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ABSTRACTS

SPECIAL SECTION

HIGH TEMPERATURE SUPERCONDUCTING MATERIALS: THE FIRST TEN YEARS

Synthesis and properties of Tl-Ba-Ca-Cu-O superconductors

M.P. Siegal, E.L. Venturini, B. Morosin, T.L. Aselage
(Sandia National Laboratories)

We review the synthesis methods and properties of single crystal, powder and thin film TlBaCaCuO high-temperature superconducting (Tl-HTS) materials. With transition temperatures ≥ 100 K for several compounds, Tl-HTS materials present real opportunities for applications above 77 K. Experiments using: 1) single crystals determined precise structural parameters and identified the complex Tl^{1+}–Tl^{3+} equilibrium model; 2) powders studied the complex thermodynamic phase diagram; and 3) epitaxial films have studied fundamental properties such as electron pair symmetry and the effect of controlled extrinsic defects on flux pinning strength, as well as providing the large-area surfaces required for device applications.

Order No.: JA711-001

Subsolidus-phase equilibria of coexisting high-T_c Pb-2223 and 2212 superconductors in the (Bi,Pb)-Sr-Ca-Cu-O system under 7.5% O_2

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The subsolidus-phase relationships of the high-T_c 2223 superconductor in the (Bi,Pb)-Sr-Ca-Cu-O (BSCCO) system have been examined at 810–820°C. All experiments were carried out at ambient pressure in a 7.5% O_2 (92.5% Ar) atmosphere. Eleven phases were found to exist in equilibrium with the 2223 phase. These 11 phases include CuO and 10 oxide solid solutions. From among these phases, a total of 48 five-phase combinations including the 2223 and 2212 phases were investigated experimentally, and 16 equilibrium assemblages were found which define a multicomponent compositional space corresponding to the 2223+2212 solid-state compatibility region. The subsolidus data form a partial basis for future investigation of the Pb-2223 primary phase field.

Order No.: JA711-002

Fabrication of NdBCO single crystal oxide superconductor with enhanced superconductive properties

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$Nd_{1+x}Ba_{2-x}Cu_3O_{6+d}$ (Nd123) single crystals have been successfully grown by the top-seeded solution-growth method. Compositions of Nd123 could be controlled by applying two different methods which were (i) controlling the oxygen partial pressure of the atmosphere and (ii) controlling the liquid composition in air. The critical temperatures of Nd123 obtained by these two methods were 96 K (oxygen control) and 95 K (liquid composition control), respectively.

The relationship between the peak effect in the J_c-H curve and heat treatment was investigated. The peak effect was found not to be an intrinsic property of Nd123, consequently it could be controlled by heat treatment.

Order No.: JA711-003

Effect of initial composition on distribution of RE211 (422) particles in RE123 superconductors

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Microstructure in melt-textured bulk $RE_1Ba_2Cu_3O_{6+d}$ crystals (RE123; RE = Sm, Nd) were investigated, changing the initial composition from the tie-line composition of RE123-$Sm_2Ba_1Cu_1O_5$ (Sm211)/$Nd_4Ba_2Cu_2O_{10}$ (Nd422) to the Ba enriched side. It was found that the Sm211/Nd422 particle size decreased in the liquid with increasing the Ba/Cu ratio of the initial composition, and this tendency was also found in the grown Sm123 crystals. Composition of the Sm123 grown crystal could be controlled by selecting the Ba enriched initial composition to obtain an

almost stoichiometric compound, which resulted in higher T_c values. Furthermore, the J_c values also increased under low magnetic fields due to the significant decrease of Sm211 particle size. Therefore, changing the initial composition toward the Ba enriched side was found to be a new process to enhance both J_c and T_c values simultaneously.
Order No.: JA711-004

Diffusion model for the crystal growth of $Pr_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$ by the top seeded crystal pulling method
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A solidification model for $Pr_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$ ternary oxides by the top seeded crystal pulling (SRL-CP; solute rich liquid-crystal pulling) method is presented in which the composition of the grown single crystals is estimated from the starting composition in the crucible. This model involves the diffusion flux balance of each element at the growth interface in the liquid considering equilibrium tie-lines in the PrOy-BaO-CuO ternary phase diagram which have been obtained experimentally. The self-diffusion coefficient for Pr and the interdiffusivities for Ba and Cu in the liquid are used in this model because this liquid is a dilute solution for Pr. The calculated results are in good agreement with the experimental ones.
Order No.: JA711-005

Controlled processing and properties of large Pt-doped YBCO pseudo-crystals for electromagnetic applications
W. Lo, D.A. Cardwell, C.D. Dewhurst, H-T. Leung, J.C.L. Chow, Y.H. Shi
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The quality and preparation of Sm-Ba-Cu-O (SmBCO) seeds, the level of Pt-doping and the temperature distribution in the thermal processing system have been investigated in detail and identified as key processing factors in the fabrication of large melt processed Pt doped Y-Ba-Cu-O pseudo-crystals. Optimization of these process variables and features has been performed to enable controlled processing of these materials up to several centimeters in diameter. Inhomogeneities and second phases present in the YBCO-Pt pseudo-crystal microstructure have been analyzed and correlated with a variation of superconducting properties within the bulk of the sample. These observations have clear implications for the controlled processing of large YBCO-Pt pseudo-crystals for electromagnetic applications.
Order No.: JA711-006

Doping high-T_c superconductors with oxygen and metallic atoms: A molecular dynamics study
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When using classical molecular dynamics based on Lennard-Jones-like potentials a mechanically stable $YBa_2Cu_3O_7$ high-T_c superconductor structure is generated. This process is controlled via interactive computer graphics. After doping atoms into or removing atoms from the sample using a recently implemented picking mechanism, the lattice oscillation energy is annihilated with a simulated annealing procedure. The remaining minimum ground state energy allows marking of the preferred doping location. Information on the doping mechanism is important because the magnetic and superconducting properties of these compounds depend very strongly on their oxygen content.
Order No.: JA711-007

Colloidal processing and mechanical properties of silicon carbide with alumina
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Submicrometer-sized SiC coated with SiO_2 of 0.4–1.8 wt% and α-Al_2O_3 powder of median size 0.2 μm were mixed in aqueous solutions in the pH range 3.0–10.0. The SiC/Al_2O_3 (4.3–6.9 wt.%) powders were consolidated by filtration through gypsum molds and hot-pressed at 1600°–2040°C under a pressure of 39 MPa. These compacts were densified to near the theoretical density at 1700°–1800°C. The sintering mechanisms were discussed based on the analysis of shrinkage curves of SiC/Al_2O_3 compacts during hot-pressing. The equiaxed SiC grains grew with low aspect ratios below 1800°C and changed to plate-like grains at 1900°C. The fracture toughness of SiC as a function of average grain size reached a maximum of 5 MPa · $m^{0.5}$ at 2.5 μm grains of low aspect ratios of 1–2. The flexural strengths at room temperature were 230–430 MPa in the SiC above 98% of the theoretical density and showed a similar grain size dependence.
Order No.: JA711-008

Biaxial alignment control of YBCO films on random Ni-based alloy with textured YSZ films formed by ion-beam-assisted deposition
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Biaxially aligned $YBa_2Cu_3O_{7-x}$ (YBCO) films were fabricated on random Ni-based alloy tapes with YSZ buffer layers deposited by ion-beam-assisted deposition (IBAD). Ar^+ ion bombardment was found to have two significant effects on the crystalline structure of the YSZ buffer layers; to align a [100] axis with the substrate normal, and a [111] axis with the bombarding beam axis. The resulting YSZ films were biaxially aligned on the random polycrystalline tapes, and the azimuthal distribution of the *a*-and *b*-axes of YBCO films on the top of the YSZ films was restricted to 10 degrees FWHM. A critical current density (J_c) of 1.13 x 10^6 A/cm^2 (77 K, 0T) was obtained, and 1.1 x 10^5 A/cm^2 was maintained at 5T (77 K, B⊥c). The existence of both intrinsic and extrinsic pinning properties were clearly observed in the angular dependence of J_c with B⊥I. The longitudinal field effect on J_c was clearly observed, which indicated straight transport currents. This is evidence for strongly coupled current paths that demonstrate the bulk pinning properties of YBCO.
Order No.: JA711-009

Conductors with controlled grain boundaries: An approach to the next generation, high-temperature superconducting wire
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Much of the conductor development effort in the last decade has focused on optimizing the processing of $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ oxide-powder-in-tube conductors and $Bi_2Sr_2CaCu_2O_8$ (Bi-2212) and $TlBa_2Ca_2Cu_3O_x$ thick film conductors. It is demonstrated that in each of these conductors, critical current densities are dictated by the grain boundary misorientation distributions (GBMDs). Percolative networks of low angle boundaries with fractions consistent with the active cross-sectional area of the conductor exist in all of these conductors. Further enhancements in the properties of these conductors require increased numbers of small angle grain boundaries. Given the processing methods used to fabricate these materials, no clear route employing a simple modification of the established processing method is apparent. To address this need, conductors with controlled or predetermined GBMDs are necessary. Development of biaxial texture appears to be the only possible way to increase the number of small angle boundaries in a practical and controllable manner. We summarize in this paper recent results obtained on epitaxial superconducting films on rolling-assisted-biaxially-textured-substrates (RABiTS). This technique uses well established, industrially scaleable, thermomechanical processes to impart a strong biaxial texture to a base metal. This is followed by vapor deposition of epitaxial buffer layers (metal and/or ceramic) to yield structurally and chemically compatible surfaces. Epitaxial $YBa_2Cu_3O_{7-\delta}$ films grown using laser ablation on such substrates have critical current densities approaching 10^6 A/cm^2 at 77 K in zero-field and have a field dependence similar to epitaxial films on single crystal ceramic substrates. Deposited conductors made using this technique offer a potential route for the fabrication of the next generation high-temperature superconducting (HTS) wire capable of carrying high currents in high magnetic fields and at elevated temperatures.
Order No.: JA711-010

Influence of deposition rate on the properties of thick $YBa_2Cu_3O_{7-\delta}$ films produced by pulsed laser deposition

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To investigate potential limits to the rate at which high-quality $YBa_2Cu_3O_{7-\delta}$ can be deposited, we have produced a series of 1 μm thick films by pulsed laser deposition on single-crystal $SrTiO_3$ substrates at average rates ranging from 2 Å/s to 240 Å/s. The critical current density of low-rate films was over 2 MA/cm^2 at 75 K, self field, but dropped linearly with rate to about 1 MA/cm^2 at the upper end of the range. In addition, the superconducting transition temperature, resistivity above the transition, and performance in an applied magnetic field were all degraded by increasing the deposition rate. A change in *c*-axis lattice parameter suggests that possible causes for this degradation are oxygen deficiency or cation disorder, with the latter being the more likely. Annealing high rate films at 790°C for as little as 20 minutes improved critical current density to within 20% of low-rate values, and resulted in dramatic improvements in other film properties as well.

Order No.: JA711-011

$Y_1Ba_2Cu_3O_{7-x}$ multilayer structures with a thick SiO_2 interlayer for multi-chip modules

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For high-temperature superconducting multi-chip modules and other related electronic applications, it is necessary to be able to fabricate several $Y_1Ba_2Cu_3O_{7-x}$ (YBCO) layers separated by thick low dielectric constant dielectric layers. In this work, we report the successful fabrication of YBCO/YSZ/SiO_2(1–2 μm) /YSZ/YBCO multilayer structures on single crystal yttria stabilized zirconia (YSZ) substrates. In contrast to previously reported work, the top YBCO layer did not show any cracking. This is due to a technique which allows for stress relief in the SiO_2 layer before the second YBCO layer is deposited. The top YBCO layer in our multilayer structure had T_c = 87 K, and $J_c = 10^5$ A/cm^2 (at 77 K), whereas the bottom YBCO layer had T_c = 90 K, and $J_c = 1.2 \times 10^6$ A/cm^2 (at 77 K) We also showed that the quality of the bottom YBCO layer was preserved during the fabrication of the multilayer due to the annealing process during which O_2 diffused into the YBCO replacing the O_2 lost during the deposition of the top YBCO layer.

Order No.: JA711-012

The effect of ion milling on the morphology of ramp-type Josephson junctions

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Artificial barriers in Josephson junctions make it possible to change the height and width of the barrier independently. This technique can be realized in high-T_c Josephson junctions using the ramp technique. In this article the fabrication of ramp-type junctions is discussed and the importance of the morphology of the ramp is pointed out. Detailed investigations are described which address the surface roughness and the damage due to ion beam structuring of ramps. It is shown that hard masks can significantly improve the ramp quality by reducing the ion impact angle. Furthermore, annealing of the so-structured ramps leads to unit cell steps enforcing a step-flow growth mode.

Order No.: JA711-013

Development of a reliable materials base for superconducting electronics

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Careful studies of the corrosion, redox, galvanic and oxygen evolution/uptake reactions associated with $YBa_2Cu_3O_{7-\delta}$ and related compounds have been completed. These studies have led to an understanding of the many factors that contribute to the poor material characteristics exhibited by these popular high-T_c phases. With knowledge of the structure-reactivity relationships, a powerful crystal engineering approach has been developed that is capable of producing cation substituted versions of $YBa_2Cu_3O_{7-\delta}$; the resulting compounds therefrom produced exhibit markedly improved processability, oxygen stability and durability characteristics. These materials have been combined in thin film structures so as to make prototype SNS junctions and SQUID sensors which exhibit promising device performance characteristics.

Order No.: JA711-014

$REBa_2ZrO_{5.5}$ (RE = La,Ce,Eu and Yb): Synthesis, characterization and their potential use as substrates for $YBa_2Cu_3O_{7-\delta}$ superconductors

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A new class of complex perovskites $REBa_2ZrO_{5.5}$ (where RE = La,Ce,Eu and Yb) have been synthesized and sintered as single phase materials by the solid state reaction method. The structure of these materials was studied by x-ray diffraction, and all of them were found to be isostructural, having a cubic perovskite structure. X-ray diffraction and resistivity measurements have shown that there is no detectable chemical reaction between $YBa_2Cu_3O_{7-\delta}$ and $REBa_2ZrO_{5.5}$ even under severe heat treatment at 950°C and that the addition of $REBa_2ZrO_{5.5}$ up to 20 vol% in $YBa_2Cu_3O_{7-\delta}$ shows no detrimental effect on the superconducting properties of $YBa_2Cu_3O_{7-\delta}$. Dielectric constants and loss factors are in the range suitable for their use as substrates for microwave applications. Thick films of $YBa_2Cu_3O_{7-\delta}$ fabricated on polycrystalline $REBa_2ZrO_{5.5}$ substrates gave a zero resistance transition temperature Tc(o) ~ 92 K, indicating the suitability of these materials as substrates for $YBa_2Cu_3O_{7-\delta}$.

Order No.: JA711-015

Nanostructured high-temperature superconductors: Creation of strong-pinning columnar defects in nanorod/superconductor composites

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A chemical approach to the formation of columnar defects involving the growth and incorporation of MgO nanorods into high-temperature superconductors (HTSs) has been developed. MgO nanorods were incorporated into $Bi_2Sr_2CaCu_2O_z$, $Bi_2Sr_2Ca_2Cu_3O_z$ and $Tl_2Ba_2Ca_2Cu_3O_z$ superconductors at areal densities up to 2 x 10^{10}/cm^2. Microstructural analyses of the composites demonstrate that the MgO nanorods create a columnar defect structure in the HTS matrices, form a compositionally sharp interface with the matrix, and self-organize into orientations perpendicular and parallel to the copper oxide planes. Measurements of the critical current density demonstrate significant enhancements in the MgO nanorod/HTS composites at elevated temperatures and magnetic fields compared with reference samples.

Order No.: JA711-016

Connectivity and flux pinning improvements in Ag-clad BSCCO-2223 tapes produced by changes in the cooling rate

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The rate at which Ag-clad $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ tapes are cooled from their final reaction heat treatment influences both the intergranular connectivity and intragranular flux pinning strength of the polycrystalline filaments. As the cooling rate from 825°C to 730°C in 7.5% O_2 was decreased over a range of 5°C/min to 0.005°C/min, J_c (77 K, 0 T) increased from ~8 to ~24 kA/cm^2, and the irreversibility field increased from ~120 to ~200 mT. The J_c (4.2 K, 0 T) increased in a similar fashion. Cooling slowly also sharpened the critical temperature transition and increased the critical onset temperature from 107 K to 109 K. These improvements in the superconducting properties occurred despite partial decomposition of the $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ phase into non-superconducting impurity phases during the slow cooling. A microstructural basis for these multiple effects is described.

Order No.: JA711-017

Structure of grain boundaries: Correlation to supercurrent transport in textured $Bi_2Sr_2Ca_{n-1}Cu_nO_x$ bulk material

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Two kinds of characteristic grain boundaries were observed in textured $Bi_2Sr_2Ca_{n-1}Cu_nO_x$ (n=2 and 3) bulk material: one (P-type) is nearly parallel to the (001) plane, and the other (N-type) is approximately normal to the (001) plane. Low angle tilt N-type boundaries are composed of arrays of dislocations. However, for a small *c*-axis misorientation, the regions between the dislocation cores are still well connected, providing "pathways" for supercurrents crossing the boundary plane. The P-type boundaries exhibit compositionally and structurally modulated faceting. Although we see local regions of the low-T_c (2201) phase at low angle tilt (<10°) P-type boundaries, there are also "pathways" crossing the boundary plane made up of the high-T_c (2212) and (2223) phases. The characteristics of such low angle tilt grain boundary structures can therefore be modeled to provide general insight into the correlation between high critical current densities and low texture breadths. On the other hand, a weak link could be formed at high angle (>10°) boundaries where there are the low-T_c (2201) or insulating phases.

Order No.: JA711-018

From meandering to straight grain boundaries: Improving the structures of artificially-induced grain boundaries in superconducting $YBa_2Cu_3O_y$ bicrystals

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This paper presents several key aspects of our approach to preparing artificially-induced [001] tilt grain boundaries (GBs) with uniform, well-defined structures in $YBa_2Cu_3O_y$ (YBCO) superconductors. GB structures formed in thin film and bulk bicrystals, respectively, will be compared. In YBCO thin film bicrystals, meandering rather than planar GBs are formed. Using a low film deposition rate has been demonstrated to reduce the magnitude of meander significantly, but complete elimination of the meander has not yet been accomplished. Thus, we have developed a dual-seeded-melt-texture process to produce uniform, planar GBs with controllable misorientation angles in YBCO bulk bicrystals. Transmission electron microscopy (TEM) studies reveal a remarkably planar and simple configuration on different length scales. Such a simple structure allows for an insightful interpretation of transport behavior across individual GBs.

Order No.: JA711-019

Surface nucleation, domain growth mechanisms, and factors dominating superconducting properties in seeded melt grown $YBa_2Cu_3O_x$

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Using a seeded melt growth (SMG) method, we have produced single-domain $YBa_2Cu_3O_x$ with high levitation forces and trapped magnetic fields. Both surface nucleation and domain growth mechanisms have been identified for SMG processing. A threshold temperature T_L has been found above which extraneous nucleation does not occur, and the domain growth can proceed throughout the entire sample, resulting in a single-domain structure. Surface nucleation has been suppressed when the top sample surface is coated with low melting compounds. The planar growth rates along the *a*- and *c*-axis have been found to be comparable within the undercooling range used in this study, and agree well with the current model. No dendritic growth has been observed. Major factors that strongly influence the levitation force have been studied in detail including domain geometry and orientation. Current physical models have been used to interpret the observed levitation force behaviors.

Order No.: JA711-020

The effects of variable oxygen partial pressures during Bi-2223 tape processing

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The effect of changes in the oxygen partial pressure during the heat treatment of Bi-2223 tapes has been investigated. Distinct differences were observed in the phase assemblages, compositions, and critical current densities. Of particular importance in the correlation of transport properties and microstructure was the redistribution of lead in both the secondary phases and the Bi-2223 matrix. The highest J_c values were associated with tapes that contained Ca_2PbO_4 as part of the phase assemblage coupled with lower concentrations of lead in the superconducting phase. The high J_c of 30.4 kA/cm^2 was obtained in tapes processed at 820°C where the oxygen partial pressure was increased from 10% O_2/Ar to 20% O_2/Ar after 25 hours of each 50 hour sinter cycle. Samples processed only in 10% O_2/Ar at 820°C did not contain Ca_2PbO_4, had slightly higher concentrations of lead in the superconducting phase, and had significantly lower J_c values.

Order No.: JA711-021

Kinetics of the alignment and the formation of the Bi(2223) platelets in the powder-in-tube processed Bi(2223)/Ag composite tapes

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The alignment and the formation of $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10}$ inside an Ag sheath was investigated for tapes from four different precursor powders. Microstructural characterization along with *in situ* transmission x-ray diffraction experiments revealed that the kinetics depended strongly on the processing technique and the phase assemblage of the precursors. The alignment process was governed by the Bi-cuprates' preferential grain growth along the a-b plane and the constraint applied by the sheath. The formation mechanism of $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10}$ was either the intercalation or the nucleation and growth, depending on whether excessive liquid was adjacent to the platelets.

Order No.: JA711-022

Effect of filament architecture on magnetic flux distributions in multifilamentary $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$/Ag composites

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Multifilamentary $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$/Ag composites have been studied by a nondestructive magneto-optical imaging technique in order to determine the effect of filament architecture on the local magnetic flux distribution. The images reveal the homogeneity of the flux distribution in the upper layer filaments under magnetizing and demagnetizing conditions, and the alignment and morphology of these filaments in tapes with nine different composite structures. Certain types of filament arrangements led to homogeneous flux distributions, while other types caused localized inhomogeneities in the flux distribution. Nonuniform filament thickness also resulted in a highly inhomogeneous flux distribution. These results are useful in selecting optimal composite structures for power applications.

Order No.: JA711-023

AC losses of BSCCO-2223 superconducting monofilament and multifilament tapes at power frequencies

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The AC magnetic losses at power frequencies (60 Hz) were investigated for mono- and multifilament Ag-sheathed $(Bi,Pb)_2Sr_2Ca_2Cu_3O_y$ (BSCCO-2223) tapes with similar I_c values at 77 K. The multifilament sample exhibited higher losses than the monofilament under the same conditions. Loss peaks are discussed in terms of intergranular, intragranular and eddy current losses. Because of BSCCO's anisotropy, field orientation has a large effect on the magnitude of these peaks, even at relatively small angles. Losses for fields applied parallel to the *c*-axis of the textured BSCCO grains are larger by over an order of magnitude than those applied perpendicular.

Order No.: JA711-024

Vortex dynamics during N-S and S-N transitions of Y-Ba-Cu-O superconductors under the effect of temperature gradient and thermal cycling
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AC susceptibility has been measured simultaneously in three different ranges of Y-Ba-Cu-O ceramic samples in the presence of a large and variable temperature gradient. The results obtained for normal-superconducting or superconducting-normal transitions under the effect of the one-dimensional non-equilibrium temperature distribution reveal the vortex motion to consist of not only conventional flux expulsion (or flux penetration), but flux exchange too, appearing between different ranges of samples and between samples and their close physical environment. The thermal cycles are shown to represent a supplementary heat treatment, increasing the sample's homogeneity and decreasing the pinning, which accelerate the process of vortex motion.
Order No.: JA711-025

COMMUNICATIONS

Epitaxial growth of β-SiC thin films on a 6H-SiC substrate using the chemical solution deposition method
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A polyvinylmethylsilane precursor has been used for the epitaxial growth of SiC thin films on 6H-SiC single crystal substrates. The films were prepared by dipping the single crystalline 6H-SiC substrates into the precursor polymer solution with subsequent thermal treatments at different temperatures. Transmission electron microscopy (TEM) was used to characterize the microstructure and chemistry of the different SiC films. At 1100°C, the film was amorphous and contained substantial oxygen. At 1600°C, an epitaxial, single crystalline β-SiC film was observed.
Order No.: JA711-026

Pulsed electrodeposition of diamond-like carbon films
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Diamond-like carbon (DLC) films have been prepared by electrolysis of methanol solution using a pulse-modulated source. The deposition rate of the films is enhanced significantly compared to that of dc value. That the films do not contain bonded hydrogen is confirmed by infrared spectra. The structures of the films are characterized by Raman spectroscopy. These films show chemical inertness and hardness values in the range 12.5–19 GPa. Current-voltage characteristic of the films are measured, indicating that the resistivity is in the 10^7 Ω cm range and the breakdown field is larger than 1 MV cm^{-1}.
Order No.: JA711-027

Equation of state of polycrystalline $Ni_{50}Al_{50}$
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Polycrystalline $Ni_{50}Al_{50}$ suitable for high pressure studies was prepared by grinding and subsequent annealing of an inert-gas atomized alloy. The equation of state was determined by energy-dispersive x-ray diffraction in a diamond anvil cell to 25 GPa. The bulk modulus B_0 and the pressure derivative of the bulk modulus B'_0 were found to be B_0=156 ± 3 GPa and B'_0 = 4.0 ± 0.5.
Order No.: JA711-028

Nano-sclerometry measurements of superhard materials and diamond hardness using scanning force microscope with the ultrahard fullerite C_{60} tip
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The new procedure for the hardness measurements of superhard materials including diamond using the scanning force microscope with the ultrahard fullerite C_{60} tip was developed. It is shown that diamond is plastically deformed under the indentation by the ultrahard fullerite indenter at room temperature. Now the correct measurements of the diamond hardness have become possible. The hardness values measured are 137 ± 6 and 167 ± 5 GPa for the diamond faces (100) and (111), respectively.
Order No.: JA711-029

ARTICLES

Growth and characterization of semiconductor nanoparticles in porous sol-gel films
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Two methods for the preparation of semiconductor doped sol-gel films, for applications in nonlinear optics, have been studied and compared. In the first, porous films are spun from sols containing the cation precursor, and then reacted with H_2S gas, and in the second, the cation is adsorbed onto the pore surfaces of passive films from aqueous solution before the gas reaction. Extensive results for CdS doping are given, and preliminary results are reported for other semiconductor species. It is shown that a sputtered silica layer can seal the structure to allow further heat treatment without loss of dopant. The effects of heat treatment of doped films are described, and the limitation of crystallite growth by pore size is shown.
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Supercritical fluid infusion of silver into polyimide films of varying chemical composition
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Polyimides can be infused with silver complexes by the use of supercritical fluids. Highly reflective polyimide films were formed by infusing (1,5-cyclooctadiene-1,1,1,5,5,5-hexafluoroacetylacetonato)silver(I) [Ag(COD)(HFA)] into a number of polyimides and then thermally curing those films at 300°C for time intervals between 30 minutes and 3 hours. Reflectivities of the films exhibited strong dependence on the infusion and cure conditions as well as on the type of polyimide used. The highest reflectivity of 67.1% was achieved with a silvered film prepared from 3,3',4,4'-benzophenonetetracarboxylic acid dianhydride (BTDA) and oxydianiline (ODA) infused at 5000 psi, 100°C, for 30 minutes and cured for 1 hour at 300°C. Reflectivities of silvered surfaces of other polyimides investigated varied from 39% to 61%. A strong correlation between the presence of a ketonic group in the polyimide structure and the formation of mirror surfaces was detected.
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New hybrid nanocomposites based on an organophilic clay and poly(styrene-*b*-butadiene) copolymers
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The preparation, by direct melt intercalation, and the properties of new hybrid organic-inorganic nanocomposites, consisting of a commercial sample of poly(styrene-*b*-butadiene) copolymer (SBS) and a commercial organophilic clay containing the dioctadecyl dimethyl ammonium salt are described. In addition, several mixtures between the same copolymer and an unfunctionalized clay were prepared and studied. XRD spectra showed a partial insertion of the SBS block copolymer segments in the interlayers of the organophilic clay, accompanied by a loss of correlation within the layers. The degree of insertion increased by annealing the nanocomposites at 120°C for increasing time periods. No interaction between the polymer matrix and the unfunctionalized clay was found. The storage modulus value, in the plateau region comprised between the glass transition processes of the polybutadiene and of the polystyrene blocks, and the glass transition temperature of the polystyrene block domain increase as both the organophilic clay content and the annealing time increase. The glass transition process of the polybutadiene block domain is practically unaffected by the filler content and the annealing treatments. These data are most promising for upgrading the thermo-mechanical behavior and the application temperature window of the SBS thermoplastic elastomers through the preparation of nanocomposites employing suitably designed organophilic clays.
Order No.: JA711-032

Simulation of nacre with TiN/Pt multilayers and a study of their hardness

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TiN/Pt multilayers with individual thickness between 1 and 8 nm were prepared by ion beam sputtering deposition to simulate the micro-laminated architecture of nacre. Multilayer hardness and the laminated structure were investigated. It was found that sharp but incoherent interfaces were formed between individual layers. The multilayer hardness had strong dependence on layer arrangement. The range of layer thickness appropriate for high hardness was experimentally determined. Hardness enhancement of 30~70% was generally observed. With the layer thickness properly adjusted, the multilayer can even be harder than the hard component (TiN). Annealing experiment indicated that the hardness enhancement was an intrinsic property of the TiN/Pt multilayers.

Order No.: JA711-033

The thermopower of $Nd_{1+x}Ba_{2-x}Cu_3O_y$ in a comparative study of effects of Ba-site doping versus chain-Cu-site doping

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We report the results of measurements of resistivity up to 300 K, and of thermoelectric power up to 400 K, on ceramic samples of $Nd_{1+x}Ba_{2-x}Cu_3O_y$ with $0 \le x \le 0.65$. The samples were fully oxygenated; they were characterized by x-ray diffraction and iodometric titration. The results are compared with data from literature reporting on effects of substitutions on the Ba-site in "1-2-3" compounds and with our earlier experiments on substitution of Co for chain Cu. The focus is on the metal-nonmetal transition and on the nonmetallic regime in all these systems. T_c and the temperature dependence of S close to the metal-nonmetal transition and in the nonmetallic state, seem to be determined by a single parameter, irrespective of the nature of the dopant. This result and its implications on the electronic structure of $RBa_2Cu_3O_y$ (R = Y, or lanthanide ion) will be discussed.

Order No.: JA711-034

Crystallization behavior and microstructure of lithium-calcium aluminogermanate (LCAG) glasses

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The crystallization behavior and microstructure of lithium-calcium aluminogermanate (LCAG) glasses have been studied by using differential thermal analysis (DTA), x-ray diffraction (XRD), scanning electron microscopy (SEM), scanning transmission electron microscopy (STEM) and electron diffraction (ED). Uniform crystallization of the LCAG glass was found to result from two stages of heating processes. The kinetics of crystallization of the LCAG glasses was studied by DTA using the non-isothermal method. The activation energy for $3CaO \cdot Al_2O_3 \cdot 3GeO_2$ crystal growth was 693 kJ/mol. The precipitated crystals determined by XRD analysis were mainly $3CaO \cdot Al_2O_3 \cdot 3GeO_2$, and minor phases of $2CaO \cdot Al_2O_3 \cdot GeO_2$ and $Li_2O \cdot Al_2O_3 \cdot 2GeO_2$. Morphology and microstructure of the glasses after heat treatment determined by SEM and STEM techniques were presented. Crystallization was observed to start at the surface of the glass sample, and then proceed toward the interior of glass matrix. The morphology of $2CaO \cdot Al_2O_3 \cdot GeO_2$ is that of a subangular bell-shaped single crystal growing in a preferred orientation though the segregated phase matrix of fine dispersion of $3CaO \cdot Al_2O_3 \cdot 3GeO_2$ crystals. The $Li_2O \cdot Al_2O_3 \cdot 2GeO_2$ phase grows anisotropically in the fine fibrillar morphology and parallel to the $[\bar{3}31]$.

Order No.: JA711-035

Structural characterization of sputter-deposited $LaNiO_3$ thin films on Si substrate by x-ray reflectivity and diffraction

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X-ray reflectivity and diffraction were applied to characterize the highly (100)-textured thin films of $LaNiO_3$, which were deposited on Si substrate via radio frequency magnetron sputtering at temperatures ranging from 250 to 450°C. Two interference fringes of different period were observed from the reflectivity curves, and the fitting result indicates that in addition to the normal lanthanum-nickel oxide layer, a transition layer, which has a larger mass density than the previous one, exists in the sputter-deposited films. A comparison of the measured x-ray diffraction intensity with that calculated from layer thickness and mass density obtained from reflectivity data indicates that the transition layer is noncrystalline. The x-ray diffraction result also shows that there is a significant decrease of (100) diffraction intensity relative to that of (200) as increasing the deposition temperature. Using the reflectivity and diffraction data along with results of electron diffraction and film composition analysis from our other studies, such a change of relative intensity between the two diffraction peaks is attributed to the increasing content of two also highly textured La-rich phases, i.e., (110)-textured $La_4Ni_3O_{10}$ and (100)-textured La_2NiO_4, in addition to the $LaNiO_3$.

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Adhesion mechanisms of copper films deposited onto laser-irradiated alumina

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Strong adhesion between a deposited copper film and an alumina substrate takes place when the substrate is laser-irradiated prior to deposition. A post-deposition annealing is required to achieve the strong bonding. In this work, the interfacial region between the copper film and the alumina substrate was analyzed using Auger Electron Spectroscopy (AES). It was found that a transitional region is always present in couples that have a high adhesion strength, while little or no transitional region was found in weakly bonded couples. The transitional region depends on the laser irradiation atmosphere. In the case of laser irradiation in air, oxygen excess was found on the surface of the alumina substrate and in the copper/alumina couple the transitional region consists of a copper oxide and a Cu-Al double oxide. When the laser irradiation was performed in a reducing atmosphere (Ar-4% H_2), substoichiometric alumina and metallic aluminum were found on the surface of substrate and also a reaction between copper and the substoichiometric aluminum oxide was detected in the subsurface. Although the substoichiometric alumina is formed on the surface irradiated in Ar-4%H_2, a stable Al_2O_3 thin layer is formed on the outmost surface because the irradiated substrate is exposed to the atmosphere before deposition. This re-oxidized layer remains whole at the interface of the couple upon low temperature (at least up to 300°C) annealing, while it is ruptured upon higher temperature annealing (500°C in this work). In the latter case, the copper film can contact and react with the substoichiometric alumina formed in the subsurface of the substrate irradiated in the Ar-4%H_2 atmosphere. It is concluded that the Cu-Al-O interfacial compound formed in the transitional region causes the strong adhesion between the copper film and the alumina substrate.

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