin films on silicon substrates has been explored. Hydrogen has been used as a carrier gas along with liquid precursors, dibromotetrafluoro-p-xylene and 1,4-bis(trifluoromethyl)benzene, to deposit PA-F. The properties of this film have been compared with the films obtained by the Gorham dimer method {[W.F. Gorham, U.S. Patent 3 342 754], [W.F. Gorham and J.T.C. Yeh, J. Org. Chem. **34**, 2366 (1965)], [W.F. Gorham, J. Poly. Sci., Pt. A-1 **4**, 3027 (1966)]} and the liquid precursor method [L. You, G-R. Yang, C-I. Lang, T-M. Lu, J.A. Moore, and J.F.P. McDonald, U.S. Patent 5 268 202] using FTIR, XPS, and XRD. The PA-F films deposited by the dimer or liquid precursor acquired some kind of microcrystallinity on annealing. However, the PA-F films deposited in the presence of hydrogen were amorphous on annealing. This property could be potentially exploited for application in microelectronic device fabrication.
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Morphology of melt-crystallized poly(ethylene 2,6-naphthalate) thin films studied by transmission electron microscopy
M. Tsuji, F.A. Novillo L., M. Fujita, S. Murakami, S. Kohjiya
(Kyoto University)

Thin films of poly(ethylene 2,6-naphthalate) (PEN) were isothermally crystallized at 190 °C after being melted at 300 °C. Morphological observation by transmission electron microscopy (TEM) showed the spherulitic textrure in the films. Selected-area electron diffraction (SAED) indicated that the crystals in the films are the α–form, as expected from our thermal condition for crystallization. The SAED pattern from the untilted specimen was characterized by the fairly intense reflection ring accompanied by other weak rings, and this intense ring was indexed as 010. A series of SAED patterns, which were obtained from the same specimen area tilted at various angles in the TEM column, suggested that the crystallites are oriented with their (001) planes being preferentially parallel to the film surface. Subsequently, a set of the dark-field images of the two-dimensional spherulite taken by using two different parts of the 010 reflection ring revealed that most of the crystallites in such a spherulite are oriented with their (010) planes being parallel in its radial direction. In addition, the spehrulites in small pieces of PEN, which had been crystallized under the same thermal condition as above, were determined to be negatively birefringent by polarizing light microscopy.
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The influences of reactant composition and substrate material on the combustion synthesis of diamond
C.A. Wolden*, C.E. Draper+, Z. Sitar+, J.T. Prater#
*(*Colorado School of Mines, +North Carolina State University,*
#Army Research Office)

It has been observed that diamond deposition by flat flame chemical vapor deposition is achieved over a very narrow range of reactant composition. We demonstrate that this diamond deposition window is strongly determined by the nature of the substrate material. Furthermore, once a continuous diamond film is formed, the window appears to be independent of the original material. Substrates examined include silicon, glass, titanium, tungsten, nickel, and molybdenum. The dependence of growth rate, morphology, and quality on reactant composition have been quantified using scanning electron microscopy, Raman spectroscopy, and secondary ion mass spectroscopy (SIMS). It was found that the highest quality diamond was grown at conditions where diamond does not nucleate on ultrasonically scratched silicon. Thus, the production of high quality diamond on silicon by combustion synthesis requires different conditions for nucleation and growth.
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Superstructure and boundary structure in stage-4 MoC_5–graphite intercalation compounds studied by atomic force microscopy and scanning tunneling microscopy

V. Vignal*, H. Konno*, M. Inagaki*, S. Flandrois+, J.C. Roux+
*(*Hokkaido University, +Université Bordeaux)*

Intercalated domains on stage-4 $MoCl_5$–graphite intercalation compounds ($MoCl_5$–GICs) were observed by AFM and STM. On large intercalated domains, a superstructure was found, in relation with a modulation of the electronic properties of the first layer of carbon. From that, the structure of the chloride ions layer was discussed and a model including dimer molecules was proposed. At the boundaries between large intercalated and non-intercalated domains, corrugations were observed along certain crystallographic directions of graphite. Their morphology was studied in detail at atomic scale, and formation mechanisms were proposed. Small intercalated domains were also observed. Their shapes were irregular but their boundaries were clear cut.

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Ion-beam mixing in energetic collision cascades: Thermal-spike model and experiments

B. Park*, H. Lee+
*(*Seoul National University, +Georgia Institute of Technology)*

A phenomenological model of ion-beam mixing during energetic collision cascades is developed, based on the concept of a thermal spike, to correctly predict that the mixing rate *Dt* depends linearly on nuclear stopping power (instead of a power-law dependence), and is correlated with a heat of mixing (analogous to Darken's relation). Previous ion-beam mixing experiments from 25 different metallic bilayers agree well with the model's predictions: mixing rates (*Dt*)/(ion-dose) ~ 1 nm^4, and an activation enthalpy of approximately 1 eV for atomic diffusion in liquid-like cascades.

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Thermodynamic characterization of hydrogen interaction with iridium polyhydride complexes

R.A. Zidan, R.E. Rocheleau
(University of Hawaii at Manoa)

Hydrogen interaction with solid iridium complexes $IrXH_2(PPr^i_3)_2$ (X = Cl, I) was investigated. Gaseous hydrogen was found to react reversibly with solid chloro-iridium complex $IrClH_2(PPr^i_3)_2$, forming $IrClH_2(PPr^i_3)_2H_2$. The relative partial molal enthalpy and entropy were obtained from equilibrium isotherms at different hydrogen concentrations. The decrease in entropy with increasing hydrogen concentration and the absence of plateaus in the equilibrium isotherms were consistent with a single phase solid solution with two chemical components. Hydrogen release from solid iodo-iridium complex $IrIH_2(PPr^i_3)_2H_2$ was not observed at temperatures up to 350 K, indicating stronger hydrogen bonding.

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Substrate effects on nanoindentation mechanical property measurement of soft films on hard substrates

T.Y. Tsui, G.M. Pharr
(Rice University)

Substrate effects on the measurement of thin film mechanical properties by nanoindentation methods have been studied experimentally using a model soft film on hard substrate system: aluminum on glass. The hardness and elastic modulus of aluminum films with thicknesses of 240, 650, and 1700 nm sputter-deposited on glass were systematically characterized as a function of indenter penetration depth using standard nanoindentation methods. Scanning electron and atomic force microscopy of the hardness impressions revealed that indentation pileup in the aluminum is significantly enhanced by the substrate. The substrate also affects the form of the unloading curve in a manner which has important implications for nanoindentation data analysis procedures. Because of these effects, nanoindentation measurement techniques overestimate the film hardness and elastic modulus by as much as 100% and 50%, respectively. depending on the indentation depth. The largest errors occur at depths approximately equal to the film thickness.

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Distribution of intercalant in copper chloride– and iron chloride–graphite intercalation compounds

S-S. Tzeng, M-H. Nien
(Tatung Institute of Technology)

$CuCl_2$– and $FeCl_3$–graphite intercalation compounds were prepared by a gas phase reaction method using different carbon host materials. The effect of the microstructure of carbon host materials on the amount of intercalation and the distribution of intercalant was investigated using x-ray diffraction and electron microprobe analysis. For a fiber sample with a core region of onion skin structure in the cross-section, a lower concentration of intercalant was measured in the core region. For fiber samples with an oriented-core transverse microtexture, different intercalant distributions along the long axis (oriented-core direction) and the short axis of the elliptical cross-section was found.

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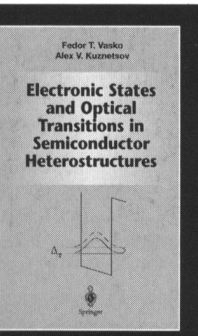

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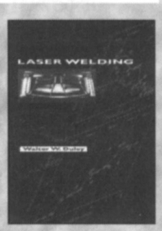

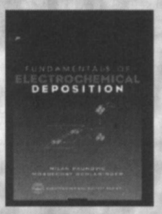

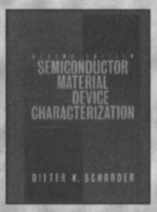

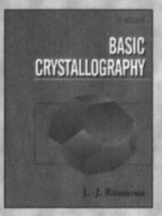

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