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ABSTRACTS

COMMUNICATIONS

Occurrence of plastic instabilities in dynamic microhardness testing
G. Bérces, N.Q. Chinh, A. Juhász, J. Lendvai
(Eötvös University)

Plastic instabilities were observed to appear during dynamic ultra microhardness testing of a solid solution Al-3.3 wt.%Mg alloy. The tests were carried out at room temperature with a Vickers hardness indenter in a computer controlled dynamic ultra microhardness testing machine. During the tests the applied load was increased from 0 to 2000 mN at constant loading rate. The instabilities appear as characteristic steps in the continuously recorded load-indentation depth curves. The physical basis for the occurrence of the instabilities is the interaction between moving dislocations and solute atoms, a phenomenon termed in the literature as serrated yielding, jerky flow or Portevin-Le Châtelier effect. The instabilities start at a critical load, F_c in the depth-load curve. Varying the loading rate, μ, by two orders of magnitude F_c was found to increase linearly with the loading rate.
Order No.: JA806-001

Preparation of wurtzitic AlN thin films with a novel crystallographic alignment on MgO substrates by molecular-beam epitaxy
J.R. Heffelfinger, D.L. Medlin, K.F. McCarty
(Sandia National Laboratories)

Thin films of wurtzitic AlN have been deposited by molecular-beam epitaxy onto (001) oriented MgO substrates. The films are epitactic and align with the $(2\bar{1}\bar{1}0)_{AlN} \parallel (220)_{MgO}$ and the $[01\bar{1}1]_{AlN} \parallel [001]_{MgO}$, as evidenced by transmission electron microscopy. This configuration, which matches a close-packed direction of the film and substrate, allows for growth of two symmetrically equivalent orientation variants of the AlN film. These variants are distinguished by a 90° rotation about the $[01\bar{1}1]_{AlN}$ direction that is normal to the substrate surface. Each variant also aligns the $(0\bar{1}12)_{AlN} \parallel (2\bar{2}0)_{MgO}$ and the $(1\bar{1}01)_{AlN}$ to within 5° of being parallel to the $(200)_{MgO}$. The microstructure of the AlN films and origins of these novel alignments are discussed.
Order No.: JA806-002

Micron thick epitaxial (100) Ag film growth on MgO
A.C. Carter, W. Chang, S.B. Qadri, J.S. Horwitz, R. Leuchtner, D.B. Chrisey
(Naval Research Laboratory)

Epitaxial films of (100) Ag were deposited onto (100) MgO substrates to a thickness of 4 μm with no evidence of (111) nucleation. Deposited films were smooth and had large areas, 50 x 50 microns square, free of morphological defects. Films were deposited using a two step process. First, pulsed laser deposition was used to grow a 1000 Å Ag (100) seed layer on the MgO substrate. Second, e-beam evaporation was used to grow the film to the desired thickness. The high quality of the resulting films will allow them to be used as templates for further epitaxial deposition of other applied materials.
Order No.: JA806-003

Epitaxial growth of $SrTiO_3$ (00h), (0hh) and (hhh) thin films on buffered Si(001)
F. Sánchez, R. Aguiar, V. Trtik, C. Guerrero, C. Ferrater, M. Varela
(Universitat de Barcelona)

Epitaxial $SrTiO_3$(STO) thin films have been grown successfully on Si(001) buffered with single and double buffer layers by pulsed laser deposition. Depending on the buffer structure and under appropriate substrate temperature and oxygen pressure values, epitaxial films are grown with single orientations. Epitaxial STO films with (0hh), (00h) and (hhh) out-of-plane orientation have been obtained for the first time on yttria stabilized zirconia(YSZ)/Si(001), CeO_2/YSZ/Si(001), and TiN/YSZ/Si(001), respectively. Secondary ion mass spectrometry analyses show sharp interfaces and

good uniformity of the elements in each layer. The films are practically free of droplets and the rms value of roughness is smaller than 0.5 nm.
Order No.: JA806-004

Secondary ionic forces in lead molybdate melt solidification
H.C. Zeng, L.C. Lim
(National University of Singapore)

We report a dendritic crystallization of ionic melt of lead molybdate ($PbMoO_4$) under a concentric thermal field. The solidified melt is a $PbMoO_4$ single crystal with [001] axis normal to surface. The dendrite arms propagate and branch along <310> and <130>, forming a well-organized surface structure. It is evident that the interaction between a cation to its second-nearest anions determines the dendrite development and melt solidification.
Order No.: JA806-005

Oxygen diffusivity in MoO_3 as determined by a temperature programmed method
T.P. St. Clair, J.M. Restad, S.T. Oyama
(Virginia Polytechnic Institute and State University)

It is well established that solid state diffusivities can be indirectly measured by monitoring the concentration of a species at an external surface. A relationship can then be formulated that correlates the external concentration with an internal gradient. For example, if the rate of loss of a diffusing species can be measured, then a flux can be calculated, which in turn can be applied to an appropriate solution of Fick's equation to give the diffusivity (D). A technique for determining diffusivities has been previously applied to the oxygen-vanadium system, in which case the diffusivity of oxygen in V_2O_5 was determined using a temperature programmed method.
Order No.: JA806-006

REVIEW

Radiation effects in crystalline ceramics for the immobilization of high-level nuclear waste and plutonium
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This review provides a comprehensive evaluation of the state-of-knowledge of radiation effects in crystalline ceramics that may be used for the immobilization of high-level nuclear waste and plutonium. The current understanding of radiation damage processes, defect generation, microstructure development, theoretical methods, and experimental methods are reviewed. Fundamental scientific and technological issues that offer opportunities for research are identified. The most important issue is the need for an understanding of the radiation-induced structural changes at the atomic, microscopic, and macroscopic levels and the effect of these changes on the release rates of radionuclides during corrosion.
Order No.: JA806-007

ARTICLES

Electron microscopy study of interfacial structure and reaction of $YBa_2Cu_3O_7$/Y-ZrO_2 films on $LaAlO_3$ substrates
J.Y. Dai, F.H. Kaatz, P.R. Markworth, D.B. Buchholz, X. Liu, W.A. Chiou, R.P.H. Chang
(Northwestern University)

The detailed structure and interfacial reaction of epitaxial $YBa_2Cu_3O_7$/Y-ZrO_2 (YBCO/YSZ) films grown by chemical vapor deposition (CVD) on $LaAlO_3$ (LAO) substrates are investigated by means of high-resolution electron microscopy (HREM), analytical transmission electron microscopy, and scanning transmission electron microscopy (STEM). The epitaxial relations of YBCO/YSZ/LAO are: $[100]_{YBCO}//[110]_{YSZ}//[100]_{LAO}$, and $(001)_{YBCO}//(001)_{YSZ}//(001)_{LAO}$. The optimum atomic configuration at the YSZ/LAO interface, in which oxygen is the first atomic layer on LAO, is proposed by using HREM combined with image simulation based on the atomic structure models of the interface. Near the YBCO/YSZ interface, two localized interfacial reaction products are formed: (1) a Y-rich modulated ZrO_2 structure at the surface of the YSZ film, which may be caused by the diffusion of Y into the YSZ grains; (2) an intergranular $BaZrO_3$ phase formed by the diffusion of Ba along the columnar grain boundaries of the YSZ film during YBCO growth.
Order No.: JA806-008

Nanoscale liquid phase epitaxy between Si and Au nanoparticles
Y. Wakayama, H. Fujinuma, S-I. Tanaka
(Japan Science and Research Corporation)

A self-assembly technique was used for fabrication of a Si/metal interface in nanometer scale. Fine particles of gold of nanometer-order diameter were generated by a gas-phase condensation method and deposited on a Si substrate. Through a heat-treatment and a cooling process, a nanoscopic Si-Au composite structure was formed on the surface of the Si substrate. Then, surface diffusing Si atoms played an important role for fabrication of the Si-Au structure which were epitaxially grown projectingly onto the substrate. Furthermore, the Si/Au interface was atomically flat with no mixed-layer formation and the Au nanoparticles also had the same crystal orientation as that of the Si dots in spite of a large lattice constant mismatch between them. This structure was considered to be fabricated as a result of minimization of the total surface and interface energy of the Si/Au system.
Order No.: JA806-009

The development of microstructure of Ni_3Al during rapid cooling and heating
L. Wang, H. Liu, K. Chen, Z. Hu
(Chinese Academy of Sciences)

The processes of rapid solidification from liquid to solid and heating from glass to crystalline for Ni_3Al are simulated using molecular dynamics method. An amorphous state can be obtained by rapid solidification as long as the cooling rate is sufficiently large, which is very difficult to get in an experiment. An fcc-type crystalline is obtained by heating the amorphous with a small heating rate. Based on the pair analysis technique, the microstructures of liquid, supercooled liquid, amorphous, and crystalline states of Ni_3Al have been analyzed. Furthermore, the effects of cooling rate and heating rate on microstructures of Ni_3Al during rapid solidification and heating processes have been discussed.
Order No.: JA806-010

Investigation of ordering kinetics in Cu_3Au with the tomographic atom probe
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Kinetics of congruent ordering in Cu_3Au at 350°C was investigated by means of a three-dimensional atom-probe. This instrument, called a Tomographic Atom Probe (TAP), enables atomic resolution images of a small volume ($10 \times 10 \times 100\ nm^3$) of the material reconstructed in the three dimensions of space. The time evolution of ordered domains at 350°C shows that a $t^{1/2}$ law is followed as soon as 5 min. For this aging time, the nucleus diameter is close to 1.7 nm. This scaling law was observed even before domains came into contact (t = 50 min). Competitive growth was observed to start as soon as 5 min. The number density was observed to decrease rapidly up to t = 50 min. A slower decrease was observed when domains begin to impinge. Experimental conditions and requirements as well as advantages of TAP as compared to HREM for the study of ordering are discussed in detail.
Order No.: JA806-011

Formation of ultrafine powders of binary alloy systems by plasma jet
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Recently a new method, i.e., a plasma jet method, was developed in our lab for the production of ultrafine powders. In the present work, we investigated the formation of binary Al-Fe, Al-Si, Fe-Si, Al-Cu, Al-Ni, Ni-Ti, Fe-Cu and Fe-Ti ultrafine powders using this method. Pre-mixed pure elemental powders of various compositions of Al-Fe, Al-Si, Fe-Si, Al-Cu, Al-Ni, Ni-Ti, Fe-Cu and Fe-Ti were used as starting materials. These premixed powders were injected into the plasma jet of Ar-N_2 working gas to form ultrafine powders. The obtained ultrafine powders were characterized by x-ray diffraction and transmission electron microscope to check the microstructures of ultrafine particles.

Order No.: JA806-012

The use of reliability factors in analyzing powder patterns in Pt-Si sputtering targets and subsequent films
A. Rahman, W.P. Lowe, C.W. Bates, Jr.
(Howard University)

X-ray powder diffraction was used to characterize a Pt-Si sputtering target and subsequent films. The powder patterns of each sample indicated lines due to diffraction from different phases. We have initiated a preliminary study through which we have analyzed and characterized these films. The results presented for these samples corroborate with results observed for this system in the planar configuration.

Order No.: JA806-013

Structural characterization of combustion synthesized $MoSi_2$-Si_3N_4 composite powders and plasma sprayed $MoSi_2$-Si_3N_4 composites
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A systematic structural characterization has been conducted on combustion synthesized $MoSi_2$-Si_3N_4 powders and plasma sprayed composites by transmission electron microscopy (TEM). The powders are cleaner and have less surface oxide contamination than most commercial powders. The distribution of the two phases can be tailored by modifying the synthesis conditions, which has resulted in improved retention of Si_3N_4 after plasma spraying. TEM studies performed on deformed composites indicate a strong link between even dispersion of second phases and enhanced dislocation plasticity in the plasma sprayed composites.

Order No.: JA806-014

Hi-Nicalon fiber-reinforced celsian matrix composites: Influence of interface modification
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(NASA-Lewis Research Center)

Unidirectional celsian matrix composites having 42–45 volume percent of uncoated or BN-SiC coated Hi-Nicalon fibers were tested in three-point bend at room temperature. The uncoated fiber-reinforced composites showed catastrophic failure with strength of 210 ± 35 MPa and a flat fracture surface. In contrast, composites reinforced with coated fibers exhibited graceful failure with extensive fiber pullout. Values of first matrix cracking stress and strain were 435 ± 35 MPa and 0.27 ± 0.01%, respectively, with ultimate strength as high as 960 MPa. The elastic Young's modulus of the uncoated and coated fiber-reinforced composites were 184 ± 4 GPa and 165 ± 5 GPa, respectively. Fiber push-through tests and microscopic examination indicated no chemical reaction at the uncoated or coated fiber-matrix interface. The low strength of composite with uncoated fibers is due to degradation of the fiber strength from mechanical damage during processing. Because both the coated- and uncoated-fiber reinforced composites exhibited weak interfaces, the beneficial effect of the BN-SiC dual layer is primarily the protection of fibers from mechanical damage during processing.

Order No.: JA806-015

Elastic stress in composite FeTi hydrogen storage materials
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A simple model of the elastic stress in a composite hydrogen absorbing material is developed to account for the hydrogen storage properties of nanocrystalline FeTi with a network of intergranular phase having a wide storage site energy distribution. The model accounts for the equilibrium properties of nanocrystalline FeTi hydrogen absorbers made by ball-milling such as the narrowing of the miscibility gap and changes in plateau pressure. A second model is proposed for disconnected inclusions of the second phase. The effect of chemical disorder is also briefly examined.

Order No.: JA806-016

Fabrication of on-chip barium strontium titanate capacitors by metallo-organic decomposition
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Metallo-organic thin film decomposition (MOD) was used in forming barium strontium titanate (BST) thin film capacitors on phosphorus doped polysilicon films deposited on 4" silicon wafers. A single step deposition process yielded highly uniform, crack free BST films ranging up to 0.25 μm in thickness and having various step heights and dimensional area. Scanning electron microscopy (SEM) showed very good step coverage and planarization of the BST. The capacitors had capacitance densities above 200 nF/cm^2, leakage current densities less than 1.55 μA/cm^2 at a bias voltage of 10 volts, and a dielectric breakdown field above 1 MV/cm. Small temperature coefficients of capacitance and dissipation (tan δ) were also observed. Frequency response measurements were made using the BST capacitors and on-chip resistors in low pass and high pass circuit configurations. A plot of relative gain and phase angle versus frequency showed excellent agreement with predicted results.

Order No.: JA806-017

Sol-gel synthesis of phosphate ceramic composites II
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Phosphate ceramics were synthesized using the sol-gel technique of direct reaction of P_2O_5 with tetraethoxy silane (TEOS) or titanium tetraethoxide (Ti(OEt)$_4$) . The reaction mechanism of P_2O_5 and TEOS was deduced using liquid and solid-state NMR. Hexacoordinated silicon in phosphosilicate gels was observed. A specially structured titanium phosphate-layered $Ti(HPO_4)_2{\cdot}2H_2O$ was synthesized for the first time through the sol-gel method. The gelation process and sintering properties were investigated.

Order No.: JA806-018

Improvement on the degradation of microwave sintered ZnO varistors by post-annealing
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Microwave sintering process not only densified the ZnO materials in a higher rate, but also resulted in significantly better varistor characteristics. Large nonlinear coefficient and low leakage current density were attained by cooling the samples under a rate of 80°C/min after sintering, followed by 600°C post-annealing for 60 min under oxygen atmosphere. Inappropriate annealing deteriorated the varistor characteristics that can either be attributed to the insufficient reoxidation along grain boundaries when annealed in N_2 (or air) or loss of Zn-species in these regions when annealed at 750°C (900°C). By contrast, the degradation behavior of these materials can be improved by annealing process regardless of the annealing atmosphere or temperature.

Order No.: JA806-019

W-Ti-O layers for gas sensing applications. Structure, morphology, and electrical properties

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The kinetics of phase transition and phase segregation induced by annealing temperature on Ti-W-O gas sensing layer was studied by x-ray diffraction, Raman spectroscopy and scanning electron microscopy. The main goal was to identify, on the basis of kinetics studies, structurally stable Ti-WO_3 thin film phases and compare their response to polluting gases in order to determine possible correlations between structural and electrical properties of the sensing layers.

Order No.: JA806-020

The role of microstructure and processing on the proton conducting properties of gadolinium-doped barium cerate

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The influence of grain boundary conductivity and microstructure on the electrical properties of $BaCe_{0.85}Gd_{0.15}O_{3-\delta}$ have been examined. Grain sizes were varied by sintering at various temperatures. Impedance data were analyzed using the brick layer model, and some new consequences of this model are presented. The specific grain boundary conductivity exhibits an activation energy of ~0.7eV, and for similar processing routes, is independent of grain size. An isotope effect was observed, indicating that protons (or deuterons) are the mobile species. TEM investigations showed the intergranular regions to be free of any glassy phase that could account for the differences in bulk and grain boundary properties. Single crystal fibers, grown by a modified float zone process, were notably barium deficient, and exhibited a low conductivity, comparable to that of polycrystalline $Ba_{0.96}Ce_{0.85}Gd_{0.15}O_{3-\delta}$.

Order No.: JA806-021

Epitaxial films of $Li_{1-x}Nb_{1-x}W_xO_3$ prepared by chemical solution deposition

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The growth of epitaxial thin films of $Li_{1-x}Nb_{1-x}W_xO_3$ from solution precursors on single crystal $LiNbO_3$ substrates is reported. An all-alkoxide solution readily gave single phase powders after simply mixing the constituent components, whereas an acetate-alkoxide system required additional solution processing stages to give phase pure powders. Heat treatment of films on single crystal, basal plane $LiNbO_3$ substrates at 600°C formed a nanocrystalline, porous film which was converted to an epitaxial film after heating to 800°C. Measurements of second harmonic generation in powders indicate an increase in SHG efficiency with increasing tungsten content. Optical absorption data for films were calculated using reflectance and transmittance spectroscopy data, and indicate a decrease in band gap with increasing tungsten.

Order No.: JA806-022

Mechanically induced reaction between alkaline earth metal oxides and TiO_2

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(Australian National University)

This paper outlines the formation of alkaline earth metal titanates, of the general formula $MTiO_3$, directly from the metal oxides and rutile by mechanical activation in a laboratory ball mill at room temperature. X-ray diffraction analysis showed that the reaction was essentially complete within 100 hours for all metals except magnesium. The titanates formed all had a Scherrer crystallite size of 11-12 nm and a lattice strain of 0.5–0.6%, neither of which were affected by extended milling. Annealing studies confirmed that the titanate was formed during milling and showed that grain growth could be achieved at temperatures below that generally used for their formation. Mixed cation titanates could also be formed by milling, but tended to be barium rich until annealed.

Order No.: JA806-023

Grain morphology and cation composition heterogeneity of $Pb(Zr_xTi_{1-x})O_3$ thin films deposited by metal-organic chemical vapor deposition

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The preferred orientation, grain morphology and composition heterogeneity of the polycrystalline $Pb(Zr_xTi_{1-x})O_3$ (PZT) thin films were characterized by x-ray diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM), and x-ray energy dispersive spectroscopy (EDS). PZT thin films with nominal x = 0.5 were grown by metal-organic chemical vapor deposition (MOCVD) on (110)- and (101)-textured RuO_2 bottom electrodes at temperatures ≤ 525°C. Columnar grain microstructure with strongly faceted surface morphology was observed in both films. The grain morphology and surface roughness of the PZT films were observed to depend on those of the underlying RuO_2 layers. TEM-EDS analysis shows notable cation composition heterogeneity in length scales of 0.2–2 μm. Pronounced Pb composition deficiency and heterogeneity were also observed in PZT/(110)RuO_2 in length scales above 40 μm. The grain morphology and cation heterogeneity of the PZT films are discussed on the basis of diffusion-limited columnar growth mechanism.

Order No.: JA806-024

Reverse burning phenomenon in self-propagating high-temperature synthesis

J-H. Lee, A-Y. Lee, C-C. Chen
(National Chung Cheng University)

An interesting reverse burning phenomenon was observed during the combustion synthesis of zirconium-based materials. When an external heat was applied to one end of a green pellet, the ignition was initiated at the other end. Also, the ignition position, measured from the heated end, was proportional to the apparent green density of the compact. The possible explanations for this reverse burning phenomenon are discussed.

Order No.: JA806-025

Heterogeneous junction of YPSZ by superplastic flow

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Layers of different composition and/or grain size of yttria partially stabilized zirconia have been compressed, with the stress perpendicular to the interface of the layers and temperature of 1400°C, in order to produce a joint using the microstructural feature of superplasticity found in fine grained ceramics. The pieces joined have been characterized by SEM, showing a clean interface with no cavitation. The stiffness of the junction was checked using Vickers indentation at room temperature at the interface and compression tests at the same conditions (T = 1400°C in air) used for the joining and the stress parallel to the interface. The observation and comparison between the cracks developed around the indents at the interface and in the bulk of the pieces joined as well as the absence of cavities along the interface in the samples compressed parallel to the interface shows that this technique is useful to produce a joint with clean and strong interface.

Order No.: JA806-026

Variations in the synthesis of barium hexaferrite doped with iridium and its effect in the catalytic combustion of hydrocarbons

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This paper discusses the influence of the synthesis technique on the porous structure, phase transformation and catalytic properties of barium hexaferrite. Barium hexaferrite was synthesized by two different citrate gel methods. The first one involved metal salts, citric acid and ammonia, while the second dispersed the same components in a polyacrylamide gel. X-ray diffraction analysis was used to determine the nature of the phase while the Brunauer, Emmett and Teller (BET) analysis was used to study the porous structure of the materials which were heat treated at 700°C, 900°C and

1200°C. The catalytic activity of all materials in the combustion of methane was also measured.

Order No.: JA806-027

Niobium-titanium oxide powders obtained by laser-induced synthesis: Microstructure and structure evolution from diffraction data

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The influence of the niobium content on the anatase-to-rutile phase transition in nanopowders of Nb-Ti oxides was studied and the changes in the particle size and microstrain distribution obtained at different temperatures were analyzed. A correlation is found between the initial microstructure in the $Ti_{1-x}Nb_xO_2$ (x = 0.03, 0.2) powder and the niobium content. The presence of Nb was found to inhibit the growth of both the anatase and the rutile phases.

Order No.: JA806-028

An experimental device for depth-sensing indentation tests in mm-scale

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A device for spherical indentation using a tip radius of 2 mm and loads up to 10 kN is presented. This facility can be applied e.g. to verify methods for characterizing the behavior of materials exhibiting homogeneous and isotropic constitutive properties. The indentation device can be driven both load- and depth-controlled. The accuracy of measurements is about 1 N for load and 0.2 μm for depth at a total depth of 200 μm.

Two materials, an austenitic steel and an aluminum alloy, have been tested and their Young's moduli have been determined. For determining the Young's modulus from spherical indentation data, use is made of a so-called L_t-method, which had been developed in Reference 1. Results obtained in this way are compared with corresponding values measured by one-dimensional homogeneous tensile experiments.

Order No.: JA806-029

Influence of surface hardening by laser irradiation on the loss factor of cast iron

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(Nagoya Municipal Industrial Research Institute)

Surface hardening by laser irradiation was examined regarding the loss factor, which is the parameter of the vibration-damping characteristics, for flake graphite cast irons and spherical graphite cast irons. The surface of the samples was quenched by changing speed of beam, gas pressure, and beam frequency. It was found that the depth of the hardening region was from 0.12 mm to 1.11 mm, and the hardness of the boundary between the hardening and the non-hardening regions changed suddenly. It was confirmed that the loss factor of the laser hardened specimens increased considerably as compared with that of the as cast specimens. The internal friction at the vicinity of the boundary between the hardening and the non-hardening regions may be responsible for this result.

Order No.: JA806-030

Preparation of silver particles by spray pyrolysis of silver-diammine complex solutions

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A novel precursor solution system, containing NH_3 as a complexing agent, was used for the production of Ag powders by spray pyrolysis. Solutions of Ag_2CO_3, Ag_2O, and $AgNO_3$ with NH_4HCO_3 were used in this study. Ag powders were obtained at unexpectedly low temperatures, i.e. 400°C or less. The Ag powders with a shell-like morphology were obtained from Ag_2CO_3 and Ag_2O solutions, whereas dense Ag particles of about 1 μm diameter were obtained from $AgNO_3$-NH_4HCO_3 solution. These morphologies are explained in terms of NH_3 release during spray pyrolysis.

Order No.: JA806-031

Simultaneous real-time study of initial hillocking and changes in overall stress in evaporated Al films during heating

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Optical quality Al films were evaporated by an electron beam onto Si wafers in an ultra high vacuum system. The as-deposited samples were radiative heated at a rate of 3°C/s in air environment. During heating, measurements of *initial* hillocking and changes in overall film stress were performed simultaneously and in real time as a function of time and temperature with a specially designed optical instrument. The physical principle of this instrument is based on laser beam deflection caused by film stress induced wafer bending and partial integrated light scattering from surface roughening. The experimental results show how the *initial* hillocking is accompanied by changes in the overall stress and yield a very good correlation between the onset of hillock formation and the maximum change in overall stress.

Order No.: JA806-032

SIMS study of erbium diffusion in lithium niobate crystals

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A systematic investigation of erbium diffusion in lithium niobate ($LiNbO_3$) crystal as a function of crystal cut direction, diffusion process parameters (temperature and time) and initial film thickness is reported. Depth concentration profiles of erbium are obtained by secondary-ion-mass spectrometry. Combining experimental data with diffusion theory, the relevant diffusion parameters are derived. Diffusion from an infinite source of erbium ions is studied to evaluate the solid solubility lower limit of Er in $LiNbO_3$. Thin film diffusion regime, with complete depletion of ion source, is also investigated. A comparison of Er diffusion with Er/Ti co-diffusion in $LiNbO_3$ crystals is reported.

Order No.: JA806-033

Electron energy-loss study of titania particles

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(Virginia Tech)

Small titania particles, prepared by hydrolysis and condensation using *in-situ* steric stabilization, have been studied by high-energy, transmission, electron energy-loss spectroscopy. Electron diffraction patterns and energy-loss spectra as a function of momentum transfer were measured for as-prepared particles (amorphous titania), particles annealed at 600°C (primarily anatase), and particles annealed at 1000°C (primarily rutile). The energy-loss spectra at low momentum disagreed with the loss function calculated from optical data (rutile) and disagreed with theory (rutile and anatase). The data was fit by an Elliot-like model for a resonant exciton interacting with a continuum of levels. The translational effective mass of the exciton derived from the fitting was quite large indicating that it was self-trapped.

Order No.: JA806-034

Atomic force microscopy studies of ZnS films grown on (100) GaAs by the successive ionic layer adsorption and reaction method

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In this study zinc sulfide thin films were grown by the successive ionic layer adsorption and reaction (SILAR) technique on (100) GaAs substrates from aqueous precursor solutions. The atomic force microscopy (AFM) method was used to study the growth of the films up to a thickness of 180 nm. The ZnS thin films on (100) GaAs were smooth with an rms roughness of 0.2–1.9 nm depending on the film thickness. After the GaAs surface was covered with ZnS the growth appeared to be nearly layerwise. In addition, in-situ AFM studies were carried out to analyze the dissolution of (100) GaAs in water, which is a process competing with the thin film deposition by the SILAR.

Order No.: JA806-035

Shrinkage anisotropy of glass powder compacts sintered in dilatometers
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Dilatometer push-rods exert uniaxial compressive stresses on powder compacts undergoing sintering. In this study the effect of such stresses on the shrinkage anisotropy behavior of glass powder compacts is considered. A shrinkage anisotropy factor (k), defined by the ratio between axial and radial strain, is used to quantify the anisotropy in cylindrical compacts ($k = 1$ represents isotropic shrinkage). The available experimental data in the literature indicate that the effect of the applied dilatometer stress is to increase the value of k over that of free sintering conditions. For the small stresses exerted by the dilatometer push-rods, it is shown that k is always >1. The common practice of calculating the sintered density from uniaxial dilatometer data, ignoring the existence of shrinkage anisotropy, is shown to lead to significant errors, even for values of k only slightly different from $k = 1$.

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Morphology control and texture of hematite particles by dimethylformamide in forced hydrolysis reaction
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The effects of dimethylformamide (DMF) on morphology and texture of hematite particles produced from a forced hydrolysis reaction of $FeCl_3$-HCl solution were investigated by TEM, XRD, FTIR, TG-DTA, N_2 and H_2O adsorption, and zeta potential measurements. The morphology of synthetic hematite particles was concentration dependent; they changed from large sphere with diameter of ca. 600 nm to diamond-like shape with increasing DMF concentration in the aging solution accompanying a reduction of their size to 80 nm without incorporation of DMF in the particles. This fact was explained by an acceleration of phase transformation from β-FeOOH to hematite with an elevation of the solution pH owing to dimethylamine produced from a hydrolysis of DMF at an elevated temperature. TEM and XRD suggested that the diamond-like hematite particles formed above 6~10 vol% DMF possess a single crystal nature. Gas adsorption technique revealed that the particles produced above 10 vol% DMF possess a high thermal stability. TG and FTIR indicated that the hematite particles produced with DMF contained small amounts of OH^- ions in the lattice though they provided a single crystal nature.

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A study on the migration of Co(II) and Fe(III) ions in the process of thermal phase separation of an alkali-borosilicate glass containing cobalt and ubiquitous trace iron
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Absorption spectra of added Co(II) ions and EPR spectra of both Co(II) and ubiquitous Fe(III) ions in an alkali-borosilicate glass of vycor type composition are studied both before and after thermal phase-separation of the glass as well as in a silica glass derived from the phase-separated glass through vycorization.

The results show that although almost all Co(II) ions migrate to the separated alkali borate phase at the time of phase separation, only the interstitial ions in the case of Fe(III) take part in similar migration. The Fe(III) ions in the substitutional silicon sites, however, remain unaffected. It is also revealed that as a consequence of the process of phase separation a small fraction of the interstitial Fe(III) ions gets converted into substitutional ions leading to a net increase in the concentration of substitutional Fe(III) ions in the glass.

Analysis of both the absorption and EPR spectra of the Co(II) ions suggests that change of host of the ion due to its migration does not bring about any significant change in its coordination structure which is typically a distorted tetrahedron both in the parent glass as well as in the phase separated borate glass.

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Diffusion-limited and asymmetric growth of amorphous layer in Ni/Zr bilayer upon annealing
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Asymmetric growth of amorphous layer in a Ni/Zr bilayer, in which a thin disordered interlayer is present, upon annealing at medium temperatures is observed by molecular-dynamics simulation with an n-body potential. It is shown that the amorphous layer is extended from the interlayer with different speeds towards two opposite directions and that the growth kinetics follows a time dependence of $t^{1/2}$, indicating amorphization upon annealing in the Ni/Zr bilayer is indeed through a diffusion-limited reaction. Besides, two low temperature limits allowing the growth of amorphous layer towards Ni and Zr layers are also obtained.

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Measured and calculated thermoelastic properties of supersaturated fcc Ni(Al) and Ni(Zr) solid solutions
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Metastable face-centered cubic (fcc) solid solutions of $Ni_{1-x}Al_x$ and $Ni_{1-x}Zr_x$ have been prepared in thin-film form using dc planar magnetron sputtering in a UHV system. In both these alloy systems, extended solubilities in the fcc phase and a pronounced (111) texture are observed after sputter-deposition. An amorphous phase is found to form in $Ni_{1-x}Al_x$ for $x \geq 0.30$ and in $Ni_{1-x}Zr_x$ for $x \geq 0.05$. Lattice constants, thermal expansion coefficients, and Debye temperatures were derived from x-ray diffraction measurements. These parameters were also calculated by using *ab initio* methods in the framework of the local-spin density and coherent potential approximations for the electronic subsystem and the Debye-Grüneisen model for the vibrational properties of the nuclei subsystem. Experiment and theory are compared and discussed.

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Generation of microwave plasma under high pressure and fabrication of ultrafine carbon particles
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A microwave plasma generator, which functions under high pressure, has been developed and used in the fabrication of fine carbon particles. The plasma generator is a two-stage-type resonator, which consists of rectangular and semicylindrical-type resonators which are coupled in series for torching plasma and keeping it stable under high pressure. The plasma can be torched in helium gas at 3×10^6 Pa by tuning the dimensions of apparatus elements. Fine carbon particles of ~50 nm are obtained using a mixture of helium and methane gas. The particles are found to be crystalline from the results of transparent electron microscopy and diffraction analysis.

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