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ABSTRACTS

COMMUNICATION

From small angle x-ray scattering to reflectivity: Instrumentation and sample study
D.W. Hua*, G. Beaucage+, M.S. Kent#
*(*University of New Mexico, +University of Cincinnati, #Sandia National Laboratory)*

In this study, we describe the first results from an x-ray reflectometer which has been modified from an existing Kratky small angle x-ray scattering camera at the UNM/Sandia scattering center. Typically, 7 orders of magnitude of reflectivity can be obtained over a range of 0.02 to 0.5 $Å^{-1}$ in q. This allows the resolution of surface features of 10 to 1000 Å. The conversion to reflectometer is reversible and can be achieved in a short time allowing for dual use of an existing Kratky camera.
Order No.: JA602-001

ARTICLES

Phase development of Bi-2212 superconductor: A time-resolved neutron powder diffraction investigation
D.N. Argyriou, J.A. Garcia, J.F. Mitchell, J.D. Jorgensen, D.G. Hinks
(Argonne National Laboratory)

Time resolved *in-situ* neutron powder diffraction and Rietveld refinement have been used to study the synthesis of Bi-2212 from hydroxide precursors in a 2% O_2 atmosphere. Bi-2212 was found to form within the temperature range 770-800°C. Studies at 800°C show that Bi-2212 rapidly grows at the expense of Bi-2201. Upon lowering the temperature to 500°C and changing the atmosphere to Ar, a rapid increase in the lattice parameters was observed. We attribute this change to the loss of oxygen from the Bi-2212 lattice. The final material exhibited a T_c of 94 K.
Order No.: JA602-002

Interface structure of a $YBa_2Cu_3O_{7-x}$ /N/ $YBa_2Cu_3O_{7-x}$ superconductor/normal metal/superconductor Josephson junction using $YBa_2Cu_{2.79}Co_{0.21}O_{7-x}$ as the normal barrier N
S. Rozeveld*, K.L. Merkle*, K. Char+
*(*Argonne National Laboratory, +Conductus, Inc.)*

Superconductor-normal-superconductor (SNS) edge junctions consisting of $YBa_2Cu_3O_7$ / $YBa_2Cu_{2.79}Co_{0.21}O_{7-x}$ / $YBa_2Cu_3O_7$ were fabricated on (001) YSZ substrates using laser deposition. In contrast to other SNS junctions, e.g. with $La_{0.5}Sr_{0.5}CoO_3$, $CaRuO_3$ or $SrRuO_3$ as the barrier layer, these devices do not display an excess normal-state resistance. High-resolution and conventional transmission electron microscopy (TEM) techniques were employed to investigate the SN interface structure and possible interface defects. Results are compared to recent TEM investigations of $CaRuO_3$ SNS junctions.
Order No.: JA602-003

Numerical calculation of the crystal rotation effect on $YBa_2Cu_3O_{7-x}$ single crystal growth by the pulling method
Y. Namikawa, M. Egami, Y. Shiohara
(Superconductivity Research Laboratory-ISTEC)

Series of numerical calculations of convection were performed for the $YBa_2Cu_3O_{7-x}$(Y123) single crystal growth by the modified pulling method (Solute Rich Liquid Crystal Pulling method; SRL-CP method). The finite-difference method was used to calculate the steady state of the axisymmetric 2-dimensional incompressible viscous fluid system. Effect of the crystal rotation on the flow pattern and the temperature distribution in the melt was studied. Increase of the crystal diameter and/or the crystal rotation rate increased the strength of the forced convection in the melt, and as a result, the temperature at the crystal growth interface increased. These results were consistent with the experimental results.
Order No.: JA602-004

Air oxidation of undoped and B-doped polycrystalline diamond films at high temperature
K. Miyata, K. Kobashi
(Kobe Steel, Ltd.)

Air oxidation of undoped and B-doped polycrystalline diamond films was investigated at temperatures between 500 and 700°C. Diamond (111) facets were etched for both undoped and B-doped films after one hour at 700°C. The etching rate of (111) facet due to oxidation was approximately 50% lower by B-doping of $1x10^{19}$ cm^{-3}, presumably because of the decrease of sp^2 bands and lattice defects that were identified by Raman and photoluminescence spectroscopy. X-ray photoelectron and electron energy loss spectroscopy revealed that by the high temperature treatment, the diamond surface was initially converted into graphite and successively etched by oxygen.
Order No.: JA602-005

Surface and optical properties of porous silicon

S.M. Prokes
(Naval Research Laboratory)

Although silicon is the material of choice in the semiconductor industry, it has one serious disadvantage: it is an extremely poor optoelectronic material. This is because it is an indirect gap semiconductor, in which radiative transition results in extremely weak light emission in the infrared part of the spectrum. Thus, the discovery of strong visible luminescence from a silicon based material (porous silicon) has been quite surprising and has generated significant interest, both scientific and technological. This material differs from bulk silicon in one important way, in that it consists of interconnected silicon nanostructures with very large surface to volume ratios. Although the first mechanism proposed to explain this emission process involved carrier recombination within quantum size silicon particles, more recent work has shown that the surface chemistry appears to be the controlling factor in this light emission process. Thus, the aim of this work is to outline the data and arguments which have been presented to support the quantum confinement model, along with the shortcomings of such a model, and to examine more recent models in which the chemical and structural properties of the surface regions of the nanostructures have been incorporated.

Order No.: JA602-006

Characterization of InGaAsP materials by ultra high intensity post ionization mass spectrometry: Relative sensitivity factors for zinc vs. bulk constituents

M.L. Wise, S.W. Downey, A.B. Emerson
(AT&T Bell Laboratories)

The first relative sensitivity factors (RSF) for detecting the major and dopant elements of optical materials by ultra high intensity post ionization (UHIPI) mass spectrometry are determined. The post ionization is performed using a single laser wavelength with intensities greater than 10^{14} W/cm^2. Zn-implanted InP and $In_{0.4}Ga_{0.1}As_{0.3}P_{0.2}$ are used to investigate the photoionization of sputtered atoms and molecules. Under optimal conditions, the UHIPI RSF's for atomic singly charged In, Ga, and Zn are nearly equal, that is, the ratio of UHIPI signals is equal to the concentration ratio. In principle, no standards are needed for quantitative analysis. Arsenic and P, with higher ionization potentials, are not detected as efficiently as other elements. The detected mass balance is usually group III rich. An entire mass spectrum is necessary for complete characterization of all elements and adjustment of their RSF's because many sputtered molecules are detected containing the group V elements. Multiple charged species comprise about 10% of the detected ions.

Order No.: JA602-007

X-ray diffraction and x-ray photoelectron spectroscopy study of the Ru-Cu/SiO_2 system prepared by low temperature reduction: Occurrence of a metastable amorphous or nanocrystalline phase

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Bimetallic copper-ruthenium catalysts supported on silica were prepared by the reduction of the metallic salts in aqueous solution at room temperature. The concentration of the two metal components was selected to span the entire range of composition. In spite of the known immiscibility for the copper-ruthenium equilibrium phase diagram, x-ray diffraction (XRD) measurements combined with x-ray photoelectron spectroscopy (XPS) data indicate that this method of preparation is able to produce nanocrystalline extended solid solutions and/or amorphous metastable phases. In the case of ruthenium-rich compositions, the hexagonal close packed ruthenium crystallites are covered by copper atoms which grow with the same hcp sequence of the ruthenium core. For intermediate compositions a nanocrystalline and/or amorphous phase is observed, while in the case of copper-rich samples a single-phase fcc extended solid solution is found. The surface composition of the samples appears systematically enriched with Cu, as obtained from XPS semiquantitative results. The phenomena of phase separation and growth induced by thermal annealing at 870 K are also presented and discussed.

Order No.: JA602-008

Phase identification in a U-Zr/Ni-Cr diffusion couple using synchrotron radiation

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(Argonne National Laboratory)

The diffusion zone between a U-23 at.% Zr alloy and a Ni-16.4 at.% Cr alloy exhibited nine distinct phase layers, many of which were mixtures of two phases. Four single-phase regions were less than 10 μm wide. To identify these phases by diffractometry, a synchrotron x-ray beam was collimated by a 50 μm by 1 mm slit. This beam was translated across the sample to obtain diffraction patterns throughout the diffusion zone. In this way, only a few phases were simultaneously within the beam, easing identification of the phases. Strains in the lattice due to solid solution were also observed. These micro-diffraction techniques are applicable to a wide range of material systems.

Order No.: JA602-009

Fullerenic nanostructures in flames

K. Das Chowdhury, J.B. Howard, J.B. VanderSande
(Massachussetts Institute of Technology)

High-resolution transmission electron microscopy (HRTEM) was used to characterize nanostructures in soots produced in flames of benzene, acetylene or ethylene premixed with oxygen and an inert diluent gas. The nanostructures ranged from ~2 nm to ~30 nm in size with a hollow core measuring about ~1 nm to ~10 nm in diameter and containing 5 to 20 shells. The shapes of the nanostructures included spherical, spheroidal, tubular and trigonous.

Order No.: JA602-010

Effects of La_2O_3 and MnO_2 on the piezoelectric properties of $0.02Pb(Y_{2/3}W_{1/3})O_3$-$0.98Pb(Zr_{0.52}Ti_{0.48})O_3$

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The effects of the addition of La_2O_3(0.1 wt%~1 wt%) and MnO_2(0.1 wt%~1 wt%) on the piezoelectric properties of $0.02Pb(Y_{2/3}W_{1/3})O_3$-$0.98Pb(Zr_{0.52}Ti_{0.48})O_3$ system were investigated to develop the composition available for actuator. In the case of the addition of La_2O_3 to the system, the maximum values of d_{33} and k_p were observed in the 0.1 wt% La_2O_3, and the values of Q_m did not change with the amount of La_2O_3. The values of d_{33}, k_p, and Q_m were $428x10^{-12}$C/N, 57%, and 71, respectively. On the other hand, the introduction of 0.5 wt% MnO_2 as an acceptor into the system resulted in the maximum Q_m value of 741, exhibiting a d_{33} of $298x10^{-12}$C/N, and a k_p of 50%. In the case of the simultaneous addition of La_2O_3 and MnO_2, the best piezoelectric properties are obtained from the composition of $0.02Pb(Y_{2/3}W_{1/3})O_3$-$0.98Pb(Zr_{0.52}Ti)O_3$ + 0.1 wt% La_2O_3 + 0.3 wt% MnO_2. The values of d_{33}, k_p, and Q_m were $345x10^{-12}$C/N, 55%, and 741, respectively.

Order No.: JA602-011

Sintering kinetics of silica-titania sol-gel films on silicon wafers

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The sintering behavior of 80 SiO_2-20 TiO_2 sol-gel thin films on Si wafers, prepared by spin coating, was studied by the calculation of

density as a function of temperature, from refractive index measurements and the Lorenz-Lorentz relationship. The sintering kinetics of the films were fit to the Mackenzie and Shuttleworth model, over the temperature range of 700-850°C. Using this model, the viscosity was determined as a function of temperature. These gel films sintered to full density at 850°C.
Order No.: JA602-012

Rapid consolidation processing of silicon nitride powders
J.A. Schneider, S.H. Risbud, A.K. Mukherjee
(University of California at Davis)

Using a plasma assisted sintering (PAS) process, submicron size, silicon nitride powders were consolidated to >99% of the theoretical density (TD) at 1750°C in less than 5 minutes with retention of the α phase and the submicron grain size. The silicon nitride powders were sintered with 5 wt% Y_2O_3 and 5 wt% Y_2O_3 + 5 wt% $MgAl_2O_4$ additives. The PAS processing method for the silicon nitride additive mixtures is attractive for retention of fine grained microstructures favorable for superplastic deformation. Post superplastic forming heat treatments to transform the α-Si_3N_4 to lath-like, creep-resistant β-Si_3N_4 is another feature of the present processing method.
Order No.: JA602-013

Preparation and characterization of GeS_2
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Synthesis of GeS_2 via a sol-gel process using germanium ethoxide and hydrogen sulfide in toluene resulted in a gel aggregate with an apparent Ge/S ratio 1:1:8. Special precautions were necessary to protect the reaction mixture from water contamination which produced GeO_2. Results indicated that the main source of water was the hydrogen sulfide gas. Heat treatment of the produced GeS_x gel yielded a product with Ge/S ratio 1:2:3. The sol-gel prepared materials and their heat-treated products were characterized by various methods.
Order No.: JA602-014

Characterization of boron nitride thin films prepared from a polymer precursor
V. Z-H. Chan, J.B. Rothman, P. Palladino, L.G. Sneddon, R.J. Composto
(University of Pennsylvania)

Excellent quality boron nitride (BN) thin films on silicon have been produced by a simple procedure involving spin-coating solutions of the "single-source" polymeric precursor polyborazylene, $(B_3N_3H_{-4})_x$, on a silicon substrate, followed by pyrolysis at 900°C. Rutherford backscattering spectrometry (RBS) indicates that the B/N ratios are 1.37 and 1.09 for conversions carried out in a vacuum oven at 900 and 1250°C, respectively. Forward recoil spectrometry (FRES) showed that the atomic percent of residual hydrogen is 10 and 9%, respectively. Plain-view and cross-sectional scanning electron microscopy (SEM) studies showed that the samples annealed at 900°C were clean and uniform in thickness. A thickness of $800x10^{15}$ atoms/cm^2 was determined by ion scattering. Films annealed to 1250°C likewise showed a continuous unbroken boron nitride layer, but also exhibited morphological features resulting from reactions of the underlying silicon oxide-silicon interface in the substrate. Auger electron spectroscopy and atomic force microscopy showed that the BN coating produced at this higher temperature remained unbroken but had a surface area of ~15% covered by dimples 2-7 nm in depth. Compared to typical films made by chemical vapor deposition, BN films produced from this "single-source" method have lower hydrogen and carbon concentrations.
Order No.: JA602-015

Metal-organic deposition of thin-film yttria-stabilized zirconia-titania
K.E. Swider, W.L. Worrell
(University of Pennsylvania)

Mixed-conducting yttria-stabilized zirconia-titania (YZTi) has attractive applications in solid oxide fuel cells (SOFCs) and electrocatalysis, particularly when used as a thin film to reduce its electrical resistance. Thin films of yttria (12 mol %) stabilized zirconia-titania (8 mol %) have been prepared using metal-organic deposition (MOD) whereby metal-organic solutions of Zr-, Y-, and Ti-2-ethylhexanoates are spun onto suitable substrates. Variables affecting the film surface-morphology, chemistry, and crystal structure are examined using scanning electron microscopy (SEM), auger electron spectroscopy (AES), and x-ray diffraction (XRD), respectively. Uniform, pore-free films having a low carbon content (<1 at%) are made by sintering on a hot plate at 530°C. The effect of thermal cycling on the chemical compatibility and adherence is examined for YZTi films on yttria-stabilized zirconia substrates.
Order No.: JA602-016

Size distribution of ultra-fine particles in KDP aqueous solutions
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Quantitative and qualitative investigations on the size distribution of ultra-fine particles in KDP solutions were performed by a laser light scanning particle counter and by comparing with scattering polystyrene particles of standard size. The ultra-fine particles are of a size distribution from smaller than 70 nm to 2000 nm, and the density of the particles sharply decreased with increasing particle size. Most of them were smaller than 100 nm, and almost no particles were larger than 1000 nm. The size of the visual particles, which are distinguished individually by a laser light-scattering technique[3], were estimated in the size range from 200 nm to 1000 nm. The reliability of the results was evaluated.
Order No.: JA602-017

Chemical influence of inert gas on the thin film stress in plasma enhanced chemical vapor deposited a-SiN:H films
M.J. Loboda, J.A. Seifferly
(Dow Corning Corporation)

The growth of amorphous hydrogenated silicon nitride (a-SiN:H) films by plasma enhanced chemical vapor deposition (PECVD) of SiH_4-NH_3-N_2 reactive gas mixtures has been studied. Films were deposited at low temperature (T<250°C) in a commercial PECVD system commonly used to grow a-SiN:H for semiconductor integrated circuit passivation. It has been observed that the stress of the a-SiN:H film can be controlled through dilution of the film precursors with an inert gas. Experiments indicate that the influence of the inert gas on the process extends from growth kinetics and plasma chemistry to hydrogen bonding, elemental composition and biaxial elastic modulus. The stress in films deposited without dilution is tensile. When argon is added to the plasma, Si-H_x plasma chemistry and film hydrogen bond density change producing a reduction in the amount of tensile stress. Dilution with helium can be used to shift the film stress from tensile to compressive with minimum change in growth rate. The observed helium/film stress relationship is associated with helium-based Penning ionization processes, which create metastable reactive gas species. In turn, the metastables influence nitrogen and hydrogen incorporation into the film. Nitrogen incorporation produces volume expansion of the film, increasing the compressive character of the film stress. This effect is similar to that observed when the RF power is varied or when low or multi-frequency plasma excitation is used during PECVD growth of a-SiN:H.
Order No.: JA602-018

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Characterization of highly oriented (110) TiN films grown on epitaxial Ge/Si(001) heterostructures

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(North Carolina State University)

The characteristics of epitaxial growth of titanium nitride films on Ge/Si(001) have been studied. The growth of titanium nitride and germanium films on (001)Si was carried out *in-situ* in a high-vacuum chamber ($<10^{-7}$ torr) using a multi-target stage in a pulsed laser deposition system. Electrical resistivity, stoichiometry, crystallinity and epitaxial relationships as a function of deposition temperature have been studied. Electrical resistivity of the titanium nitride films grown at deposition temperatures in the range of 450-750°C was measured using a four-point probe. The stoichiometry of these films was investigated using Auger electron spectroscopy and Raman spectroscopy. The crystalline quality and epitaxial nature of TiN films grown at different substrate temperatures were characterized using x-ray diffraction and transmission electron microscopy. Highly oriented titanium nitride films with (110) orientation were obtained on Ge(001) film when the substrate temperature was maintained between 550°C and 650°C. The epitaxial growth of the titanium nitride films was found to be a function of two-dimensional or three-dimensional growth of germanium film on silicon (001) substrate. Titanium nitride films grown at a substrate temperature of 650°C exhibited the lowest room temperature resistivity (26 μΩ-cm), highest nitrogen content (close to stoichiometry), and the best epitaxiality with the Ge(001) films on Si(001). The epitaxial relationships for the TiN/Ge/Si(001) heterostructure are found to be: [001]TiN || [110]Ge || [110]Si and [$\bar{1}$10]TiN || [1$\bar{1}$0]Ge || [1$\bar{1}$0]Si. To explain the epitaxial growth in a large mismatch system (~28%) such as TiN/Ge(001), domain matching mechanism is proposed. Domains of size 4x(001)TiN by 17x(2$\bar{2}$0)TiN in the titanium nitride film match closely with domains of size 3x(220)Ge by 16x(2$\bar{2}$0)Ge in the germanium film, respectively. The lattice matching epitaxy involving a 4% mismatch between Ge and Si provides a mechanism for epitaxial growth of Ge on Si(001).

Order No.: JA602-019

A parametric study of titanium silicide formation by rapid thermal processing

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(University of Rochester)

A parametric study of titanium silicide formation by rapid thermal processing was conducted to determine the effects of annealing temperature (650°C, 750°C), annealing time (30 s, 60 s), wet etching (no HF dip, with HF dip), sputter etching (no sputter etch, with sputter etch) and annealing ambient (Ar, N_2) on the completeness of conversion of 60 nm Ti on (111)-Si to C54-$TiSi_2$ based on sheet resistance and the uniformity of the sheet resistance measurements across the entire wafer. Statistical analysis of the results showed that temperature, annealing ambient and sputter etching had the greatest influence. Increasing the temperature and using argon gas instead of nitrogen promoted conversion of the film to C54-$TiSi_2$. On the other hand, sputter etching retarded it. The results also indicated significant interactions among these factors. The best uniformity in sheet resistance was obtained by annealing at 750°C without sputter etching. The different sheet resistance profiles showed gradients which were consistent with expected profile behaviors arising from temperature variations across the wafer due to the effect of a flowing cold gas and the effects of the wafer edge and flats.

Order No.: JA602-020

Effect of zirconia doping on the electrical behavior of yttria

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The ac electrical behavior of yttria doped with zirconia concentration ranging from 0.15 to 20 mol% is investigated in the temperature range of 800 to 1300°C. The ac electrical data, obtained in the range from 5 Hz through 13 MHz, indicated two distinct relaxations when analyzed in the impedance plane. These relaxations are attributed to lumped grains, trapping within grain-boundaries including possible electrode/sample effects. The admittance plane analysis revealed a semicircular relaxation in the low-frequency region indicating identical response to that of the low-frequency relaxation of the impedance plane. The incorporation of zirconia into yttria is found to lower the activation energy of conduction in the grains and enhance ionic contribution to the overall electrical conduction. The P_{O2} studies and transference number measurements near atmospheric region indicate that *p*-type conduction dominates for the lightly doped yttria. An ionic contribution to the conduction processes becomes significant in heavily doped samples at/near atmospheric P_{O2}.

Order No.: JA601-021

Influence of copper on the structural characteristics of carbon nanofibers produced from the cobalt catalyzed decomposition of ethylene

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(The Pennsylvania State University)

We have used a combination of techniques to examine modifications in the structural characteristics of carbon nanofibers produced from the interaction of cobalt and copper-cobalt powders with ethylene at temperatures over the range 425 to 700°C. The nanofibers generated from the interaction of cobalt with ethylene at 600°C were found to be highly crystalline in nature. Incorporation of as little as 2% copper into the cobalt created a major modification in the conformation of the solid carbon deposit, which was composed of multiple nanofiber limbs emanating from a single catalyst particle and in this state the carbon structures tended to be disordered. As the composition of the bimetallic was progressively changed to the point where copper became the major component, there was a significant increase in the degree of crystalline perfection of the nanofibers even though they maintained their multidirectional form. The transformation in structural characteristics of the carbon nanofibers is rationalized according to a concept wherein the crystalline order of the deposit is related to the wetting properties of the bimetallic particles with graphite.

Order No.: JA602-022

Mechanical properties of nanocrystalline copper produced by solution phase synthesis

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(Washington University)

Nanocrystalline copper powder was produced by $NaBH_4$ reduction of CuCl in a simple solution phase room temperature reaction. Uniaxial hot pressing in a closed tungsten die was used to compact powder into dense specimens. Samples were analyzed by x-ray diffraction, precision densitometry, electron microscopy, energy dispersive x-ray analysis, and selected area diffraction. Mechanical properties of the consolidated samples were determined by microhardness measurements, three-point bending of rectangular specimens, and compression tests. Yield strength measured for nanocrystalline Cu in the present work was over two times that reported in the literature for Cu with comparable grain size and over five times that of conventional Cu. Restricted grain growth observed in the hot pressed samples and improved mechanical properties are attributed to the presence of boron. A unique method of obtaining homogeneous *in-situ* nanosized reinforcements to strengthen the grain boundaries in nanocrystalline materials is identified.

Order No.: JA602-023

Deformation, recovery and recrystallization behavior of nanocrystalline copper produced from solution phase synthesized nanoparticles

R. Suryanarayanan, C.A. Frey, S.M.L. Sastry, B.E. Waller, W.E. Buhro
(Washington University)

Nanocrystalline copper produced by a solution-phase chemistry approach and compacted by hot pressing was subjected to room temperature deformation. Uniaxial compression and rolling were used to deform the samples to >90% reduction in thickness. Samples were subjected to several heat treatments to study microstructure and property evolution as a function of heat treatment. Thermal response of the as-pressed and deformed nanocrystalline Cu was also studied by differential scanning calorimetry. Optical metallography, scanning and transmission electron microscopy, and selected area diffraction were used to characterize microstructures after heat treatments. Samples exhibited an endotherm upon heating at 322°C which was reversible upon cooling. This was attributed to either dissolution and formation of Cu-B precipitates or the diffusion of B from the grain boundaries to the bulk and back to the grain boundaries. Exaggerated recrystallization occurs in the temperature range of 399-422°C. Samples maintained high dislocation density, deformation bands, and fine grain size up to 322°C. Beyond the recrystallization, temperature grains grew at a faster rate to sub-micron or micron levels. The strain hardening observed in the samples of the present study is attributed to the presence of boron. Two mechanisms are suggested for the role of B: (a) segregation of B to the grain boundaries leading to strengthening of grain boundaries, and (b) formation of Cu-B nanoprecipitates leading to precipitation strengthening.

Order No.: JA602-024

Eutectoid temperature of carbon steel during laser surface hardening

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(National Cheng Kung University)

A new method was developed to determine the eutectoid temperature, A_{c1}, of carbon steel during laser surface hardening. In the method, a three-dimensional heat flow model with temperature dependent physical properties was set up and solved for the temperature distribution employing a finite element method (FEM). Workpieces were heat-treated to produce a melted and hardened zone by a single-pass of a continuous-wave TEM_{∞} CO_2 laser beam. The depth profile of the melted zone was used as a calibrator to solve the uncertainty imposed by the unknown surface absorptivity. Obtained was an A_{c1} of, on average, 770°C, a superheat of 47°C compared to the equilibrium A_{c1} of 723°C. Furthermore, the numerical model was also employed to predict the hardened depth, and the results show that, for a depth more than 100 μm, the eutectoid temperature 770°C leads to a depth about 10% smaller than that predicted at 723°C. The use of the temperature dependent physical properties is critical; an error up to 80% could result if constant physical properties are used.

Order No.: JA602-025

Artificial dielectrics of conductive fibers in polymers: Effects of viscosity and matrix composition on permittivity and loss

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We report the microwave dielectric properties of composites made of nickel coated graphite fibers in two different types of silicone polymers. The influence of matrix viscosity on fiber alignment, fiber filamentation in the composites, and how those affect the permittivity and loss tangent of the composite are discussed. The dependence of the permittivity and loss of the composites on the permittivity and loss of the matrix will also be compared.

Order No.: JA602-026

Synthesis of α-Fe_2O_3 particle/oligomer hybrid material

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(Nagoya University)

Nanocrystalline α-Fe_2O_3 particles/oligomer hybrid could be synthesized by polymerization of iron(III) 3-allylacetylacetonate (IAA) followed by *in-situ* hydrolysis. The polymerization of IAA was dependent upon the polymerization temperature and solvent. GPC measurement showed that the polymerization degree of the IAA oligomer ranged from ~3 to ~6. Magnetic particle/oligomer hybrid was synthesized by hydrolysis of the IAA oligomer under neutral or alkaline condition. Crystalline particles from 10 to 40 nm were dispersed finely in the oligomeric matrix depending upon the hydrolysis conditions. The nanocrystalline particles below 10 nm in diameter were identified to be α-Fe_2O_3 by electron diffraction. The nano-sized α-Fe_2O_3/oligomer hybrid was found to show superparamagnetic behavior.

Order No.: JA602-027

Residual strain energy in composites containing particles

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(Toshiba Corporation)

Selsing's formula for radial tension at the particle-matrix interface is extended into a general formula which includes the effects of the amount of dispersed particles. It is a derived relationship between individual volumes of strained unit cells in the crystal lattices of the particles and of the surrounding matrix. These relationships are used to predict the effect of the particles (2H-TiB_2, 2H-ZrB_2 and t-WB) on their unit cells and on the unit cell of the surrounding 6H-SiC matrix. The precision of these predictions was 7.1% or better. Hence, in principle, it is possible to investigate the distributions of residual bulk stress/strain. Estimates of characterizing values of the three composite systems are attempted on the rough basis of the elastic constants of the SiC matrix, confirming the physical validity of this approach as a first approximation. Further, the residual bulk strain energies of the particles and the matrix are discussed in connection with the elastic term involved in the fracture energy of such composites.

Order No.: JA602-028

Stimulated surface crystallization of β-barium borate on glass due to ultrasonic treatment and second harmonic generation

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(Okayama University)

A glass of $47.5BaO{\cdot}47.5B_2O_3{\cdot}5Al_2O_3$ (in mol%) having a shorter optical absorption edge (~236nm) and suitable glass formation was selected as the mother glass for studying the surface crystallization of β-BaB_2O_4. β-BaB_2O_4 was not the main surface crystallized phase when only a conventional heat treatment was applied. Crystallization of β-BaB_2O_4 was stimulated due to ultrasonic surface treatment with an ethanol suspension of β-BaB_2O_4 particles and subsequent heat treatment. After the ultrasonic treatment β-BaB_2O_4 was the main crystallized phase. Transparent and dense β-BaB_2O_4 thin films/glass prepared showed second harmonic generation. The tensor components of the second order nonlinear optical susceptibility were estimated.

Order No.: JA602-029

The dynamic compressive behavior of beryllium bearing bulk metallic glasses

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In 1993, a new beryllium bearing bulk metallic glass with the nominal composition of $Zr_{41.25}Ti_{13.75}Cu_{12.5}Ni_{10}Be_{22.5}$ was discovered at Caltech. This metallic glass can be cast as cylindrical rods as large as 16 mm in diameter, which permitted specimens to be fabricated with

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geometries suitable for dynamic testing. For the first time, the dynamic compressive yield behavior of a metallic glass was characterized at strain rates of 10^2 to 10^4/sec by using the split Hopkinson pressure bar. A high-speed infrared thermal detector was also used to determine if adiabatic heating occurred during dynamic deformation of the metallic glass. From these tests it appears that the yield stress of the metallic glass is insensitive to strain rate and no adiabatic heating occurs before yielding.
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On the intergranular coupling in soft nanocrystalline materials
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The magnetic softness of nanocrystalline materials prepared from amorphous precursors is attributed to the average of the local magneto-crystalline anisotropy of the individual crystallites. We have studied the effective magnetic anisotropy of Fe-based nanocrystalline samples with different microstructures. These microstructures were produced by using different heating rates when crystallizing the precursor material by means of continuous heating treatments. From the results of our study of the magnetic properties of the samples, carried out from the measurement of the bias field dependence of the transverse susceptibility, it was possible to discern the occurrence of intergranular coupling and to evaluate the typical dimensions of the coupled units. Since these dimensions were larger than the characteristic length of the microstructure, we suggest that the enhancement of the soft properties is linked to the decrease of the microstructure-magnetization interactions originating in large units of coupled magnetic moments.
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How to tailor the porous structure of alumina and aluminosilicate gels and glasses
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Optically clear monolithic (OCM) gels of mesoporous aluminosilicates (average pore diameter d: 3.6 nm) and alumina (d: 6 nm) have been prepared by slow hydrolysis-polycondensation of alkoxides and converted into OCM mesoporous glasses by heating. In order to change the properties, different ways of modifying the pore size and structure are proposed. We show that addition of boron oxide reduces the average pore diameter. A higher effect can be obtained by addition of a surfactant. In this case the mesoporous matrix becomes microporous (d<2 nm). Another way of modifying the pore structure consists in introducing nanoprecipitates inside the porosity by an impregnation process. Modifications of the porous structure are different in alumina and aluminosilicates.
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A method of monitoring water absorption in polymers using a depth sensing indentation system
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The mechanical properties of many polymers are known to change as they absorb water. This fact has been used to monitor the absorption of water into the surface layers of an epoxy adhesive with a depth sensing indentation system. Two methods have been demonstrated. The sample can be immersed in water for a period of time then removed and tested in air; or, the sample can be tested *in-situ*. In the second method, the transport of water through the adhesive can clearly be seen in hardness/depth profiles. Hardness, elastic modulus and creep strain of the adhesive change with time until a stable value is reached which corresponds to full plasticization of the adhesive to the influence depth of the indenter. The initial mechanical properties of the epoxy are mostly recovered on drying.
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Elastic Green's function for a damaged interface in anisotropic materials
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We present the derivation of the elastic Green's function for an anisotropic bimaterial in a state of plane strain. A Fourier transform method is used to calculate the Green's function. A discontinuity in displacement is permitted across the interface between the two solids. This provides a useful functional form for parameterizing damage along an interface. We show several examples for the form of the displacement discontinuity and calculate the displacement Green's function for each. The Green's function derived here is applicable to a variety of interface problems between two different anisotropic solids or for two similar solids at different orientations.
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