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ABSTRACTS

SPECIAL SECTION
MATERIALS SYNTHESIS AND PROCESSING

This manuscript is part of a series of papers that are featured in a special issue on Materials Synthesis and Processing in the April 1996 Journal of Materials Research.

Synthesis of mullite coatings by chemical vapor deposition

R.P. Mulpuri, V.K. Sarin
(Boston University)

Formation of mullite on ceramic substrates via chemical vapor deposition was investigated. Mullite is a solid solution of Al_2O_3 and SiO_2 with a composition of $3Al_2O_3{\cdot}2SiO_2$. Thermodynamic calculations performed on the $AlCl_3$-$SiCl_4$-CO_2-H_2 system were used to construct equilibrium CVD phase diagrams. With the aid of these diagrams and consideration of kinetic rate limiting factors, initial process parameters were determined. Through process optimization, crystalline CVD mullite coatings have been successfully grown on SiC and Si_3N_4 substrates. Results from the thermodynamic analysis, process optimization, and effect of various process parameters on deposition rate and coating morphology are discussed.

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COMMUNICATIONS

Microstructural development of $BaTiO_3$ powders synthesized by aqueous methods

L. Zhao, A.T. Chien, F.F. Lange, J.S. Speck
(University of California-Santa Barbara)

The hydrothermal growth of perovskite $BaTiO_3$ powders has been studied by transmission electron microscopy. The growth is carried out under highly alkaline conditions (pH~14) achieved with $Ba(OH)_2$. Anatase (TiO_2) is used as a titanium source. The perovskite $BaTiO_3$ nucleates heterogeneously on anatase TiO_2 particles with an epitaxial relationship of $(001)TiO_2 \parallel (001)BaTiO_3$ and $[010]TiO_2 \parallel [010]BaTiO_3$. This epitaxial relationship preserves the parallel alignment of the oxygen octahedra between the structures. A mosaic misorientation between $(001)TiO_2$ and $(001)BaTiO_3$ along <110> is seen in this relationship due to the lattice mismatch between TiO_2 and $BaTiO_3$. After complete conversion of the anatase to $BaTiO_3$, the $BaTiO_3$ particles develop into {111} octahedrons with ~10 nm {001} and {110} microfacets on the {111} faces. This evolution suggests that {111} becomes the stable crystallographic facet for $BaTiO_3$ under highly alkaline conditions.

Order No.: JA606-002 **© 1996 MRS**

Effect of mechanical damage on thermal conduction of plasma sprayed coatings

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(National Institute of Standards and Technology)

A thermal wave methodology for monitoring the thermal conduction of ceramic coatings with accumulating mechanical damage is described. Tests are conducted on a model alumina coating containing laminar defect intralayers. Controlled subsurface damage introduced with a spherical indenter is observed using a pre-sectioned specimen. Microcrack damage accumulates progressively with increasing contact load and number of cycles. Associated changes in thermal diffusivity, specifically in the through-thickness direction, are imaged and quantified point-by-point using laser-generated thermal waves. The effective thermal resistance of the coating increases with crack density, up to the point of failure.

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Waveguide formation of $KTiOPO_4$ by multi-energy MeV H^+ implantation

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*(*Shandong University, +University at Albany)*

X-cut potassium titanyl phosphate ($KTiOPO_4$ or KTP) was implanted by multi-energy MeV He^+ implantation with a total dose of 2×10^{16} ions/cm^2 at liquid nitrogen temperature. The energy and dose used are as follows: 3.3 MeV and 2×10^{15} ions/cm^2, 3.2 MeV and 4×10^{15} ions/cm^2, 3.1 MeV and 4×10^{15} ions/cm^2, and 3.0 MeV and 1.0×10^{16} ions/cm^2 to reduce tunnelling effect. The 22 dark modes were measured by isosceles prism coupling method. The 15 bright modes were observed after 250°C 60 min. annealing. The result shows that the waveguide formation of $KTiOPO_4$ implanted by MeV He^+ is not strongly dependent on the cut direction, which is different from the waveguide formation of $KTiOPO_4$ by ion exchange process.

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ARTICLES

Origin of the $\phi\sim\pm$ 9° peaks in $YBa_2Cu_3O_{7-\delta}$ films grown on cubic zirconia substrates

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c-axis oriented $YBa_2Cu_3O_{7-\delta}$ films grown on (001) yttria-stabilized cubic zirconia (YSZ) substrates often contain domains whose in-plane alignment is rotated approximately 9° from the cube-on-cube epitaxial relationship, in addition to the more commonly observed 0° and 45° in-plane rotations. We have investigated the origin of this ~9° orientation using *in-situ* electron diffraction during growth and *ex-situ* 4-circle x-ray diffraction. Our results indicate that the ~9° orientation provides the most favorable lattice match between the interfacial (110)-oriented $BaZrO_3$ epitaxial reaction layer, which forms between $YBa_2Cu_3O_{7-\delta}$ and the YSZ substrate. If epitaxy occurs directly between $YBa_2Cu_3O_{7-\delta}$ and the YSZ substrate, i.e., before the $BaZrO_3$ epitaxial reaction layer is

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formed, the 0° and 45° domains have the most favorable lattice match. However, growth conditions which favor the formation of the $BaZrO_3$ reaction layer prior to the nucleation of $YBa_2Cu_3O_{7-\delta}$, lead to an increase in ~9° domains. The observed phenomenon, which results from epitaxial alignment between the diagonal of a square surface net and the diagonal of a rectangular surface net, is a general method for producing in-plane misorientations, and has also been observed for the hetero-epitaxial growth of other materials, including $(Ba,K)BiO_3/LaAlO_3$. The $YBa_2Cu_3O_{7-\delta}$/YSZ case involves epitaxial alignment between $[111]_{BaZrO_3}$ and $[110]_{YSZ}$, resulting in an expected in-plane rotation of 11.3° to 9.7° for fully-commensurate and for fully-relaxed $(110)_{BaZrO_3}$ on $(001)_{YSZ}$, respectively.

Order No.: JA606-005 **© 1996 MRS**

Process optimization for the fabrication of $Tl_2Ba_2Ca_2Cu_3O_{10}$ thin films

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(DuPont Central Research and Development)

A process has been developed for the fabrication of nearly single phase superconducting $Tl_2Ba_2Ca_2Cu_3O_{10}$ thin films on (100) $LaAlO_3$ substrates with superconducting transition temperature T_c of 120 K and low microwave surface resistance at temperatures up to 110 K. Amorphous BaCaCuO precursor films were first deposited by rf magnetron sputtering and then thallinated at elevated temperature. The double TlO layer phases ($Tl_2Ba_2Ca_2Cu_3O_{10}$ + $Tl_2Ba_2CaCu_2O_8$) formed preferentially over the single TlO layer phases ($TlBa_2Ca_2Cu_3O_9$ + $Tl_2Ba_2CaCu_2O_7$) at high Tl_2O partial pressures. Thin films containing $Tl_2Ba_2Ca_2Cu_3O_{10}$ and a small amount of CuO were prepared from Cu-rich precursor films (Cu/Ba > 1.7), while lower Cu content lead to the formation of $Tl_2Ba_2CaCu_2O_8$ as a secondary phase. $Tl_2Ba_2Ca_2Cu_3O_{10}$ film epitaxy was enhanced by carrying out the thallination in reduced oxygen partial pressures of 0.01-0.05 atm. Following the thallination step, the $Tl_2Ba_2Ca_2Cu_3O_{10}$ thin films had a superconducting transition temperature T_c of only 106±4 K. An additional 62 hour anneal at 800°C or an 8 hour anneal at 850°C in a Tl_2O/O_2 atmosphere increased the T_c to 120 K. The increase in T_c was accompanied by a decrease in the *c*-axis lattice constant, an enhancement in the long range order in the *c* direction, and the formation of a small amount of $Tl_2Ba_2CaCu_2O_8$ as a secondary phase. Minimization of surface resistance at high temperature (95–110 K) requires that the fraction of $Tl_2Ba_2CaCu_2O_8$ secondary phase in the films be kept low. Process routes are also described for the formation of nearly single phase $TlBa_2Ca_2Cu_3O_9$ and $TlBa_2CaCu_2O_7$ thin films and the formation of a new ordered intergrowth phase, $Tl_4Ba_4Ca_3Cu_5O_{18}$, which consists of alternating $Tl_2Ba_2CaCu_2O_8$ and $Tl_2Ba_2Ca_2Cu_3O_{10}$ layers.

Order No.: JA606-006 **© 1996 MRS**

Texture and chemical composition analyses on the $Hg_{0.66}Pb_{0.33}Ba_2Ca_2Cu_3O_y$ superconductor using the sealed quartz tube technique

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Pb substituted Hg-based superconductor of $Hg_{0.66}Pb_{0.33}Ba_2Ca_2Cu_3O_y$ has been fabricated using the sealed quartz tube technique. R-T and XDP measurements show that the specimen has a T_c of 135 K and contains mainly the Hg-1223 phase. SEM/EDX and TEM/EDX were employed to study the texture and chemical composition of the specimen. It is found that the specimen contains round-shaped grains with a mixture of Hg-1223, $BaCuO_2$ and $Ca_{0.85}CuO_2$ phases, square-shaped grains with a formula of $PbBa_2O_3$, small single crystals with single Hg-1223 phase, and crystal-like layers with a mixture of Hg-1223 and $BaCuO_2$ phase. We consider that though the doping of Pb can benefit the stabilization of the Hg-1223 phase, it introduces other impurity phases and textures in the specimen at the same time.

Order No.: JA606-007 **© 1996 MRS**

Effect of Ag-particulate addition on processing, microstructure and properties of MgO-whisker reinforced bulk BPSCCO high-temperature superconductor composites

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(University of Houston)

In associated papers,[1,2] it has been shown that weak thermo-mechanical properties of a bulk monolithic high-T_c superconductor (HTS) can be improved by introducing strong ceramic whiskers into the HTS ceramic materials. In this paper, we report a further study of incorporating Ag particulates, $(Ag)_p$, in a bulk monolithic BPSCCO and in the MgO-whisker reinforced BPSCCO composite. Effects of the $(Ag)_p$ addition on processing, microstructure, and superconducting and mechanical properties of the bulk monolithic BPSCCO and the $(MgO)_w$/BPSCCO composite are investigated. The results indicate that the highly ductile Ag particulates promote densification of the BPSCCO matrix phase in the composite during hot pressing. The microstructure of the HTS composite with the $(Ag)_p$ addition is similar to that in the HTS material without the $(Ag)_p$. The $(MgO)_w$/BPSCCO composite with 10% (by weight) Ag particulates has been shown to possess excellent superconducting properties. The $(Ag)_p$ addition to both the monolithic BPSCCO and the $(MgO)_w$/BPSCCO is found to increase appreciably their fracture toughnesses, but has little effects on mechanical strengths of the materials. Quantitative relationships have been established among solid-state processing variables, HTS phase developments, microstructures and superconducting and mechanical properties of the $(Ag)_p$/BPSCCO and the $(MgO)_w/(Ag)_p$/BPSCCO HTS composites.

Order No.: JA606-008 **© 1996 MRS**

Faceting, dislocation structure, and various scales of heterogeneity in a $YBa_2Cu_3O_{7-\delta}$ low-angle [001] tilt boundary

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The grain boundary topography and grain boundary dislocation network structure of a 6° [001] bicrystal of $YBa_2Cu_3O_{7-\delta}$ were studied using diffraction contrast transmission electron microscopy (TEM). Saw-tooth shaped arrays of facets composed of facets with lengths of a few tens of nanometers were observed in each of two widely separated sections of the boundary. The facet planes were {110}, {310}, and {221}. Further subfaceting of the (130) facets into a smaller-scale (a few nanometers) saw-tooth configuration of (100) and (110) facets produced a hierarchy of facets in at least one boundary section. The dislocation content observed in each type of facet agreed well with Frank's formula. However, the dislocations within individual facets frequently were inhomogeneously distributed, contrasting the picture of evenly-spaced dislocations that is derived for boundaries of infinite extent. Certain types of dislocations repeatedly were grouped near the facet centers and ends. Well-separated partial dislocations frequently were observed near the facet mid-sections, but not near the facet junctions. Extended (~30 nm) strain contrast was observed at all of the facet junctions formed by facets with dimensions on the order of tens of nanometers. This long range strain may be due to the finite extent of the individual facets. These results all suggest that structural inhomogeneities occur on various length scales ranging from macroscopic to just a few nanometers. Such structural heterogeneity is consistent with the electrical heterogeneity that is indicated for many $YBa_2Cu_3O_{7-\delta}$ grain boundaries.

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Cation sublattice stacking faults in Cu-rich chalcopyrite $CuInSe_2$

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Using transmission electron microscopy, we have found stacking faults on the cation sublattice in the chalcopyrite structure of $CuInSe_2$.

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These films are grown by molecular beam epitaxy under Cu-rich conditions. These stacking faults are found to extend large distances in the plane of the film, and are not found to be present in samples not grown in Cu-rich conditions. We suggest that this defect is triggered by a Cu-induced transformation of the surface structure of the growing film.
Order No.: JA606-010 **© 1996 MRS**

A low-temperature technique for measuring enthalpies of formation
T.P. Weihs, T.W. Barbee Jr., M.A. Wall
(Lawrence Livermore National Laboratory)

A technique to accurately measure the formation enthalpies of transition metal compounds at relatively low temperatures using thick multilayer foils and differential scanning calorimetry is demonstrated. The enthalpy of formation of $Cu_{51}Zr_{14}$ was measured using 25 μm thick, free-standing Cu-Zr multilayer foils. The multilayers were deposited onto Si substrates using a planetary, magnetron source sputtering system. They were removed from their substrates, cut into 6 mm diameter specimens and scanned in temperature from 50°C to 725°C in a Differential Scanning Calorimeter. Three distinct exothermic reactions were systematically observed. The heats from the first two reactions were summed and then analyzed using a simple model that accounts for interfacial reactions and heat losses during deposition. The enthalpy of formation for $Cu_{51}Zr_{14}$ was measured to be 14.3±0.3 kJ/mol. This quantity agrees with the single value of ΔH_f = 14.07±1.07 kJ/mol reported in the literature for this Cu-Zr compound. The advantages of measuring formation enthalpies using thick multilayer foils and low-temperature calorimetry are discussed.
Order No.: JA606-011 **© 1996 MRS**

Syntheses of metastable tetragonal (*t'*) zirconia-calcia solid solution by pyrolysis of organic precursors and coprecipitation route
M. Yashima*, M. Kakihana*, K. Ishii+, Y. Ikuma+, M. Yoshimura+
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Three kinds of chemical processes: (1) citrate gel process, (2) acetate gel process, and (3) coprecipitation route, have been applied to the synthesis of homogeneous metastable tetragonal (*t'*) and cubic solid solutions of ZrO_2-Xmol% CaO (X=4–20). From Raman scattering study, the citrate gel process based on the gelation of the aqueous solution of citric acid containing Zr and Ca ions was found to produce compositionally homogeneous samples in comparison with the other two methods. Axial ratio *c/a* decreases with increasing concentration of CaO and becomes unity around 8–10 mol% CaO composition.
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Calculation of metastable immiscibility region in Al_2O_3-SiO_2 system
T. Ban, S. Hayashi, A. Yasumori, K. Okada
(Tokyo Institute of Technology)

Metastable liquid-liquid immiscibility region in Al_2O_3-SiO_2 system was calculated by a regular solution model using three sets of liquidus data from the stable phase diagrams reported. These calculations indicated that the immiscibility region extended to richer in Al_2O_3 composition than those reported before. A miscibility gap calculated using the liquidus data reported by Klug et al. (model A) ranged from 2.6 to 71 mol% Al_2O_3 at around 1000°C and it was the most compatible result with the idea that metastable pseudotetragonal mullite crystallized at around 1000°C became Al_2O_3 rich composition due to the immiscibility phase separation before mullitization among three sets of liquidus data.
Order No.: JA606-013 **© 1996 MRS**

Dielectric properties of TiO_2-Nb_2O_5 crystallographic shear structures
R.J. Cava, J.J. Krajewski, W.F. Peck Jr., G.L. Roberts
(AT&T Bell Laboratories)

The dielectric constants (K) have been measured near ambient temperature for polycrystalline bulk ceramics of the crystallographic shear compounds $TiNb_{24}O_{62}$, $Ti_2Nb_{10}O_{29}$ and $TiNb_2O_7$. These show enhanced K's over Nb_2O_5 and TiO_2, with the 2:10:29 phase displaying an ambient temperature K of approximately 130. We also report the effects of partial substitution of Nb by Ta. In general, the dielectric constants are enhanced for 5–10% Ta substitution, with the detailed behavior differing for the three phases.
Order No.: JA606-014 **© 1996 MRS**

High-temperature plasticity of cubic bismuth oxide
A. Vilette, S.L. Kampe
(Virginia Polytechnic Institute and State University)

Cubic (δ) bismuth oxide (Bi_2O_3) has been subjected to high temperature deformation over a wide range of temperatures and strain-rates. Results indicate that bismuth oxide is essentially incapable of plastic deformation at temperatures below the monoclinic to cubic phase transformation which occurs at approximately 730°C. Above the transformation temperature, however, Bi_2O_3 is extensively deformable. The variability of flow stress to temperature and strain rate has been quantified through the determination of phenomenological-based constitutive equations to describe its behavior at these high temperatures. Analysis of the so-determined deformation constants indicate an extremely strong sensitivity to strain rate and temperature, with values of the strain rate sensitivity approaching values commonly cited as indicative of superplastic behavior.
Order No.: JA606-015 **© 1996 MRS**

Diffraction analysis of non-uniform stresses in surface layers; application to cracked TiN coatings chemically vapor deposited onto Mo
W.G. Sloof, B.J. Kooi, R. Delhez, Th.H. de Keijser, E.J. Mittemeijer
(Delft University of Technology)

Variations of residual stresses in layers on substrates can occur in directions parallel and perpendicular to the surface as a result of compositional inhomogeneity and/or porosity or cracks. Diffraction methods to evaluate such stress variations are presented. Comparison of the experimental value for the stress with a calculated value of the "diffraction averaged stress," on the basis of a model for the local stresses, proved to be a useful method of stress analysis. It is shown that a direct evaluation of occurring stress-depth profiles is less practical.

The method of stress analysis proposed is applied to chemically vapor deposited TiN coatings on Mo substrates. In these coatings a large tensile stress parallel to the surface develops during cooling from the deposition temperature, due to difference in thermal shrink between coating and substrate. As a result of the cooling-induced stress, cracking of the coating occurs. The mesh width of the crack pattern allows determination of the fracture surface energy and the fracture toughness of the coating material. Conceiving the cracked coatings as assemblies of free-standing columns, and assuming full elastic accommodation of the thermal mismatch at the column/substrate interface, the stress variations in the coating are calculated. On this basis the diffraction averaged stress and the depth profile of the laterally averaged stress can be predicted accurately for the cracked TiN layers.
Order No.: JA606-016 **© 1996 MRS**

Characteristics of titanium nitride films grown by pulsed laser deposition
R. Chowdhury, R.D. Vispute, K. Jagannadham, J. Narayan
(North Carolina State University)

Laser physical vapor deposition (LPVD) has been used to grow titanium nitride films on hydrogen terminated silicon(100) substrates at deposition temperatures ranging from room temperature to 600°C. A pulsed KrF excimer laser (λ = 248 nm, τ = 25 ns) was used with the deposition chamber maintained at a base pressure of 10^{-7} Torr prior to deposition. The properties of the films were investigated by x-ray

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diffraction, Auger electron spectroscopy, Raman spectroscopy, optical, scanning and high resolution transmission electron microscopy, and measurement of electrical resistivity. When the substrate temperature was low (at and below 500°C) oxygen atoms from the residual gases were incorporated in the films. The microstructures and resistivities of TiN films were found to be strongly dependent on the temperature of the silicon substrates. The TiN films deposited at 600°C were oxygen-free, as observed from Auger analysis and the room temperature resistivity was found to be 14–15 μΩ-cm. Raman spectroscopy of the films showed that the nitrogen related optical phonon peak increased in comparison with the titanium related acoustic peak. The transmission electron microscopy and x-ray diffraction analyses showed that the films were polycrystalline at low temperature with grain size ranging from 300–600 Å depending on the temperature of the substrate. At 600°C, the films were found to be single crystals with occasional presence of dislocation loops. The spacing of Moiré fringes in TiN/Si samples deposited at 600°C established the nearly periodic elastic strain field extending into the TiN and Si at the interface. Although there exists a large misfit between TiN and Si (24.6%), the epitaxial growth of TiN films on Si(100) substrates was explained by means of domain matched epitaxy with a 4-to-3 match in unit cells for TiN/Si structure giving rise to a residual lattice misfit of only 4%.

Order No.: JA606-017 **© 1996 MRS**

Microstructural instability in single crystal thin films

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Epitaxial $PbTiO_3$ thin films were produced from a mixed Pb-Ti-double alkoxide precursor by spin-coating onto single crystal (001) $SrTiO_3$ substrates. Heat-treatment at 800°C produces a dense and continuous, epitaxial lead titanate film through an intermediate Pb-Ti-fluorite structure. A microstructural instability occurred when very thin single crystal films were fabricated; this instability caused the films to become discontinuous. Scanning electron microscopy and atomic force microscopy observations show that single crystal films with a thickness less than ~80 nm developed holes that expose the substrate; thinner films broke up into isolated, single crystal islands. The walls of the holes were found to be (111) perovskite planes. A free energy function, which considered the anisotropic surface energies of different planes, was developed to describe the microstructural changes in the film and to understand the instability phenomenon. The function predicted that pre-existing holes greater than a critical size are necessary to initiate hole growth and it predicted the observed morphological changes in the current system. Morphological stability diagrams that explain the stability fields for different film configurations, i.e., either completely covered, with holes, or single crystal islands, can be calculated for any film/substrate system.

Order No.: JA606-018 **© 1996 MRS**

Low-pressure chemical vapor deposition of silicon nitride using the environmentally friendly tris(dimethylamino)silane precursor

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This study investigates the use of the environmentally benign precursor tri(dimethylamino)silane (TDMAS) with NH_3 to synthesize silicon nitride films by low pressure chemical vapor deposition. The growth kinetics are investigated as a function of deposition temperature, total pressure, and NH_3/TDMAS flow ratios. The deposits are found to be essentially stoichiometric and to contain ~5 at% carbon when appropriate NH_3 concentrations are present. The films are found in all cases to be amorphous and highly tensile. For optimized processing conditions, values of the refractive index are close to those reported for Si_3N_4. The film density is observed to increase with higher deposition temperatures up to 800°C and then decrease due to the onset of gas phase nucleation effects. This behavior is readily reflected in the etch rate of those films. FTIR spectra reveal the presence of hydrogen even at high deposition temperatures (900°C). The hardness and Young's modulus of the films are seen to increase with higher deposition temperatures reaching saturation values near 20 and 185 GPa, respectively, above 800°C.

Order No.: JA606-019 **© 1996 MRS**

Glass-ceramic sealants for solid oxide fuel cells: Part I. Physical properties

K.L. Ley, M. Krumpelt, R. Kumar, J.H. Meiser, I. Bloom
(Argonne National Laboratory)

A family of sealant materials has been developed for use in the solid oxide fuel cell (SOFC) and in other applications in the temperature range of 800–1000°C. These materials are based on glasses and glass-ceramics in the SrO-La_2O_3-Al_2O_3-B_2O_3-SiO_2 system. The coefficients of thermal expansion (CTE) for these materials are in the range of 8–13 × 10^{-6}/°C, a good match with those of the SOFC components. These sealant materials bond well with the ceramics of the SOFC and, more importantly, form bonds that can be thermally cycled without failure. At the fuel cell operating temperature, the sealants have viscosities in the range of 10^4–10^6 Pa-s, which allow them to tolerate a CTE mismatch of about 20% among the bonded substrates. The gas tightness of a sample seal was demonstrated in a simple zirconia-based oxygen concentration cell.

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Surface microstructure of $Zr_{41.25}Ti_{13.75}Cu_{12.5}Ni_{10.0}Be_{22.5}$, a bulk metallic glass

M.A. LaMadrid, S.D. O'Connor, A. Peker, W.L. Johnson, J.D. Baldeschwieler
(California Institute of Technology)

The surface of $Zr_{41.25}Ti_{13.75}Cu_{12.5}Ni_{10.0}Be_{22.5}$, a bulk metallic glass prepared by RF induction melting, has been imaged using atomic force microscopy. The untreated surfaces were very smooth—features were no higher than 3 nm over a 10 × 10 μm region, comparable to many polished surfaces. Two types of microstructure were also observed; periodic striations forming either a striped or a checkered structure were present, with wavelengths between 1 and 2 μm, and amplitude of approximately 2 nm; in other cases, "cracked mud"-like patterns were observed. These microstructures could be related to strain induced surface roughening; preliminary calculations are presented that are consistent with this hypothesis.

Order No.: JA606-021 **© 1996 MRS**

Competitive gas-solid reactions realized by ball milling of Zr in ammonia gas

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(The Australian National University)

Competitive gas-solid reactions have been observed during high-energy ball milling of zirconium metal powder under an ammonia atmosphere at room temperature. The milling processes were investigated by monitoring pressure changes and subsequently analyzing milled powders with x-ray diffraction, differential thermal analysis and elemental composition analysis. It is shown that during an early stage of milling, while the pressure is decreasing, a hydridation reaction predominates and leads to the formation of ZrH_2 phase. During further milling the pressure increases again, and corresponds to a nitridation reaction between ZrH_2 and available nitrogen (either excess NH_3 or incorporated nitrogen in the powder) to form ZrN. Hydrogen gas is liberated and complete conversion to ZrN occurs after prolonged milling. The above reaction sequence was confirmed by the nitridation reaction of ZrH_2 ball milled in NH_3 atmosphere.

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Lithium-aluminum-carbonate-hydroxide hydrate coatings on aluminum alloys: Composition, structure, and processing bath chemistry

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(Sandia National Laboratories)

A new corrosion resistant coating, being designed for possible replacement of chromate conversion coatings on aluminum alloys, was investigated for composition, structure, and solubility using a variety of techniques. The stoichiometry of the material, prepared by immersion of 1100 Al alloy into a lithium carbonate-lithium hydroxide solution, was approximately $Li_2Al_4CO_3(0H)_{12}*3H_2O$. Processing time was shown to be dependent upon the bath pH, and consistent coating formation required supersaturation of the coating bath with aluminum. The exact crystal structure of this hydrotalcite material, hexagonal or monoclinic, was not determined. It was shown that both the bulk material and coatings with the same nominal composition and crystal structure could be formed by precipitation from an aluminum supersaturated solution of lithium carbonate.

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Pulsed laser ablation-deposition of $La_{0.5}Sr_{0.5}CoO_3$ for use as electrodes in non-volatile ferroelectric memories

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(North Carolina State University)

$La_{0.5}Sr_{0.5}CoO_3$ (LSCO) thin films have been deposited on (100) MgO substrates using pulsed laser ablation-deposition (PLAD). The crystallographic orientation of LSCO was found to be dependent on the surface treatment of (100) MgO prior to deposition. PLAD deposition param-eters were optimized to yield LSCO films with an RMS surface roughness of 5–6 Å. A smooth surface morphology was reproduced as long as the oxygen content of the LSCO target was preserved. Otherwise, "splashing" occurred which resulted in the transfer of condensed particles from molten spherical globules of LSCO from the target to the substrate. Splashing was subsequently eliminated and smooth surface quality was restored after annealing the LSCO target at 550°C in oxygen for 3 hours. Optical emission spectroscopy (OES) of the LSCO's plume identified excited atomic cobalt neutrals, excited singly ionized strontium and lanthanum, and excited molecular LaO species. Oxygen interaction with the plume produced no new species. Furthermore, the OES data suggests that the observed LaO molecules were not created by the chemical reaction between La and O_2 during ablation, but were ejected directly from the target during the PLAD process.

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The effects of particle size distribution and induced unpinning during grain growth

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The effect of a second-phase particle size distribution on grain boundary pinning was studied using a Monte Carlo simulation technique. Simulations were run using a constant number density of both whisker and rhombohedral particles, and the effect of size distribution was studied by varying the standard deviation of the distribution around a constant mean particle size. The results of present simulations indicate that, in accordance with the stereological assumption of the topological pinning model, changes in distribution width had no effect on the pinned grain size. The effect of induced unpinning of particles on microstructure was also studied. In contrast to predictions of the topological pinning model, a power law dependence of pinned grain size on particle size was observed at T=0.0. Based on this, a systematic deviation to the stereological predictions of the topological pinning model is observed. The results of simulations at higher temperatures indicate an increasing power law dependence of pinned grain size on particle size, with the slopes of the power law dependencies fitting an Arrhenius relation. The effect of induced unpinning of particles was also studied in order to obtain a correlation between particle/boundary concentration and equilibrium grain size. The results of simulations containing a constant number density of monosized rhombohedral particles suggest a strong power law correlation between the two parameters.

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Impression creep behavior of SiC particle-$MoSi_2$ composites

D.P. Butt, D.A. Korzekwa, S.A. Maloy, H. Kung, J.J. Petrovic
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Using a cylindrical indenter (or punch), the impression creep behavior of $MoSi_2$-SiC composites containing 0–40% SiC by volume, was characterized at 1000–1200°C, 258–362 MPa punch pressure. Through finite element modeling, an equation that depends on the material stress exponent was derived that converts the stress distribution beneath the punch to an effective compressive stress. Using this relationship, direct comparisons were made between impression and compressive creep studies. Under certain conditions, compressive creep and impression creep measurements yield comparable results after correcting for effective stresses and strain rates beneath the punch. However, rate controlling mechanisms may be quite different under the two stressing conditions; in which case impression creep data should not be used to predict compressive creep behavior. The addition of SiC affects the impression creep behavior of $MoSi_2$ in a complex manner by pinning grain boundaries during pressing, thus leading to smaller $MoSi_2$ grains and by obstructing or altering both dislocation motion and grain boundary sliding.

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Crack-face fiber bridging: Finite element analysis, analytical model, and experimental result

J-W. Cao, M. Sakai
(Toyohashi University of Technology)

Prior to considering crack-face bridging, the stress/strain-field ahead of the crack-tip in the compact tension (CT) geometry is numerically assessed by means of finite element method (FEM). The stress-field along the crackline which has a decreasing profile of tensile stresses from the crack-tip converts to monotonically increasing compressive stresses toward the back face of the CT-specimen via a rotational center. Based on these stress-field analyses, a novel crack-face bridging model for fiber reinforced brittle matrix composites is presented. The application of the model to the experimental result of a 2D-*C/C* composite enables estimation of the crack-face fiber bridging stresses and their distribution profile.

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Effects of TiO_2 on the microstructure and mechanical properties of Al_2O_3/ZrO_3 composites

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(National Cheng-Kung University)

The microstructure and mechanical properties of hot-pressed zirconia toughened alumina (ZTA), fabricated from the ZTA powders containing $(Zr,Ti)O_2$ in Ar gas, were investigated. In hot-pressed ZTA, the presence of Ti concentration in the grains of Al_2O_3 or $(Zr,Ti)O_2$ was analyzed by the EDS attached to the AEM apparatus. Experimental results showed that some Ti ions diffused to the grain boundary and into the Al_2O_3 grains, thereby enhancing the densification of hot-pressed ZTA. It was possible to retain tetragonal ZrO_2 at room temperature in the ZTA specimens containing ≥ 5.5 mol% TiO_2 and hot-pressed at 1350°C for 1 h. The bending strength and toughness of hot-pressed ZTA were enhanced by the addition of TiO_2.

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In-situ characterization of vapor phase growth of iron oxide-silica nanocomposites: Part I. 2-D planar laser-induced fluorescence and Mie imaging

B.K. McMillin*, P. Biswas+, M.R. Zachariah*
*(*National Institute of Standards and Technology, +University of Cincinnati)*

Planar laser-based imaging measurements of fluorescence and particle scattering have been obtained during flame synthesis of iron-oxide/silica superparamagnetic nanocomposites. The theory and application of laser induced fluorescence, the spectroscopy of FeO(g), and the experimental approach for measurement of gas phase precursors to particle formation are discussed. The results show that the vapor phase FeO concentration rapidly rises at the primary reaction front of the flame and is very sensitive to the amount of precursor added, suggesting nucleation controlled particle growth. The FeO vapor concentration in the main nucleation zone was found to be insensitive to the amount of silicon precursor injected, indicating that nucleation occurred independently for the iron and silicon components. Light scattering measurements indicate that nanocomposite particles sinter faster than single component silica, in agreement with TEM measurements.

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Al_2O_3/Al particle reinforced aluminum matrix composite by displacement reaction

M.R. Hanabe, P.B. Aswath
(University of Texas at Arlington)

The development of a novel Al matrix composite is described based on a simple displacement reaction when an SiO_2 particulate preform is brought into contact with liquid Al at temperatures between 1273 and 1373 K. This interaction leads to the wetting of the SiO_2 particles by Al and its eventual transformation to a composite with Al_2O_3/Al particles in an Al-matrix. Infiltration of the preform as induced by this reaction takes place with the simultaneous formation of the Al_2O_3/Al particles *in-situ.* Synthesis of engineered multiphase composites, wherein reinforcements of other materials incorporated into the preform and reacted with liquid Al is also presented.

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Plasma treatment of polyaniline films: Effect on the intrinsic oxidation states

E.T. Kang*, K. Kato+, Y. Uyama+, Y. Ikada+
*(*National University of Singapore, +Kyoto University)*

Surface modification of emeraldine (EM) and nigraniline (NA) base films by argon plasma treatment was investigated by x-ray photoelectron spectroscopy (XPS). Argon plasma treatment, followed by atmospheric exposure, results in the oxidation of some carbon atoms, first to C-O species and then to C=O and COOH species for samples with extended plasma treatment time. Most important of all, Ar plasma treatment and the accompanied carbon oxidation readily cause a decrease in the intrinsic oxidation state ([=N-]/[-NH-] ratio) of the aniline polymers.

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Stick-slip in the scratching of styrene-acrylonitrile copolymer

K. Li*, B.Y. Ni+, J.C.M. Li*
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Stick-slip process occurred during the scratch test of styrene-acrylonitrile copolymer. For the first time a bamboo-like morphology of the scratch track corresponding to the stick-slip phenomenon was observed. A "joint" was formed during the stick stage and during slip a uniform "stem" was made. The period and amplitude of the stick-slip both increase with the vertical load and decrease with the driving speed. A theoretical model is constructed based on the stiffness of the system and the plastic deformation of polymer both in the vertical and horizontal directions. The model assumes no distinction between the coefficients of static and kinetic friction and gives quantitatively consistent results with experiments.

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REVIEW

Electrically active organic and polymeric materials for thin-film-transistor technologies

A.J. Lovinger, L.J. Rothberg
(AT&T Bell Laboratories)

Organic and polymeric materials have seen a tremendous growth in research in the last five years as potential electroactive elements in thin-film-transistor (TFT) applications. These are driven by the increasing interest in flat panel-display applications, for which organic and polymeric materials offer strong promise in terms of properties, processability, cost, and compatibility with eventual lightweight, flexible plastic displays. In this review we summarize the current status of our knowledge on the science of these organic and polymeric semiconducting materials. Most of these are based on linear thiophenes, especially α-hexathienyl, which has elicited by far the most attention. Mobility values in the 10^{-2}-10^{-1} cm^2/Vs and especially source-drain current on/off ratios of up to 10^6 make this a highly promising potential alternative to amorphous silicon. Other thienyl compounds are also discussed, as are polymeric analogues. A brief discussion of technological potential, limitations, and problems that need to be overcome is given at the end.

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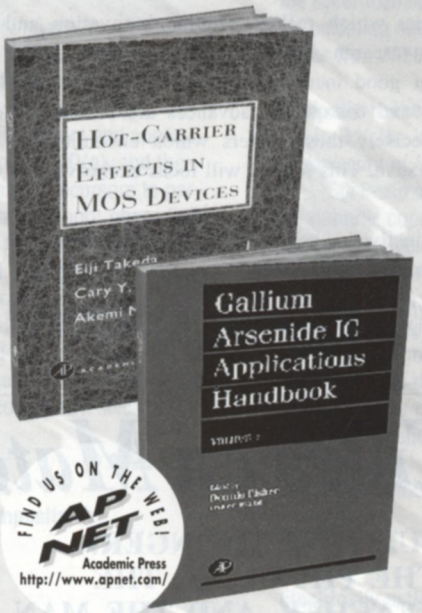

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