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ABSTRACTS**COMMUNICATIONS****Study of the growth mechanism of highly in-plane aligned a-axis $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ thin films on LaSrGaO_4 substrate by high resolution electron microscopy**J.G. Wen, S. Mahajan, H. Ohtsuka, T. Morishita, N. Koshizuka
(*Superconductivity Research Laboratory-ISTEC*)

Highly in-plane aligned a-axis $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ thin films deposited on (100) LaSrGaO_4 substrates by a self-template method were studied by high-resolution electron microscopy along three orthogonal $\langle 100 \rangle$ axes of the substrate. Plan-view images confirm that the majority of the film preferentially aligns across the entire substrate except for very few misaligned domains with average size 10 nm^2 . Cross-sectional images along the [100] orientation of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ reveal that in-plane aligned a-axis $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ is grown on a template layer dominated by c-axis oriented film. This strongly suggests that the in-plane alignment of a-axis $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ thin films on (100) LaSrGaO_4 substrates is governed by the different stresses along the b and c axes of the substrate. Cross-sectional images along [001] of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ thin film reveal that the 90° domains easily nucleate in the region between a-axis $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ and the $\text{YBa}_4\text{Cu}_3\text{O}_x$ phase. Cracks along the (001) plane of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ are found to be due to the large mismatch between the c parameters of the thin film and substrate.

Order No.: JA612-001

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Growth of highly (111) oriented, highly coalesced diamond films on platinum (111) surface—A possibility of heteroepitaxy

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(The University of Tokushima)

A highly (111)-oriented, highly coalesced diamond film was grown on platinum (111) surface by microwave plasma chemical vapor deposition (MPCVD). Scanning electron microscopy and x-ray diffraction analyses revealed that the (111) diamond facets were azimuthally oriented epitaxially with respect to the orientation of the Pt (111) domain underneath, with the neighboring facets of diamond being coalesced with each other. The film was confirmed as diamond using Raman spectroscopy.

Order No.: JA612-002

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High efficiency deposition of diamond film by hot filament chemical vapor deposition

Y. Chen, Q. Chen, Z. Lin

(Chinese Academy of Sciences)

A new designed reaction chamber with new relative distribution of filament and substrates has been adopted in order to increase the deposition

area of diamond films and thus increase the deposition efficiency in conventional hot filament chemical vapor deposition (HFCVD) system. The relative small reaction chamber was cuboid shaped ($50 \times 25 \times 2 \text{ mm}^3$) made of molybdenum wafers. It was established in the vacuum chamber. A tungsten filament was hung up vertically in the center of the small chamber and parallel to the gas flow path. At the four inner sides of the reaction chamber, four Si(100) substrates ($30 \times 10 \times 0.5 \text{ mm}^3$) were installed to grow diamond films. The deposition results indicate that uniform diamond films can be obtained on the four substrates, and the film growth rates are the same at both ends of the substrates. The diamond film growth rate was about $1\text{--}2 \mu\text{m/h}$ with deposition conditions similar to those of the conventional HFCVD method. Thus the deposition area and efficiency can be increased four times in the case without the filament number, gas flow rate and the power consumption.

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Band gaps in diamond-graphite hybrids

R. Sen, R. Sumathy, C.N.R. Rao

(Indian Institute of Science and Jawaharlal Nehru Center for Advanced Scientific Research)

The HOMO-LUMO gaps have been estimated in a graphite-like sp^2 carbon network with progressive increase in the fraction of sp^3 carbons, taking into account several possible structural alternatives for each composition. The gap is shown to increase exponentially with the fraction of sp^3 carbons. Accordingly, the gap in a diamond-like sp^3 network decreases with the increase in the fraction of sp^2 carbons.

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A new method for hardness determination from depth sensing indentation tests

J. Gubicza, A. Juhász, J. Lendvai

(Eötvös University)

A new semi-empirical formula is developed for the hardness determination of the materials from depth sensing indentation tests. The indentation works measured both during loading and unloading periods are used in the evaluation. The values of the Meyer hardness calculated in this way agree well with those obtained by conventional optical observation, where this latter is possible. While the new hardness formula characterizes well the behavior of the conventional hardness number even for the ideally elastic material, the mean contact pressure generally used in hardness determination differs significantly from the conventional hardness number when the ideally elastic limiting case is being approached.

Order No.: JA612-005

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Combustion co-synthesis of Si₃N₄-based *in-situ* composites

J.T. Li, W.S. Liu, Y.L. Xia, C.C. Ge

(University of Science and Technology Beijing)

The feasibility of synthesizing silicon nitride-silicon carbide-titanium carbonitride composites by combustion reactions is demonstrated. With titanium carbonitride taken to be an ideal solid solution, its composition is determined as TiC_{0.36}N_{0.64}. Thermodynamic analysis supports the experimental results.

Order No.: JA612-006

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ARTICLES**Microstructural study of growth of a YBa₂Cu₃O_{7-x}/LaAlO₃/YBa₂Cu₃O_{7-x} trilayered film by pulsed laser deposition**

Y.H. Li*, A. Staton-Bevan*, J.A. Kilner*, Z. Trajanovic*, T. Venkatesan*

(*Imperial College, *University of Maryland)

The growth process of a YBCO/LaAlO₃/YBCO trilayered film made by pulsed laser deposition has been studied by HRTEM. The high resolution images of the cross-section samples have shown that a 7 nm layer of LaAlO₃ has been grown epitaxially between c-axis oriented YBCO layers having the nominal thickness of 250 nm. A stacking fault in the LaAlO₃ layer may introduce a stacking fault into the YBCO layer, which may form nucleation sites for a-axis oriented grains. A second phase had been formed at the interface between the LaAlO₃ layer and the lower YBCO layer, which has been identified by image simulation and EDX analysis as a new tetragonal La-Al-Cu-O phase based on LaAlO₃ in which some of the Al atoms have been replaced by Cu. The approximate lattice parameters of the new phase are: a = 0.38 nm and c = 0.76 nm. However, no second phase was found at the interface between the lower YBCO layer and the LaAlO₃ substrate.

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Study of growth kinetics in melt-textured YBa₂Cu₃O_{7-x}

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Directional solidification has been shown to be a successful way of achieving high current densities in bulk YBCO. The lack of understanding of the growth kinetics, however, makes it difficult to fabricate longer samples and reduce the processing times. To study the growth kinetics, quenching experiments of undoped YBa₂Cu₃O_{7-x} (Y-123) and Y-123 doped with Pt and Nd from above the peritectic temperature with different holding times, *t*, were conducted. The results of these experiments indicate that the average 211 particle size varies as *t*^{1/3}. Growth rate experiments were also conducted on these samples to determine the maximum growth rate for plane front solidification, *R*_{max}. This quantity was measured for undoped and doped Y-123 and it was found that the addition of Pt did not increase *R*_{max} while the addition of Nd doubled the growth rate. Using the coarsening results together with the growth rate experiments, the diffusivity of Y in liquid and the 211-liquid interfacial energy for undoped and doped Y-123 were calculated.

Order No.: JA612-008

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A transmission electron microscopy study of the crystallinity and secondary phase formation in melt processed YBa₂Cu₃O_{7-x}

Y. Yan, D.A. Cardwell, A.M. Campbell, W.M. Stobbs

(University of Cambridge)

The microstructure of large grain melt processed YBa₂Cu₃O_{7-x} containing 10 molar % excess Y₂BaCuO₅ prepared and oxygenated under atmospheric pressure has been investigated by transmission electron microscopy (TEM) and optical microscopy. These materials always contain parallel structural and microscopic platelet-like features in the crystallographic a-b plane of a few microns spacing which have been variously

described as grain boundaries or microcracks. We have observed such features, which clearly influence the flow of current in melt processed YBCO, to consist of copper deficient, impurity phase material which can be either amorphous or crystalline in nature. A variety of defects have been observed by HREM in the vicinity of these platelet boundaries, including double and triple CuO layer stacking faults, which may constitute effective flux pinning sites.

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Single-crystal structure refinement of four compounds in Y_{1-x}Pr_xBa₂Cu_{3-y}Al_yO_{7-δ} system

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(*University of Ljubljana, *Graz University of Technology, #National Institute of Chemistry)

X-ray single crystal diffraction data were used for structural refinement of the title compounds with different x (0.15, 0.27, 0.49 and 0.89). Crystals were grown in alumina crucibles using self-flux method. Aluminum, which originates from the crucibles, substitutes only Cu(1), and thus induces tetragonal symmetry which was observed in all four crystals. The main structural effect of praseodymium is an increased separation of superconducting layers. Substituent concentrations (x and y in the formula) have been refined and compared with the values obtained by EDX (energy dispersive x-ray analysis) in an electron microscope. It was indicated that the refined values of Y : Pr ratio and the oxygen content are more reliable than those obtained by EDX while the refinement is less sensitive for Cu(1) : Al ratio and this value is more uncertain. This is in accordance with the result of wet chemical analysis.

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Preparation and characterization of several group 12 element (Zn, Cd)-bis(thiolate) complexes and evaluation of their potential as precursors for 12-16 semiconducting materials

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(Georgia Institute of Technology)

Compounds of the general formula M(SR)₂ (M = Zn, Cd; R = *i*-Pr, *t*-Bu, Bz) have been prepared and explored as potential unimolecular starting materials for the preparation of binary group 12 metal sulfides. These new compounds have been characterized by IR spectroscopy and thermogravimetric analysis. Chemical derivatization of these insoluble metal-bis(thiolate) compounds by complexation with *N*-CH₃-imidazole renders them more soluble. These adducts were investigated by elemental analysis. Thermolytic decompositions of both the parent and derivatized compounds have been carried out both in the solid state and by heating a suspension of the appropriate metal-bis(thiolate) compound in an inert high boiling hydrocarbon medium. The thermolysis products have been studied by GC/MS (liquids) and XRPD, SEM and particle size determination (solids).

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Photoelectron spectroscopy study of amorphous silicon-carbon alloys deposited by plasma enhanced chemical vapor deposition

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X-ray photoelectron spectroscopy (XPS) coupled with Fourier transform infrared (FTIR) and optical transmission spectroscopy (OTS) has been used for the characterization of silicon carbon alloys (a-Si_{1-x}C_x:H₂F) deposited via plasma, by varying the CH₄ amount in SiF₄-CH₄-H₂ feeding mixture. XPS measurements have shown that carbon rich a-Si_{1-x}C_x:H₂F alloys include large amounts of fluorine (> 11 at. %), which make the films susceptible to the air oxidation. In addition, the effect of the alloying partner carbon on the valence band (VB) and on the VB edge position of amorphous silicon is also described.

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Microstructure and phase stability of single crystal NiAl alloyed with Hf and ZrI.E. Locci⁺, R.M. Dickerson[#], A. Garg⁺, R.D. Noebe⁺, J.D. Whittenberger⁺, M.V. Nathal⁺, R. Darolia[#]

(*Case Western Reserve University, +NASA Lewis Research Center, #NYMA, Inc., \$General Electric Aircraft Engines)

Six near stoichiometric, NiAl single-crystal alloys, with 0.05-1.5 at.% of Hf and Zr additions plus Si impurities were microstructurally analyzed in the as-cast, homogenized, and aged conditions. Hafnium-rich interdendritic regions, containing the Heusler phase (Ni₂AlHf), were found in all the as-cast alloys containing Hf. Homogenization heat treatments partially reduced these interdendritic segregated regions. TEM observations of the as-cast and homogenized microstructures revealed the presence of a high density of fine Hf (or Zr) and Si rich precipitates. These were identified as G-phase, Ni₁₆X₆Si₇, or as an orthorhombic NiXSi phase, where X is Hf or Zr. Under these conditions the expected Heusler phase (β') was almost completely absent. The Si responsible for the formation of the G and NiHfSi phases is the result of molten metal reacting with the Si-containing crucible used during the casting process. Varying the cooling rates after homogenization resulted in the refinement or complete suppression of the G and NiHfSi phases. In some of the alloys studied, long term aging heat treatments resulted in the formation of Heusler precipitates, which were more stable at the aging temperature and coarsened at the expense of the G-phase. In other alloys, long term aging resulted in the formation of the NiXSi phase. The stability of the Heusler or NiXSi phases can be traced to the reactive element (Hf or Zr) to silicon ratio. If the ratio is high, then the Heusler phase appears stable after long time aging. If the ratio is low, then the NiHfSi phase appears to be the stable phase.

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X-ray diffraction and Raman scattering studies of FeCl₃-SbCl₅-graphite bi-intercalation compounds

T. Abe, Y. Yokota, Y. Mizutani, M. Asano, T. Harada, M. Inaba, Z. Ogumi (Kyoto University)

X-ray diffraction (XRD) and Raman spectroscopy have been used for the study of the bi-intercalation of SbCl₅ into a stage 5 FeCl₃-graphite intercalation compound (GIC). The stage 5 FeCl₃-GIC is prepared by an ordinary two-bulb method with the temperature of graphite at 788 K and that of FeCl₃ at 573 K. The FeCl₃-SbCl₅-graphite bi-intercalation compound (GBC) with one SbCl₅ layer is obtained when the temperature of the stage 5 FeCl₃-GIC is held at 443 K and the temperature of SbCl₅ at 373 K in the two-zone system. The stacking sequence of the GBC is found to be an admixture of G(FeCl₃)GG(SbCl₅)GGG(FeCl₃)G and G(FeCl₃)GGG(SbCl₅)GG(FeCl₃)G by XRD, where G, (FeCl₃), and (SbCl₅) are the graphite, FeCl₃, and SbCl₅ layers, respectively. The Raman spectrum of the GBC shows two peaks associated with the E_{2g}⁽²⁾ and E_{2g}⁽²⁾b modes at 1588 cm⁻¹ and 1610 cm⁻¹, respectively. For the temperatures of stage 5 FeCl₃-GIC at 443 K and SbCl₅ at 403 K in the two-zone system, the FeCl₃-SbCl₅-GBC with two SbCl₅ layers is obtained. The stacking sequence of the GBC is determined to be an admixture of G(FeCl₃)GG(SbCl₅)GG(SbCl₅)G(FeCl₃)G and G(FeCl₃)G-(SbCl₅)GG(SbCl₅)GG(FeCl₃)G. In the Raman spectrum of this GBC, two peaks associated with the E_{2g}⁽²⁾b mode are observed at 1616 and 1624 cm⁻¹.

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Synthesis and solid solution in ceramic (Ca_{1-x}Sr_x)₂PbO₄M. Xu⁺, D.K. Finmore⁺

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Ceramic (Ca_{1-x}Sr_x)₂PbO₄ samples have been prepared with x = 0.00, 0.25, 0.50, 0.75, and 1.00 in order to study the change in lattice constants with the ratio of Ca/Sr. This phase is frequently an intermediate phase in the fabrication of Bi-Sr-Ca-Cu-O superconductors and it is important to connect the lattice constants with stoichiometry. The behavior of the lattice constants closely follows Vegard's law.

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Description and classification of uranium oxide hydrate sheet anion topologiesM.L. Miller^{*}, R.J. Finch⁺, P.C. Burns^{*}, R.C. Ewing^{*}
(*University of New Mexico, +University of Manitoba)

The sheets of uranyl ions (U⁶⁺O₂)²⁺ in the structures of all uranyl oxide hydrates (UOH) (and the structurally related α - and β - forms of U₃O₈) are based on only four structural unit chains. Each sheet type may be reduced to its underlying sheet anion topology to determine the chains present within each topology and to describe the structural relationships among these phases. Each sheet type is described by a chain stacking sequence. The four chain types required to construct the UOH sheet anion topologies are the **H**-chain, the **R**-chain, the **P**-chain, and the directional "arrowhead" chains denoted by **U** and **D**.

The **H**-chain is found only in the sheet anion topology of α -UO₂(OH)₂ and consists of hexagonal sites sharing opposing edges. In α -UO₂(OH)₂, all hexagons are populated with uranyl ions. The "arrowhead" chain is composed of pentagonal sites populated with uranyl ions and sharing edges and alternating with vacant triangular sites. Arrowhead chains are present in the sheet anion topologies of all other UOH sheets. Arrowhead chains are directional and can occur in both a **U** and **D** "sense" within a single anion topology. The **P**-chain consists of edge-sharing pentagonal sites populated with uranyl ions forming a zigzag chain. The **P**-chain is flanked on both sides by arrowhead chains of the same "sense". The remaining structural unit is a discontinuous "chain" of rhombic sites. This "**R**-chain" is produced when nested, adjacent **U** and **D** "arrowhead" chains are translated diagonally. The **R**-chain occurs in the sheet anion topologies of sheets which contain only 4-coordinate uranyl ions and those containing both 4- and 5-coordinate uranyl ions. The rhombic sites may be populated with a uranyl ion, a U⁴⁺ or other cation and two apical oxygens, or they may be vacant.

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Toughness determination of zirconia toughened alumina ceramics from growth of indentation-induced cracksD. Basu, B.K. Sarkar
(Jadavpur University)

Short surface cracks were generated by Vickers indentation on the polished surface of alumina and different zirconia toughened alumina (ZTA) specimens and their morphology was studied by serial sectioning. These cracks were grown in three point bend tests under stepwise loading, and variation of toughness with crack extension was plotted to graphically separate the contributions from residual stress intensity and applied stress intensity factors. The plateau toughness determined from the intercept height of the crack extension plots exhibited an upward trend with zirconia content up to 15 vol% ZrO₂ addition in the composition which was proportional to the fraction of transformable tetragonal grains contributing to transformation toughening.

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Barium titanate-added lead nickel niobate ferroelectrics: Accelerated perovskite formation and dielectric propertiesC-H. Lu, J-F. Wu
(National Taiwan University)

The addition of BaTiO₃ to Pb(Ni_{1/3}Nb_{2/3})O₃ has been confirmed to vary the formation kinetics of the perovskite solid solutions of Pb(Ni_{1/3}Nb_{2/3})O₃-BaTiO₃, and to suppress the generation of the pyrochlore phase. A semi-quantitatively calculated reaction conversion confirmed that increasing the BaTiO₃ content significantly accelerated the formation of the perovskite solid solutions. The formed solid solutions of Pb(Ni_{1/3}Nb_{2/3})O₃-BaTiO₃ (up to 90 mol% of BaTiO₃) exhibited a cubic symmetry at room temperature. The lattice parameter monotonously decreased with an increase in the BaTiO₃ content. The structural stability of the perovskite phase was found to be enhanced by the addition of BaTiO₃ as well. The formed solid solutions were able to maintain the perovskite struc-

ture without decomposition when heated up to 1250°C. The frequency dependence of the apparent Curie temperature and the diffuseness of the dielectric peak of sintered specimens were increased with increasing the BaTiO₃ content up to 50 mol%. Whereas with further addition, the relaxor characteristics in the specimens became obscure, associated with lower frequency dependence and less broadening of the dielectric maximum. The largest broadening of the dielectric peak occurred at $x = 0.5$, implying that this composition exhibited the most disordered structure which is probably related to the most random arrangement of B-site cations in oxygen octahedron.

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Wet forming, sintering behavior and dielectric properties of BaTi_{0.8}Zr_{0.2}O₃

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(Kagoshima University)

Density, grain size, lattice parameter and dielectric properties were measured on BaTi_{0.8}Zr_{0.2}O₃ sintered at 1100–1600°C. The hydrothermally prepared BaTi_{0.8}Zr_{0.2}O₃ particles of 128 nm diameter were consolidated by filtration of 2-methoxyethanol suspensions and subsequently compressed by isostatic pressing under a pressure of 294 MPa to form a uniform microstructure of high density (52% of the theoretical density). These green compacts were sintered to relative density of above 99% in the temperature range from 1350 to 1600°C where rapid grain growth to above 30 μm occurred. The increase of sintering temperature was accompanied by the increase of lattice parameter and dielectric constant of BaTi_{0.8}Zr_{0.2}O₃ at room temperature. The sintered BaTi_{0.8}Zr_{0.2}O₃ showed a diffuse phase transition from paraelectric (higher temperature) to ferroelectric state (lower temperature) at 32°C.

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Effect of defect structure on gas sensitivity of LaCrO₃

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(Motorola Inc.)

The isopropyl alcohol gas sensitivity of LaCrO₃ at 250°C is found to depend on the amount of TiO₂ content and cation stoichiometric ratio of the sample. The gas sensitivity enhancement is related to the defect structure and electrical conduction behavior of p-type donor doped semiconductive oxides. The high resistivity coupled with the increasing point defects by the donor dopants are responsible for the high gas sensitivity of TiO₂ doped LaCrO₃. It is believed that the positively charged ionic type defects created by dopants act as trapping sites to adsorb oxygen.

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Titania and silica powders produced in a counterflow diffusion flame

A.J. Rulison, P. F. Miquel, J.L. Katz
(The Johns Hopkins University)

Earlier publications describe the counterflow diffusion flame burner and its unique capability to produce oxide particles having certain structures, such as spheres of one material coated with another, spheres of one composition with attached bulbs of another composition, and uniform multicomponent mixtures. Here we describe the production and properties of bulk quantities of powders produced using this burner. Measurements were made of specific surface area and, for titania, of phase composition. It was found that the controls over powder characteristics used in other forms of flame-synthesis are equally effective in the counterflow diffusion flame burner. We found that the specific surface area of both silica and titania powders decrease with increasing precursor concentrations. Transmission electron microscopy analysis of the titania powders indicates that the mean size of the particles that comprise these powders increases with increasing concentration. These trends are consistent with the collision-coalescence theory of particle growth. In addition, the crystalline phase of titania can be controlled by selecting the appropriate feed stream. For example, over the ranges of TiCl₄ precursor concentrations tested, feeding it only into the oxidizer stream yields mainly anatase TiO₂ powders, while

feeding only into the fuel stream yields mainly rutile TiO₂ powders. These trends can be explained by the known atmosphere-dependent anatase-rutile transformation. The present data demonstrate that, in addition to its unique capability to produce certain particle shapes and morphologies, the counterflow diffusion flame burner can be manipulated to produce either of the major commercial titania phases, and also silica with a wide range of specific surface areas.

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Effects of the metal workpiece properties on the residual stresses in silicon nitride-metal brazed joints

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Modeling, but more importantly, measurements of residual stresses are needed to guide the design and development of high integrity ceramic/metal joints. This study evaluates the influence of the metal workpiece on the residual stress state present in the ceramic part of the joint. Si₃N₄ was directly bonded via the active metal brazing route to several metals Cu, Mo, W, Ta, Nb, Zr, Ti, AISI 316, selected to cover an extended range of thermo-mechanical properties. The residual strains in the joints were measured using an x-ray diffraction technique. The results indicate that the maximum residual stresses scale with the thermal mismatch for metals with low coefficients of thermal expansion. The experimental results are compared with analytical calculations of the residual stresses.

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Characterization of polyparaphenylene (PPP)-based carbons

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Polyparaphenylene (PPP)-based carbons heat-treated at temperatures (T_{HT}) from 600°C up to 3000°C have been characterized both structurally and in terms of their physical properties. Special attention is given to PPP heat-treated at 700°C (denoted by PPP-700), since samples heat-treated to this temperature were observed to have exceptionally high lithium affinities when electrochemically doped with Li. At low T_{HT} near 700°C it is found that the local structure of PPP-based samples can be characterized mostly in terms of a disordered polymer. As a result of heat-treatment to high temperature, PPP-based carbon shows graphitization behavior with regard to x-ray diffraction d_{002} (graphite c-axis d-spacing) development and to the increase of the Raman I_G/I_D intensity ratio (where I_G and I_D are the integrated intensities of the 1580 cm⁻¹ and 1360 cm⁻¹ Raman modes, respectively), as is found in so-called graphitizing carbons. However, development of the c-axis crystallite size (L_c) is restricted to very small values, in the range of so-called non-graphitizing carbons, while the a-axis crystallite size (L_a) attains values up to roughly 120 Å for heat-treatments near 3000°C. These structural properties of PPP-based carbons are consistent with the observed electrical characteristics and their dependence on T_{HT} . Low temperature magnetic susceptibility measurements were analyzed, along with Raman spectra, allowing for the characterization of disorder in terms of localized spin states for several heat-treated PPP samples. By interpreting the results of these various characterization techniques, we are able to present an insightful perspective on the nature of PPP-based carbons and the role of PPP-700 as an effective lithium host material for secondary battery applications.

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Structure of nanocrystalline palladium and copper studied by small angle neutron scattering

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The structure of nanocrystalline palladium and copper, made by inert gas condensation and compaction, was studied using small angle neutron

scattering (SANS), optical microscopy, and scanning electron microscopy. The effects of annealing and warm compaction were also examined with these techniques. The SANS results were interpreted using a maximum entropy routine, combined with knowledge of the Archimedes density and hydrogen concentration determined by prompt gamma activation analysis (PGAA). Similar hydrogen concentrations were detected by SANS and PGAA. This hydrogen content, which was approximately 5 at% in samples compacted at room temperature, was reduced by both annealing and warm compaction. Defects in several size classes were observed, including missing grain pores ($\approx 1\text{--}50$ nm diameter) and defects of micrometer size. Warm compaction produced a lower number density of pores in nanocrystalline palladium, which led to increased density. The observed structure was correlated with Vickers microhardness and fracture surface morphology.

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Controlled double-jet precipitation of uniform colloidal crystalline particles of Zr- and Sr-doped barium titanates

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The synthesis of uniform colloidal crystalline particles of Zr- and Sr-doped barium titanates at a low temperature of 85°C by the controlled double-jet precipitation (CDJP) technique is described. The stoichiometry of the powders can be precisely controlled by adjusting the compositions of the starting reactant solutions. Barium titanate with 20% Zr substitution, sintered at 1275°C, satisfies the requirements for the Y5V multilayer capacitor application. The grain sizes are uniform and small, ranging from 1 to 3 μm . Solids with an extremely sharp change in the dielectric constant as a function of temperature, which are suitable for thermal IR detectors application, can be obtained when both Sr and Zr are incorporated as dopants.

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The selective area deposition of diamond films

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Diamond films were selectively nucleated and grown on single crystal (100) silicon by microwave plasma assisted chemical vapor deposition with sub-micron spatial resolution. A thermal silicon dioxide layer on the wafers was patterned by standard photolithography. Nucleation was performed by applying a dc bias of -250 to -350V in a hydrogen-methane plasma. Lifting-off the oxide layer by HF etching prior to growth delineated the nucleation pattern which was replicated by the diamond film after growth. The growth of polycrystalline diamond was performed in a hydrogen-carbon monoxide-methane mixture selected to facilitate (100) texturing. Individual faceted crystallites were grown on a square matrix of sites, with a pitch of 3 μm , by controlling the nucleation densities within the windows exposing the pre-nucleated silicon. However, the orientation of the crystallites were randomly aligned with respect to the (100) silicon lattice within the micron scale windows employed in this study.

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Continuous microscratch measurements of the practical and true works of adhesion for metal/ceramic systems

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Using a continuous microscratch technique, the adhesion strengths of Pt, Cr, Ti and Ta₂N metallizations to NiO and Al₂O₃ substrates have been characterized. The practical work of adhesion was determined as a function of both thickness and annealing conditions. For all except the Ta₂N films, the practical work of adhesion increases nonlinearly from a few tenths of a J/m² to several J/m² as the thickness of the thin film is increased, indicating that a greater amount of plastic work is expended in delaminating

thicker films. Further, the practical work of adhesion also increases with increasing annealing temperature indicating stronger bonding at the interface. In the limit that the film thickness tends to zero, the plastic energy dissipation in the film tends to zero. As a result, the extrapolation to zero thickness yields the true work of adhesion for that system.

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The structural changes of polycrystalline film C₆₀/C₇₀:Ni caused by Ni diffusion

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C₆₀/C₇₀:Ni films with Ni concentration 1.5%wt obtained by vacuum deposition under different thermal conditions have been investigated. The layer's structural changes were investigated by transmission electron microscopy, electron and x-ray diffraction and Raman spectroscopy. The polycrystalline structure was detected for the layers grown at approximately 450 K on the substrate. At elevated temperature and maintained temperature gradient on the substrate during the process, the change of the layer's structure and forming of Ni microcrystals were observed. The Ni microcrystals (5–10 nm in the diameter) and the elongated shapes dimensioned 10 x 150 nm were perceived.

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Transmission electron microscopy observation and optical property of sol-gel derived LiNbO₃ films

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Crystallization behavior, defects and interface structures of sol-gel derived LiNbO₃ films on three kinds of substrates were examined. The nucleation was found to occur epitaxially at the interface between the film and the substrate. The continuous film is formed by coalescence of the island-like crystallites. When sapphire substrate is used, which has large lattice mismatch with the LiNbO₃, the resulting film contains a large amount of micropores, twin structures and misfit dislocations. On the other hand, while LiTaO₃ and 5% MgO-doped LiNbO₃ substrates with smaller mismatch are used as substrates, the films show no evidence of the formation of dislocations and twins. The film on 5% MgO-doped LiNbO₃ substrate shows better optical waveguiding property.

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[NZP], NaZr₂P₃O₁₂-type materials for protection of carbon-carbon composites

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Carbon-carbon composites, if not suitably protected, suffer from the problem of oxidation of the surface in normal atmospheres at temperatures above 350°C. For this reason they need to be protected from oxidizing environments by either using an impermeable coating, or using a sacrificial protective coating of a suitable material, and/or doping of an oxidation inhibitor in the carbon. In this study we have used a new family of materials with tailorable thermal expansion characteristics, namely, the [NZP] family as the materials for developing a suitable coating material for C-C composites. The candidates selected for matching thermal expansion with that of carbon are Ca_{0.5}Sr_{0.5}Zr₄P₆O₂₄, SrZr₄P₆O₂₄, and Ba_{1.175}Zr₄P_{5.65}Si_{0.35}O₂₄. They can be sintered in inert atmosphere without decomposition of the phases, and can be hot-pressed in inert atmosphere with C-C composites at 1250°C without decomposition or chemical interaction. They are stable in the presence of carbon up to 1200°C for at least a period of 4 h. They also do not show any weight loss after exposure to various temperatures up to 1200°C for 4 h.

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Microporous SiO₂/Vycor membranes for gas separation

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In this study, porous Vycor tubes with 40 Å initial pore diameter were modified using low pressure chemical vapor deposition (LPCVD) of SiO₂. Diethylsilane (DES) in conjunction with O₂ or N₂O were used as precursors to synthesize the SiO₂ films. Both "single side" (reactants flowing on the same side of porous membrane) and "counterflow" (reactants flowing on both sides of porous membrane) reactant geometries have been investigated. The flow of H₂, He, N₂, Ar and toluene (C₇H₈) was monitored *in-situ* after each deposition period. Membranes modified by the "single side" reactants geometry exhibited good selectivities between small and large molecules. However, cracking in these membranes after prolonged deposition limited the maximum achievable selectivity values. Higher selectivities and better mechanical stability were achieved with membranes produced using the "counterflow" reactants geometry. Pore narrowing rate was observed to increase with oxidant flow (O₂ or N₂O). For membranes prepared using both oxidants, selectivities on the order of 1000:1 were readily attained for H₂ and He over N₂, Ar and C₇H₈. As compared to O₂, the use of N₂O caused improvements in both the pore narrowing rate and N₂:C₇H₈ selectivity. Membranes prepared using the "counterflow" geometry showed no signs of degradation or cracking after thermal cycling.

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REVIEW**Status of and prospects for organic electroluminescence**

L.J. Rothberg, A.J. Lovinger
(Bell Laboratories-Lucent Technologies)

We review the device and materials science behind organic electroluminescent diodes made both using discrete evaporable molecules and spin-cast organic polymers. A great deal of progress has been made in improving the efficiencies and spectral properties of organic light-emitting diodes, and these are now adequate for many applications. More work is necessary to understand the stability and degradation of emissive and charge-transporting organics, but some systems have been shown to be stable for 10⁴ hours at display brightness. Major challenges still face the community in terms of developing satisfactory systems design and processing techniques if organic electroluminescence is to realize either performance or economic advantages over alternative technologies and significantly penetrate the display market. We present an analysis of the suitability of organic light-emitting diodes for various applications, and consider the materials and manufacturing obstacles that must be overcome.

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