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ABSTRACTS

COMMUNICATIONS

Improvement in thermoelectric properties of $(ZnO)_5In_2O_3$ through partial substitution of yttrium for indium

M. Kazeoka, H. Hiramatsu, W-S. Seo, K. Koumoto
(Nagoya University)

We first measured the thermoelectric properties of layer-structured homologous compounds, $(ZnO)_mIn_2O_3$ (m=integer), and reported that they would become candidate materials for high-temperature thermoelectric energy conversion. We further tried to improve their thermoelectric properties by partially substituting yttrium for indium in $(ZnO)_5In_2O_3$. Though the ionic radius of Y^{3+} is larger than that of In^{3+}, the a-axis (hexagonal system) elongated and c-axis shrank as Y was substituted for In. The thermoelectric properties were found to vary with varying amount of Y-substitution; 3% Y-substitution gave rise to the largest thermoelectric figure of merit, i.e. 1.1~1.3 x 10^{-4} K^{-1} at 960-1100 K. The abnormal change in the lattice structure by Y-substitution was responsible for the unusual behavior of the thermoelectric properties.

Order No.: JA803-001

Surface precipitates on single crystal $LiNbO_3$ after dry-etching by CHF_3 plasma

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The CHF_3 electron cyclotron resonance (ECR) plasma etched $LiNbO_3$ (LN) surface was analyzed chemically and crystallographically to investigate the dry-etch machining process for LN crystal, which was recently needed to obtain broader-band optical modulators. The etched surface was entirely covered with amorphous-like precipitates having an ~70 nm diameter. These precipitates (or a part of them) were thought to be LiF from Auger electron and x-ray photoelectron spectroscopes. The results indicated that the LiF was formed and remained on the etched surface while the Nb was almost completely removed.

Order No.: JA803-002

REVIEW

Stresses during thermoset cure

D.B. Adolf, J.E. Martin, R.S. Chambers, S.N. Burchett, T.R. Guess
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Production problems attributed to excessive stresses generated during the cure of epoxies led us to develop a formalism to predict these stresses. In our first studies, we developed a fundamental understanding of the complex evolution of viscoelasticity as the cure progresses. We then incorporated these results into a proper tensorial constitutive equation that was integrated into our finite element codes and validated using more complicated geometries, thermal histories, and strain profiles. The formalism was then applied to the original production problem to determine cure schedules that would minimize stress generation during cure. During the pursuit of these activities, several interesting and puzzling phenomena were discovered that have stimulated further investigation.

Order No.: JA803-003

ARTICLES

Evolution of surface structure, microstructure, and superconducting properties during $YBa_2Cu_3O_7$ a-axis thin film growth

A.F. Marshall
(Stanford University)

The evolution of surface structure, grain microstructure, and superconducting transition temperature of a-axis $YBa_2Cu_3O_7$ thin films has been studied as a function of film thickness. The grain size increases and develops a bimodal distribution of grain size during film growth, concurrent with an improvement in T_c. The surface structure does not represent the grain size, but rather the development of the bimodal grain structure follow the formation of the surface structure. The results are discussed in terms of thin film growth modes.

Order No.: JA803-004

Synthesis of thin superconducting $HgBa_2CaCu_2O_{6+\delta}$ films by post-annealing of laser-ablated precursors

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Superconducting $HgBa_2CaCu_2O_{6+\delta}$ (Hg-1212) thin films have been prepared by laser ablation followed by post annealing at high temperatures. Two synthesis methods were investigated, using (1) direct reaction of Hg-Ba-Ca-Cu-O precursor-films in a Hg-controlled ambience, and (2) thermal diffusion of Hg into Ba-Ca-Cu-O precursor-films in a controlled atmosphere containing both Hg- and Tl-bearing species. Effects of the annealing temperature, time, and bulk material composition on the Hg-1212 film and residual impurities are presented. Surface morphologies, growth defects and transport properties of Hg-1212 on $SrTiO_3$ substrates are discussed. Formation of Hg-1212 films on MgO and $LaAlO_3$ substrates is briefly described.

Order No.: JA803-005

Solidification of $YBa_2Cu_3O_{6+\delta}$: Part I. Morphology

H. Shen, M.C. Flemings, M.J. Cima, J. Haggerty, S. Honjo, K. Rigby, T.H. Sung
(Massachusetts Institute of Technology)

A new quenching technique was used for detailed microstructural examination of quenched $YBa_2Cu_3O_{6+\delta}$/liquid interfaces. The examination

revealed that the growth rate and the amount of excess Y_2BaCuO_5 (211) had a strong influence on the growth morphology of $YBa_2Cu_3O_{6+\delta}$ (123). The maximum growth rate at which single crystal growth could be obtained increased from 1 μm/s to 1.5 μm/s as excess 211 content increased from 0 to 20% (wt). It then decreased to 1 μm/s as excess 211 increased to 40% (wt). Dendritic growth with distinguishable secondary arms occurred for stoichiometric 123 samples in the regime of cellular/dendritic growth. A highly curved 123 envelope was formed on 211 particles located at the 123 growth interface for stoichiometric 123 samples in the regime of single crystal growth. The microscopic 123 growth interface became flat as excess 211 content increased to 20% (wt). The engulfment of 211 particles into 123 matrix is discussed based on detailed microstructural examination. It is found that the formation of a small highly curved 123 envelope on 211 particles for stoichiometric 123 samples is due to the large 211 particle spacing.

Order No.: JA803-006

High-temperature x-ray diffraction studies of a precursor mixture for Pb-substituted Bi-2223 superconducting wires

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High-temperature x-ray diffraction measurements of a (Bi,Pb)-2223 precursor mixture used to produce high-J_c superconducting tapes were conducted on silver and ZrO_2 substrates. The precursor mixture consisted primarily of 2212, Ca_2PbO_4, and CuO. Phase evolution was markedly sensitive to oxygen partial pressure: In 10% O_2 growth of the 2223 phase on silver was rapid, proceeded at the expense of the 2212 phase, and was preceded by the disappearance of the Ca_2PbO_4 phase. When slowly heated on a silver substrate in 7.5% O_2 the 2212 phase melted near 800°C and subsequently recrystallized near 820°C in a highly textured form, but with no detectable 2223 formation. Under similar conditions on a ZrO_2 substrate, the mixture exhibited no marked changes in the XRD patterns up to 850°C. The dramatic reactivity on silver was also highly dependent on heating rate; rapid heating in 7.5% O_2 to 825°C did not result in melting of the 2212 phase or appearance of the 2223 phase. In experiments leading to formation of 2223, the c-lattice parameter of the 2212 phase contracted just prior to the onset of formation of 2223. This result is consistent with the formation of an intermediate Pb-doped phase of 2212. A transient amorphous phase appeared briefly at the onset of formation of 2223. No evidence for intergrowth conversion of 2212 to 2223 was observed.

Order No.: JA803-007

Formation mechanism of Y_2BaCuO_5 pattern in growing $YBa_2Cu_3O_x$ grains during melt-infiltration process

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When a melt of BaO and CuO mixture was infiltrated into sintered Y_2BaCuO_5(211) compact to form $YBa_2Cu_3O_x$(123) superconductor, butterfly-like plane patterns of 211-free regions were observed to form within growing 123 grains. In a 123 grain, the 211-free region was found to be a pair of vertex-shared pyramids and 211 entrapped region to be the rest of the bulk of the grain. An observation of patterns and cracks formed within 123 grains revealed the base of the pyramids to be (001) plane. The difference in entrapment, which depends on crystallographic planes and results in the formation of the pattern, was explained by the dihedral angles between 123 and 211. The dihedral angle between a- (or b-) plane and 211, which is believed to be greater than zero degree, might cause the entrapment of 211 particles in an [100](or [010]) direction. In contrast, the dihedral angle of most probably zero degree between c-plane and 211 inhibited the entrapment. The observed shape of 211 particles in front of a- (or b-) and c-planes supports the above explanation of 211 entrapment to form the butterfly-like patterns.

Order No.: JA803-008

Growth and characterization of $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ single crystals

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$(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (2223) single crystals have been grown using a fused-salt reaction of Bi_2O_3, PbO, $SrCl_2$, $CaCl_2$, CuCl and KNO_3 in a KCl flux. The pristine crystals show regular plate-like morphology with typical dimensions of 0.1 x 0.1 x 0.001~0.01 mm^3. Crystal orientation, chemical composition, phase purity and superconductivity of the pristine crystals were determined by SEM, TEM, EDX, x-ray diffraction techniques and SQUID magnetometry. The relative fraction of the Bi-2223 phase (T_c= 110 K) in as-grown crystals is ~97%. The only impurity phase, Bi-2212, occurs in some selected crystals but is present in amounts less than the detection limit of x-ray diffraction and is unobserved in the diamagnetic signal determined by SQUID magnetometry.

Order No.: JA803-009

Effects of hydrogenated amorphous carbon interlayer on diamond nucleation

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Diamond was deposited at 850°C by microwave plasma CVD on the interlayers with various intensity ratios (I_D/I_G) of the D band (~1400 cm^{-1}) to the G band (~1570 cm^{-1}) in the Raman spectra. Diamond could be grown only on the interlayers with higher ID/IG[≥1.95] and N_d was slightly increased to $3x10^6/cm^2$ with I_D/I_G. The predeposition at 350°C, which decreased the full width at half maximum of the broad D band, further increased N_d to 5 x $10^7/cm^2$. With 300 Å Pt overlayer on the interlayer, N_d was much more enhanced to 8 x $10^7/cm^2$. We suggest that the sp^3 bonded carbon clusters within the interlayer contribute to diamond nucleation but they should be survived against atomic hydrogen etching during diamond deposition by increasing the sp^3/sp^2 ratio, by increasing the degree in clustering, or by protecting them with overlayer.

Order No.: JA803-010

Optical properties of nanometer-sized CdO organosol

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In this paper, nanometer-sized CdO organosol was prepared by using microemulsion methods. Its electronic structure and optical properties are characterized through UV-visible light absorption, photoluminescence and Z-scan techniques. Some new features were observed. Fluorescence and nonlinear optical responses are mainly related to surface trapped states.

Order No.: JA803-011

Characterization of superdislocation dissociations in $Al_{66}Ti_{25}Cr_9$ with transmission electron microscopy observations and image simulations

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The nature of dissociated superlattice dislocation cores in $Al_{66}Ti_{25}Cr_9$, deformed at room temperature, has been characterized by weak-beam transmission electron microscopy (TEM) and comparison of experimental images with computer simulated images. The displacement fields associated with narrowly dissociated APB- and SISF-dissociated <110> superdislocations were calculated to account for the asymmetry in dislocation contrast and led to a better understanding of the formation of images. Such calculations are a powerful aid, when coupled with image simulations, in distinguishing the "real" intensity peaks from the supplementary peaks that can be generated under experimental imaging conditions. While both APB- and SISF-dissociated superdislocations were identified, the vast majority of superdislocations were determined to be APB-associated. Corrected values of the fault energies (γ^{APB} and γ^{SISF}) have been measured for this alloy. These energies and the observed dissociations are shown to be self-consistent.

Order No.: JA803-012

Quantitative analysis of microstructures produced by creep of Ti-48Al-2Cr-2Nb-1B: Thermal and athermal mechanisms
M.A. Morris, M. Leboeuf
(University of Neuchâtel)

A γ-based TiAl alloy with equiaxed microstructure and fine grain size has been studied to analyze the deformation mechanisms responsible for the creep behavior. The microstructures produced by creep and high temperature deformation have been examined by TEM to obtain information about the different aspects characterizing the primary and secondary stages of creep. Mechanical twinning has been confirmed to occur in a fraction of the grains that never exceeds 50% while 1/2<110> dislocations are active within all the γ grains. The twins are only responsible for a small amount of strain but they lead to a subdivision of the microstructure and determine (directly or indirectly) the hardening process observed during the primary stage of creep. We have proposed that during the secondary stage the creep rate is determined by the unblocking of pinned dislocations by processes such as a pipe diffusion or cross-slip that allow thermally activated glide of 1/2<110> dislocations on (001) planes.
Order No.: JA803-013

Superplastic constitutive equation and rate-controlling process in aluminum matrix composites with discontinuous fiber and particle reinforcements
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Superplastic behavior of aluminum matrix composites with discontinuous reinforcements has been investigated in a temperature range below the melting temperature measured by differential scanning calorimetry. The experimental results of the mechanical properties revealed that the rate-controlling process of superplastic flow was associated with dislocation movement controlled by lattice self-diffusion. The strengthening due to the presence of reinforcements was retained. It is suggested that the strongest strengthening process of the dislocation pile-up mechanism and the diffusional relaxation limitation or dislocation by-pass mechanism affects the rate-controlling process.
Order No.: JA803-014

Microstructural development of $BaTiO_3$ heteroepitaxial thin films by hydrothermal synthesis
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The hydrothermal growth of epitaxial $BaTiO_3$ thin films on single crystal $SrTiO_3$ substrates occurs by the island growth mode. The aqueous solution chemistry is found to control interfacial characteristics and plays an important role in controlling film formation and faceting. Island faceting can be changed by the introduction of additional cations during synthesis. Electrophoretic data, confirmed by adsorption measurements, show that barium is a potential determining counterion and adsorbs on $SrTiO_3$ surfaces. Initial electrical measurements show that the $BaTiO_3$ films have a dielectric constant of 141 with a loss tangent of 0.9 that decreases with heat treatment.
Order No.: JA803-015

Semiconducting $BaTiO_3$ ceramic prepared by low temperature liquid phase sintering
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(Jozef Stefan Institute)

Donor-doped $BaTiO_3$ ceramics were prepared by adding $PbO{\cdot}B_2O_3{\cdot}SiO_2$ as a sintering aid. Semiconducting $BaTiO_3$ was obtained at a sintering temperature of 1100°C. The sintered samples exhibit the positive temperature coefficient of resistivity (PTCR) effect, which depends on the amount of liquid phase, the concentration of the donor-dopant and the sintering temperature. The cold resistivity of the samples decreases when the sintering temperature increases. The increase of the grain boundary resistivity and hence of the cold resistivity at lower sintering temperatures was explained by applying the diffusion grain boundary layer model.
Order No.: JA803-016

Preparation of ceria-gadolinia electrolytes by the tape rolling technique
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The preparation of thin (*ca.* 200 μm) $Ce_{0.9}Gd_{0.1}O_{1.95}$ planar electrolytes using the tape rolling technique is described. The processing and formation of these electrolytes involved several steps: (i) the production of a processable ceramic paste, (ii) rolling and cutting, (iii) binder burnout, and (iv) sintering. The rheology of these ceramic pastes is characterized by a hardness indentation test. The material parameters which may be obtained from the hardness indentation test such as the hardness (plastic flow stress), elastic modulus, and plasticity index are provided. The rheology, as characterized by the hardness method, of the pastes are shown to be influenced by the nature and extent of processing aids and also the mixing and milling times. Further, it is shown that by using a paste formulation with appropriate rheological properties, it is possible to produce uniform thin green tapes using a twin roll mill. The binder burnout characteristics of the ceramic pastes were studied by using a specially constructed thermal gravimetric apparatus. It is shown that the heating rate and the ambient atmosphere have strong influences upon the binder burnout kinetics of these green tapes. Finally, it is shown that sintered $Ce_{0.9}Gd_{0.1}O_{1.95}$ electrolytes, with a near theoretical density and with a uniform microstructure as well as a chosen near net shape, may be prepared from the corresponding tape rolled greens.
Order No.: JA803-017

The influence of direct current bias on the initial aging of a doped lead magnesium niobate ceramic
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The initial dielectric aging behaviors of a Mg and Mn doped lead magnesium niobate ceramic were investigated over a wide range of direct current (dc) bias. Both the dielectric constant–log(time) and the loss tangent–log(time) were regressed in terms of a linear relationship. The dc bias is found to have a strong influence on the dielectric parameters at the start of aging and to suppress the aging of dielectric constant and loss tangent. The frequency dependence of the dielectric aging is also evidently affected by the dc bias.
Order No.: JA803-018

Combustion synthesis of BN powder
C-C. Hwang, S-L. Chung
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A SHS process developed in our previous study for the synthesis of Si_3N_4 has been tested for the synthesis of BN powder. Boron and NaN_3 powders were used as the reactants. In addition to ammonium halides, KHF_2, $FeCl_3$, $NaNH_2$ or a mixture of 50 mol% $FeCl_3$ and 50 mol% $NaNH_2$ was added to the reactants to examine their catalytic effect. These powders were mixed and pressed into a cylindrical compact. The compact was wrapped up with an igniting agent (i.e., $Mg+Fe_3O_4$) and the synthesis reaction was triggered by the combustion of the igniting agent. It was found that only those reagents containing both halogen and hydrogen can exert effectively the catalytic effect. The BN powder as synthesized is mostly in the form of agglomerated fine particles (0.1~1 μm in diameter) and is hexagonal in crystalline structure. Effects of various experimental parameters on the product yield were investigated. A possible reaction mechanism was proposed, which explains the effects of the experimental parameters on the synthesis reaction.
Order No.: JA803-019

A novel Cu(II) chemical vapor deposition precursor: Synthesis, characterization and chemical vapor deposition
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A non-fluorinated, β-diketonate precursor, bis(t-butylacetoacetato)Cu(II) or $Cu(tbaoac)_2$, was synthesized by modifying bis(dipivaloylmethanato)Cu(II) or $Cu(dpm)_2$ for chemical vapor deposition of copper. The complex was characterized by a variety of techniques, such as melting point determination, mass spectroscopy, infrared spectrometry, elemental

analysis, thermogravimetric and differential thermal analysis and x-ray diffraction. $Cu(tbaoac)_2$ has a higher sublimation rate than $Cu(dpm)_2$ over the temperature range 90-150°C. Pyrolysis of $Cu(tbaoac)_2$ leads to the formation of copper films at 225°C, compared to 330°C for $Cu(dpm)_2$. As-deposited copper films were highly dense, mirror-bright, adhered strongly to SiO_2 and showed a resistivity of less than 2.9 μΩ-cm at a thickness as low as 1300 Å. A possible mechanism for the decomposition of the ligand tbaoac has been proposed.

Order No.: JA803-020

High temperature epitaxial growth and structure of Nb films on α-Al_2O_3(0001)

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Epitaxial Nb thin films were grown via molecular beam epitaxy (MBE) at different substrate temperatures on α-Al_2O_3(0001) substrates. For temperatures of 900°C to 1100°C, it was found that Nb grows in the Volmer-Weber growth mode (formation of three-dimensional crystallites. Depending on the growth temperature, different epitaxial orientations of Nb films can be found. At a growth temperature of 900°C, the Nb{111} planes are parallel to the sapphire basal plane whereas at 1100°C the Nb grows with the {110} planes on the basal plane of sapphire. These orientations are present even in the initial stages of growth at both temperatures. The formation of two different epitaxial orientations of thick Nb films can only be conclusively explained by considering both the change in the total density of Nb islands with temperature and the influence of island size on the total energy of the islands. The Nb island growth process has been investigated in-situ using reflection high energy electron diffraction (RHEED) and Auger electron spectroscopy (AES). Scanning electron microscopy (SEM), x-ray diffraction (XRD) and transmission electron microscopy (TEM) were employed to determine the morphology and structure of Nb islands, Nb films and Nb/α-Al_2O_3 interfaces.

Order No.: JA803-021

Passivation of nanocrystalline Al prepared by the gas phase condensation method: An x-ray photoelectron spectroscopy study

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Nanocrystalline aluminum powders have been prepared by the gas phase condensation method. Samples have been synthesized in a conventional preparation chamber for gas phase condensation and also in the pretreatment chamber of an x-ray photoelectron spectroscopy (XPS) spectrometer so that "in-situ" studies of the passivation process of nanocrystalline aluminum can be performed. For the range of particle sizes (12–41 nm) studied in the present work we found a universal behavior during passivation with oxygen of the nanocrystalline Al^0. An Al_2O_3 overlayer of 4 nm, that protects the material from further oxidation, was obtained for all samples independently of the route of oxygen dosage. A careful analysis of the photoelectron parameters (binding energy and Auger parameters) for Al and O shows that in the early stages of passivation the alumina over-layer is so thin (<2.5 nm thickness) that the Al_2O_3-Al interface induces an increase in the relaxation energy of the photoholes as compared to that of bulk alumina. Conclusions have been drawn about the best way to proceed during passivation of Al ultrafine particles before exposure to an air atmosphere.

Order No.: JA803-022

On the reactivity of organic solvents on the particle surface of metal pigment

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For very fine, acicular particles of α-Fe prepared for audio/video magnetic, the recording media relationship between their chemical property and surface flatness of the tape material made from the particles was investigated experimentally.

It was found that adsorption capacity of acidic resin on the particle surface can be a very good index to predict the flatness of the magnetic tape. Because this index depends strongly on acidic and basic property of the particle surface, the tape performance can be well controlled by designing chemically the particle surface of α-Fe.

Order No.: JA803-023

Sub-part-per trillion detection and analysis of submicrometer particles in integrated circuit processing chemicals

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We have developed a new technique for the analysis of particulate contaminants in liquids (or gases) with an elemental detectability limit as low as 0.1 parts per trillion, and a particle concentration detectability limit as low as 1 particle/ml for particles greater than 0.2 μm in diameter. Samples are prepared using extraction replication and analyzed using analytical transmission electron microscopy. The methodology has been applied to the analysis of H_2O_2 and HF, important chemicals in integrated circuit fabrication. The new methodology should become an important tool in the identification of submicron-sized particles which adversely affect integrated circuit fabrication.

Order No.: JA803-024

TiN prepared by plasma source ion implantation of nitrogen into Ti as a diffusion barrier for Si/Cu metallization

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A method of forming TiN films for Si/Cu metallization by using plasma source ion implantation (PSII) of nitrogen into Ti is described. The PSII process utilizes a dose of 1×10^{17} ions/cm^2 and peak voltages of -10, -15 and -20 kV. The properties of such TiN films as diffusion barriers between Cu and Si were investigated by annealing Cu(2000 A)/TiN/Ti/Si films in vacuum from 500°C to 700°C, and by analyzing with four-point probe sheet resistance measurements, Rutherford backscattering spectrometry (RBS) and Auger electron spectroscopy (AES). The TiN films made at peak voltages of -15 and -20 kV were stable barriers against Cu diffusion after annealing at temperatures higher than 600°C.

Order No.: JA803-025

Spinning deposition of silica and silica-titania optical coatings: A round robin test

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A round robin test has been performed on sol-gel processing for the deposition of silica and silica-titania films on silicon substrates by spin-coating. Three solution preparation processes for silica coatings and three for silica-titania coatings were used to prepare samples at each of the participating laboratories. The films have been characterized mainly by thickness (profilometry and ellipsometry measurements), refractive index, porosity and optical scattering. Different processes gave different thicknesses. Thickness differences were found in films prepared by the same process and by the same deposition parameters, but in different laboratories, when heat treated at 500°C. Variations were reduced in samples annealed at 1000°C. Refractive index and porosity measurements suggest that variations were due to structural differences, particularly porosity. Furthermore, films heat treated at 500°C were not completely stabilized, and showed index and porosity variations after six months.

Order No.: JA803-026

An x-ray photoelectron spectroscopic study of the chemical states of fluorine atoms in calcium silicate glasses

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X-ray photoelectron spectroscopy has been used to examine the chemical states of fluorine in the glasses of composition $xCaF_2 \cdot (50-x)CaO \cdot 50SiO_2$ (x = 5, 10, 15, 20, 25 mol%) and $xCaF_2 \cdot (50-x/2)CaO \cdot$

$(50-x/2)SiO_2$ (x = 5, 10, 15, 20 mol%). The analysis of the F 1s spectra indicated that Ca^{2+} and F^- ions introduced as CaF_2 are favorably located among the Si-O skeleton forming Ca-F clusters. The fraction of the bridging and non-bridging oxygen atoms was derived from the O 1s spectra, and the network of the fluorine-containing glasses was concluded to depend only on the ratio CaO/SiO_2.

Order No.: JA803-027

Accuracy in the experimental calorimetric study of the crystallization kinetics and predictive transformation diagrams: Application to a Ga-Te amorphous alloy

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The uncertainties inherent to experimental differential scanning calorimetric data are evaluated. A new procedure is developed to perform the kinetic analysis of continuous heating calorimetric data when the heat capacity of the sample changes during the crystallization. The accuracy of isothermal calorimetric data is analyzed in terms of the peak-to-peak noise of the calorimetric signal and base line drift typical of differential scanning calorimetry equipment. Their influence in the evaluation of the kinetic parameters is discussed. An empirical construction of the time-temperature and temperature heating rate transformation diagrams, grounded on the kinetic parameters, is presented. The method is applied to the kinetic study of the primary crystallization of Te in an amorphous alloy of nominal composition $Ga_{20}Te_{80}$, obtained by rapid solidification.

Order No.: JA803-028

Layered mixed tin-titanium phosphates

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Mixed crystalline tin-titanium phosphates with variable tin to titanium molar ratio have been prepared by precipitation of soluble salts of the metal(IV) with phosphoric acid and refluxing the amorphous solids in 17 M H_3PO_4. The new materials are characterized by chemical textural and thermal analysis and x-ray powder diffraction. The tin-titanium phosphates are solid solutions showing an isomorphic substitution of tin by titanium in the α-tin phosphate lattice and tin substitution in the γ-titanium phosphate lattice. In both cases, the solubility is partial. The coexistence of both saturated phases is observed in samples of composition between the solubility limits.

Order No.: JA803-029

Preparation of refractory carbide fibers by thermal decomposition of transition metal (Ti,Zr,Hf,Nb,Ta) alkoxide-cellulose precursor gel fibers

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Gel fibers have been prepared by extruding cellulose acetate spinning solution into transition metal (Ti,Zr,Hf,Nb,Ta) acetone solution as a coagulation bath. Gel formation must be due to the coordination of metal to OH and CO groups on the pyranose ring. The resultant gel fibers have been converted into carbide fibers by pyrolyzing them in Ar and N_2 atmospheres at temperatures lower than for powder processing. This precursor gel can give a molecular scale mixture of metal and carbon sources.

Order No.: JA803-030

Texture development in Bi_2Te_3 during hot forging

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The development of crystallographic texture in hot-forged polycrystalline Bi_2Te_3 samples was studied. Texture was evaluated with the use of the March-Dollase model in conjunction with a Rietveld analysis of x-ray diffraction data. It was determined that during forging a strong (0001) texture develops along the loading axis. The magnitude of the (0001) texture increases systematically with the amount of height reduction during hot-forging. The correlation between the observed deformation and the March-Dollase texture model suggests that grain rotation is the primary mechanism for texture development in Bi_2Te_3.

Order No.: JA803-031

In-depth variations of diamond structures on Pt(111) investigated by confocal Raman spectroscopy

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We have characterized heteroepitaxial diamond films on Pt(111) using the non-destructive technique of confocal Raman spectroscopy to investigate the variation in structure and strain with depth. The spectral depth profiles of heteroepitaxial diamond showed the diamond peak at 1332-1335 cm^{-1} and four bands centered at 1230 cm^{-1}, 1470-1490 cm^{-1}, 1530-1580 cm^{-1}, and 1640 cm^{-1} near the surface. The diamond peak shifted to the single crystal peak position at 1332 cm^{-1} as the linewidth was broadened with free surface proximity. The compressive strain in the heteroepitaxial diamond crystal decreased and turned into the random strain. At the same time, the Raman band at 1470-1490 cm^{-1} grew in intensity. The constituents of nondiamond phase in the heteroepitaxial growth regions are different from those formed in the randomly oriented regions.

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Microstructure characterization of one directionally oriented ulexite

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Microstructures of ulexite were investigated by CTEM and low electron dose HREM. It was found that the longitudinal grains in ulexite were oriented to c-direction to form a bundle structure. There were a number of small angle grain boundaries and stacking faults inside a grain in the ulexite. Cleavage microcracks and stacking faults were mostly introduced on the {010} of the ulexite. The high angle grain boundaries consisted of high coincident boundaries, which was confirmed by a comparison of observed contact angles and calculated degree of coincidence at the boundaries. The light transmittance properties of the ulexite would depend on the defects such as stacking fault, small angle grain boundary, and high angle grain boundary.

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Synthesis of a bulk amorphous alloy by consolidation of the melt-spun amorphous ribbons under high pressure

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Based on the experimental observation that high pressure will considerably enhance the crystallization onset temperature of amorphous alloys, an attempt was made to consolidate the melt-spun amorphous ribbons into fully-densed three-dimensional bulk amorphous materials under high pressures. An amorphous $Ni_{69}Cr_7Fe_{2.5}Si_8B_{13.5}$ (at.%) alloy was used as a model material. Under a pressure of 1.5 GPa, the crystallization onset temperature was found to be increased by about 40 K, resulting in a widened supercooled liquid temperature region (about 68 K) beneath the onset of crystallization The high pressure consolidation of the amorphous ribbons in this temperature region yielded bulk amorphous compacts with the same density of the melt-spun ribbons. This achievement was attributed to the significant homogenous viscous flow of materials in the supercooled liquid state that could be maintained at higher temperatures during the high pressure compaction.

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