JMR Abstracts

Editor-in-Chief: Robert A. Laudise • Associate Editors: Shigeyuki Sōmiya, Heinrich J. Wollenberger

Abstracts for August 1997 Journal of Materials Research *Volume 12, Number 8*

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REVIEW

COMMUNICATIONS

Local cross-sectional profiling of multilayer thin films with an atomic force microscope for layer thickness determination
J.R. LaGraff, J.M. Murduck
(TRW Space and Defense–Superconducting Electronics Organization)

A new essentially non-destructive cross-sectional method is described for measuring the individual thicknesses of multilayer $YBa_2Cu_3O_7$ (YBCO) and $SrTiO_3$ (STO) thin films using off-axis ion milling and the atomic force microscope (AFM). Since the ion-milling is done during routine patterning of a thin-film device and the AFM requires only a small area for imaging, no additional sample preparation is required. This is a significant improvement over traditional cross-sectional techniques which often require lengthy and destructive sample preparation. Also, there is not a priori reason that this technique would not be amenable to other multilayer thin-film systems.

Order No.: JA708-001

Observations of dislocations in Cu/Nb nanolayer composites after deformation
Y-C. Lu, H. Kung, A.J. Griffin Jr., M.A. Nastasi, T.E. Mitchell
(Los Alamos National Laboratory)

Dislocations have been observed in deformed Cu/Nb nanolayer composites of wavelength 17 and 7 nm. The dislocations thread through the Cu/Nb interfaces even though there is a change of Burgers vector. Conventional and high resolution transmission electron microscopy studies show that the in-plane bowing direction of these dislocations in the Cu layers is opposite to that in the Nb layers, so that the dislocations appear to zig-zag. These observations are explained by the presence of residual tensile stresses in Cu and residual compressive stresses in Nb, which make dislocations bow in opposite directions in the alternating layers.

Order No.: JA708-002

Scanning tunneling microscopy and atomic force microscopy investigation of organic tetracyanoquinodimethane thin films
H-J. Gao, H.X. Zhang, Z.Q. Xue, S.J. Pang
(Peking University and Chinese Academy of Sciences)

Scanning tunneling microscopy (STM) and atomic force microscopy (AFM) investigation of tetracyanoquinodimethane (TCNQ) and the related C_{60}-TCNQ thin films is presented. Periodic molecular chains of the TCNQ on highly oriented pyrolytic graphite (HOPG) substrates were imaged, which demonstrated that the crystalline (001) plane was parallel to the substrate. For the C_{60}-TCNQ thin films, we found that there were grains on the film surface. STM images within the grain revealed that the well ordered rows and terraces, and the parallel rows in different grains were generally not in the same orientation. Moreover, the grain boundary was also observed. In addition, AFM was employed to modify the organic TCNQ film surface for the application of this type of materials to information recording and storage at the nanometer scale. The nanometer holes were successfully created on the TCNQ thin film by the AFM.

Order No.: JA708-003

ARTICLES

Unidirectional partial melting and solidification of SmBCO superconductor
M. Sumida*, Y. Nakamura+, Y. Shiohara+, T. Umeda*
*(*The University of Tokyo, +Superconductivity Research Laboratory-ISTEC)*

Microstructure control of SmBCO superconductor was carried out using the floating zone partial melting and solidification method. It is generally recognized that finely and uniformly dispersed non-superconductive high-temperature stable phase (Sm211) particles included in the superconductive Sm123 matrix act as effective pinning centers. Microstructure formation of the partial molten mixture (Sm211 particles and BaO-CuO liquid) by decomposition of the precursor Sm123 on melting and solidification of Sm123 from the mixture have to be controlled concurrently to fabricate the 123/211 composite fiber with the optimum microstructure. During unidirectional solidification, planar crystal growth which provides the single crystal growth of Sm123 becomes unstable with increased growth rate. During unidirectional melting, the mean diameter of aligned Sm211 particles behind the melting interface decreases with increased growth rate and with decreased temperature gradient at the melting interface. Initial composition of the precursor significantly affects the formation behavior of Sm211 particles. The contribution of process parameters to the microstructure formation is also briefly discussed.

Order No.: JA708-004

Refinement of Nd-422 phase trapped in a $Nd_{1+\delta}Ba_{2-\delta}Cu_3O_{7-y}$ superconducting matrix
F. Frangi, S-I. Yoo, N. Sakai, M. Murakami
(Superconductivity Research Laboratory-ISTEC)

The possibility of controlling Nd-422 phase dimension and morphology, both in the partially melted state and in the final superconducting

https://doi.org/10.1557/S0883769400033686 Published online by Cambridge University Press

matrix was investigated. Nd-Ba-Cu-O samples were prepared by the MPMG (Melt Powder Melt Growth) technique. Precursor materials containing Nd as either Nd_2O_3 or Nd-422 particles, formed during precursor quenching, were used. The role of Pt in Nd-422 refining was analyzed. The effect on Nd-422 nucleation and growth, of stoichiometries with increasing Nd-422 molar excesses (0%, 10%, 20% and 40%) combined with different heating processes, up to the partially melted state, were experimented with. Uniformly distributed needle-like particles (1 μm diameter and 10–15 μm length) were obtained at 1120°C and resulted in a further refined distribution trapped in completely processed samples.
Order No.: JA708-005

Modeling of peritectic $YBa_2Cu_3O_{7-x}$ growth using transparent organic analogues
R. Terborg, G.J. Schmitz
(ACCESS e.V.)

Transparent organic analogues were directionally solidified to investigate, via *in-situ* observation, the peritectic reaction occurring in the YBCO-superconductor system. Nucleation and growth of peritectic and properitectic phases were examined with respect to similarities with the solidification of the YBCO-superconductor. The selected organic system, salicylic acid-acetamide, turned out to match the requirements concerning crystal shape, small nucleation rate of the peritectic phase on the properitectic phase, existence of stoichiometric phases with no solubility limits common and undercoating ability of the peritectic phase. Several features of YBCO growth which were previously deduced only from metallographic cross-sections could be verified by direct observation. The organic analogue system will also be used in the future to improve numerical simulations.
Order No.: JA708-006

Crystalline phases and electronic structures in superconducting Bi-Sr-Ca-Cu oxides
M.D. Giardina*, R. Feduzi*, D. Inzaghi*, A. Manara*, C. Giori+, I. Natali Sora#, V. Dallacasa§
*(*C.E.C. Joint Research Centre, +Università di Parma, #Università di Brescia, §Università di Verona)*

Two classes of samples, denominated A and B, of layered Bi-Sr-Ca-Cu oxides having the same nominal composition 4:3:3:4, but different thermal histories, were investigated by using field modulated microwave absorption (ESR), powder x-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS) and x-ray absorption near edge structure (XANES). Previous electrical resistivity measurements showed that the B-samples only presented two superconducting phases with midpoints of the transition temperatures at ~80 K and ~105 K. The microwave absorption technique indicated instead the presence of islands which became superconducting at the above-mentioned temperatures also in the A samples. The crystalline and electronic structures of the two types of samples are illustrated and discussed. A plausible theoretical interpretation of the experimental results, based on a quantum percolation model with Coulomb interaction, is also given.
Order No.: JA708-007

Low energy ion impact-enhanced growth of cubic boron nitride in a supersonic nitrogen/argon plasma flow
D.H. Berns, M.A. Cappelli
(Stanford University)

This paper describes the growth and analysis of cubic boron nitride films in a low-density, supersonic nitrogen/argon plasma flow into which boron trichloride gas was injected. Both hexagonal boron nitride (h-BN) and cubic boron nitride (c-BN) were synthesized using this apparatus. Phase selectivity is obtained by applying a relatively low negative bias voltage on the substrate. All of the films described in this paper were grown on {100} silicon wafers at substrate temperatures varying from 400–700°C. Boron nitride films with greater than 90% cubic phase were successfully synthesized with this method. The films were analyzed using infrared spectroscopy, x-ray photoelectron spectroscopy, and scanning electron microscopy. The volumetric percentages of the hexagonal and cubic phases were determined from model fits to the infrared transmission spectra of the films. X-ray photoelectron spectroscopy provided qualitative evidence for the presence and/or lack of sp^2 bonding through the identification of a π-plasmon feature in the spectra. Infrared reflectance spectra are used to provide insight into the growth mechanisms leading to c-BN formation and have revealed features which are not present in the transmission spectra, specifically the 1305 cm^{-1} LO mode of c-BN and the 1610 cm^{-1} LO mode of h-BN. The mean ion energies involved with this bias-enhanced chemical vapor deposition (CVD) process are much lower than the ion energies in traditional physical vapor deposition processes; however, the ion fluxes (currents) used in this CVD process are at least an order of magnitude higher, resulting in a total momentum transfer to the deposited atoms through ion bombardment that is at least equal to or greater than that reported for many ion-enhanced PVD processes.
Order No.: JA708-008

Quantitative analysis of electromigration damage in Al-based conductor lines
O. Kraft, J.E. Sanchez Jr., M. Bauer, E. Arzt
(Max-Planck-Institut für Metallforschung and Institut für Metallkunde der Universität)

Electromigration damage in Al-based interconnects with three compositions (pure Al, Al-1%Si-0.5%Cu and Al-2%Cu) was studied quantitatively. Using scanning electron microscopy, the spacings between more than 1000 voids and hillocks were measured. The distribution of the spacings was found to be a function of the composition, the applied current density and the line width. The measurements confirm the existence of a threshold product of current density and diffusion length. In particular, a dependence of this threshold product on the Cu content was found. The results of the analysis show that there are clear correlations between the details of the microscopic damage processes and the lifetime of the conductor lines.
Order No.: JA708-009

Void nucleation on a contaminated patch
B.M. Clemens, W.D. Nix, R.J. Gleixner
(Stanford University)

The energetics of a simple model of void nucleation on a contaminated patch between the sidewall and metal in an integrated circuit interconnect are examined to determine void nucleation behavior. The conditions under which there is no void nucleation barrier are represented by a simple relationship between the volume driving force, the equilibrium contact angle, the surface energy of the metal, and the contaminated patch radius. The void nucleation barrier, when it exists, is a strong function of these same parameters, and increases sharply as the driving force decreases, and under some conditions, increases with increasing equilibrium contact angle.
Order No.: JA708-010

Deformation characteristics of quasicrystalline Al-Cu-Fe alloys
J.E. Shield*, M.J. Kramer+
*(*University of Utah, +Ames Laboratory-USDOE)*

The deformation characteristics of icosahedral Al-Cu-Fe quasicrystals were determined by high temperature creep experiments between 680 and 720°C and 15 and 41 MPa. The deformation process was determined to be controlled by grain boundary mechanisms. Both the stress and grain size sensitivity exponents were found to be 2, suggesting that grain boundary sliding was the rate-controlling deformation mechanism. Microstructural analysis supported this conclusion, as no intragranular defects were produced during the deformation experiments.
Order No.: JA708-011

Characterization of low-cycle fatigue damage in Inconel 718 by laser light scanning
K.J.C. Chou, J.C. Earthman
(University of California)

A technique for *in-situ* laser light scanning (LLS) was developed to monitor surface damage on nickel-base superalloy specimens under low-cycle fatigue conditions. This technique characterizes the surface state with a parameter called the defect frequency which minimizes memory requirements and data processing time since it does not involve image

processing. As a result, the present technique is capable of scanning speeds that are substantially greater than those achieved with image processing methods. Cylindrical Inconel 718 specimens were tested using an automated servo-hydraulic machine at ambient temperature under fully reversed strain control conditions for constant strain amplitudes ranging from 0.3% to 1%. The fatigue damage was monitored by scanning a laser beam along the gauge section of the specimens during periodic interruptions of the cyclic loading. Acetate replicas of the gauge section surface were also made on some of the specimens to characterize the damage using SEM and image analysis techniques. Comparisons of the results demonstrate the capabilities of the present light scanning technique for characterizing fatigue damage on the surface of the Inconel 718 specimens. In particular, a rapid rise in the mean defect frequency is shown to correspond to an initial increase in microcrack density that saturates at approximately 20% of the fatigue life. This transient behavior is followed by a plateau in defect frequency which corresponds to crack propagation and interlinkage until failure occurs. The number of cycles to microcrack density saturation as indicated by the defect frequency is found to be linearly related to the number of cycles to failure. Accordingly, the present system provides a characterization of microcrack damage that may be used to predict the low cycle fatigue life of Inconel 718 specimens long before failure occurs.

Order No.: JA708-012

Hardness and modulus properties in ion-beam-modified amorphous carbon: Temperature and dose rate dependences

D-H. Lee, H. Lee, B. Park
(Georgia Institute of Technology)

Ion implantation into amorphous carbon has been initiated to investigate the possibility of superhard carbon-nitride formation. Studies of implantation-temperature effects by 100 keV N^+ or 80 keV C^+ ions at 50 μA show a narrow temperature window at approximately -100°C for the optimum surface hardness and elastic modulus (measured by nanoindentation), both values much higher than those for the unimplanted amorphous carbon. No distinguishable properties are found between nitrogen and self (carbon) implantations. At a dose rate of 5 μA, however, the optimum hardness and modulus are found at a lower implantation temperature, with a broader temperature window. The enhanced strengths are well correlated with the asymmetric diffuse peak at around 1,500 cm^{-1} in Raman spectroscopy, and the increased ratio of sp^3- over sp^2- bonded carbon sites observed by electron energy loss spectroscopy.

Order No.: JA708-013

Contact area evolution during an indentation process

K. Li*, T.W. Wu+, J.C.M. Li*
*(*University of Rochester, +IBM Almaden Research Center)*

The evolution of the contact area during an indentation process has been examined by an a.c. technique and also by finite element analysis on five mechanically different materials. Constant contact area regimes were observed during the initial unloading stage and the duration of that regime depends strongly on the material properties. The consistency of the results obtained by the two approaches not only proves the validity and advantage of the a.c. indentation technique but also confirms the applicability of the contact stiffness equation. The influence of a hardness impression on unloading characteristics has also been clearly demonstrated by numerically simulating a reloading process.

Order No.: JA708-014

Formation of single-crystal $CoSi_2$ buffer layers on Si (100) substrates by high dose Co ion implantation for the deposition of $YBa_2Cu_3O_{7-x}$ thin films

Y. Li, P. Seidel, F. Machalett, S. Linzen, F. Schmidl
(Friedrich-Schiller-Universitat Jena)

High quality single crystal $CoSi_2$ layers have been successfully formed on Si (100) using low energy high dose Co ion implantation followed by subsequent annealing method as a buffer layer for the deposition of $YBa_2Cu_3O_{7-x}$ (YBCO) thin films. Rutherford backscattering spectrometry with channeling (RBS-C) measurements showed that $CoSi_2$ layers after annealing at temperatures between 850 and 950°C had a minimum yield χ_{min} of about 3%. X-ray diffraction (XRD) spectra revealed that $CoSi_2$ layers had the same orientation as the Si (100) substrates. Phi scan XRD spectra proved that $CoSi_2$ layers epitaxially grew in the cube on cube epitaxial growth mode with respect to the Si (100) substrates. YBCO films and CeO_2/YSZ buffer layers were deposited on $CoSi_2$/Si (100) substrates via laser ablation and electron beam evaporation, respectively. θ-2θ, ω, and φ scan XRD spectra illustrated that YBCO films and CeO_2/YSZ buffer layers had the epitaxial structure both in a-b plane and along the c-axis. YBCO films grown on this multilayered structure demonstrated excellent superconducting properties with the zero resistance transition temperature T_{c0} of 87–90 K. The transition width (ΔT_c) was about 1 K. Orientation and epitaxial crystalline quality of YBCO films and CeO_2/YSZ buffer layers were confirmed by XRD and RBS-C characterization. All films consisted of c-axis oriented grains. RBS-C spectra indicated a high degree of crystalline perfection with a channeling minimum yield for Ba as low as 8%, and interdiffusion between the YBCO film and buffer layers or between the YBCO film and the substrate was limited. This multilayer system shows the possibility for the application of $YBa_2Cu_3O_{7-x}$ thin films on technical Si substrates in the field of hybrid superconductor-semiconductor technology.

Order No.: JA708-015

Void nucleation in passivated interconnect lines: Effects of site geometries, interfaces, and interface flaws

R.J. Gleixner, B.M. Clemens, W.D. Nix
(Stanford University)

Stress driven nucleation of voids in passivated aluminum interconnect lines is analyzed within the context of classical nucleation theory. A discussion of sources of tensile stress in such lines leads to an upper limit of 2 GPa. Calculations suggest that even at this high stress, nucleation rates are far too low to account for observed rates of voiding. Void formation at a circular defect at the line/passivation interface is then considered. In this case, a flaw on the order of nanometers in size may develop into a void under the imposed stress. These results strongly suggest that void nucleation in aluminum interconnect lines can be controlled by eliminating defects in the line/passivation interface.

Order No.: JA708-016

***In-situ* x-ray investigation of hydrogen charging in thin film bimetallic electrodes**

N.M. Jisrawi, H. Wiesmann, M.W. Ruckman, T.R. Thurston, G. Reisfeld, B.M. Ocko, M. Strongin
(Brookhaven National Laboratory)

Hydrogen uptake and discharge by thin metallic films under potentiostatic control was studied using x-ray diffraction at the National Synchrotron Light Source (NSLS). The formation of metal-hydrogen phases in Pd, Pd-capped Nb and Pd/Nb multilayer electrode structures was deduced from x-ray diffraction data and correlated with the cyclic voltammetry (CV) peaks The x-ray data was also used to construct a plot of the hydrogen concentration as a function of cell potential for a multilayered thin film.

Order No.: JA708-017

Resistance of plasma-deposited a-C:H/fluorocarbon films to anodic breakdown in aqueous electrolytes

C. Srividya*, M. Sunkara+, S.V. Babu*
*(*Clarkson University, +University of Louisville)*

The corrosion characteristics of a-C:H/fluorocarbon composite films deposited on type 301 stainless steel substrates were investigated using potentiometry and electrochemical impedance spectroscopy. The films were deposited by radio frequency (13.56 MHz) plasma deposition from different mixtures of hexafluoroethane, acetylene, and argon. A 10 nm thick polysilicon film was plasma-deposited prior to a-C:H film deposition to improve adhesion. The anodic current densities recorded with all the coated samples, in an electrolyte consisting of 0.1 M NaCl and 0.1 M Na_2SO_4 at 1.5 V (Standard Calomel Electrode), were at least three orders of magnitude less than that for a bare steel sample. They were also at least 20 times less than that obtained with diamond-like carbon film-coated substrates. EIS spectra obtained for these samples, while exposed

to 0.6 M NaCl solution over an extended period of time (30–40 days), confirm their barrier properties. All the coatings showed near capacitive behavior in the frequency range 10 mHz–4 KHz.
Order No.: JA708-018

Secondary-phase formation and microstructural development in the interaction between $SrBi_2Ta_2O_9$ films and $Pt/Ti/SiO_2/Si$ substrates
C-H. Lu, B-K. Fang
(National Taiwan University)

The phase formation and microstructural development of $SrBi_2Ta_2O_9$ thin films prepared via spin-coating using metalorganic solution on $Pt/Ti/SiO_2/Si$ substrates have been investigated in this study. The spun-on films started to crystallize from above 550°C, and were well crystallized at 800°C. At higher than 850°C a secondary phase having a pyrochlore structure was formed in the films. Through the study of SEM and AFM, the appearance of this pyrochlore phase was found to cause microstructural irregularity and increased roughness of the films. The analysis of EDS and SIMS confirmed that the interaction between the films and the titanium species which diffused outwards from the titanium layer on substrates was the origin for the occurrence of the pyrochlore phase. In addition, varying the thickness of the coated films and platinum layers demonstrated remarkable influence on the formation amount of the pyrochlore phase.
Order No.: JA708-019

Mullite precursor I. Characterization of mullite precursor formed by a reaction of monosilicic acid on aluminium hydroxide
T. Yokoyama*, K. Nishu*, S. Torii*, Y. Ikeda*, T. Watanabe+
*(*Kyushu University, +Tokyo University of Fisheries)*

By adsorption of monosilicic acid on aluminium hydroxide, an amorphous aluminosilicate (AAS) of Si/Al ratio of 1/3 was formed. When the AAS was heated to 1000°C, mullite ($3Al_2O_3{\cdot}2SiO_2$) crystallized directly without the formation of spinel phase. It was clearly evidenced that correct adjustment of three factors, 1) the Si/Al ratio, 2) the proportion of 4- and 6-coordinated aluminiums, and the single environment of SiO_4^{4-} unit (no Si-O-Si bond), are important for the direct mullitization from AAS.
Order No.: JA708-020

$Pb(B'_{1/2}B''_{1/2})O_3$-type perovskites: Part I. Pair-correlation theory of order-disorder phase transition
H.M. Jang, S-C. Kim
(Pohang University of Science and Technology)

The limitation of the long-range order parameter and the necessity of the short-range order parameter for the thermodynamic description of $Pb(B'_{1/2}B''_{1/2})O3$-type perovskites were discussed. Based on the discussion, a statistical thermodynamic model which takes into account of the configuration of the neighboring B-site ions (B′ and B″ cations) was developed. A pair-correlation approximation was used in the calculation of the configurational entropy and the long-range coulombic interaction energy between the nearest B-site ions. The theoretical calculations using $Pb(Sc_{1/2}Ta_{1/2})O_3$ (PST) and $Pb(Sc_{1/2}Nb_{1/2})O_3$ (PSN) systems indicate that the short-range order parameter persists over a wide range of temperature examined (0 ~ 1800 K) and that there possibly occur consecutive long-range order-disorder transitions in the configuration of B-site cations. The possibility of the existence of short-range ordering above the long-range order-disorder transition temperature was also examined using the annealed PSN specimen as a typical example of $Pb(B'_{1/2}B''_{1/2})O_3$-type perovskites.
Order No.: JA708-021

$Pb(B'_{1/2}B''_{1/2})O_3$-type perovskites: Part II. Short-range order parameter as a criterion of the distinction between relaxor and normal ferroelectrics
S-C. Kim, H.M. Jang
(Pohang University of Science and Technology)

A classification scheme of $Pb(B'_{1/2}B''_{1/2})O_3$-type perovskites with respect to the B-site order parameters was proposed based on the theoretical calculation of the short-range order parameter (σ) using the pair-correlation model. The calculated order parameters predict that a $Pb(B'_{1/2}B''_{1/2})O_3$-type perovskite without any charge difference between B′ and B″ cations [e.g., $Pb(Zr_{1/2}Ti_{1/2})O_3$ (PZT)] is represented by a completely disordered state with the absence of a finite coherence length. On the other hand, a $Pb(B'_{1/2}B''_{1/2})O_3$ perovskite system having different ionic charges is characterized either by the short-range ordering with a nanoscale coherence length or by the macroscopic long-range ordering, depending on the magnitude of ionic charge difference between B′ and B″ ions. The normal ferroelectricity in $Pb(B'_{1/2}B''_{1/2})O_3$-type complex perovskites was then correlated either with a completely disordered state ($\sigma = 0$) or with a perfectly ordered state ($\sigma = 1$), whereas the relaxor behavior was attributed to the nanoscale short-range ordering ($0 < \sigma < 1$) in the configuration of the B site cations.
Order No.: JA708-022

Characterization of the induced plastic zone in a single crystal TiN(001) film by nanoindentation and transmission electron microscopy
M. Odén, H. Ljungcrantz, L. Hultman
(Linköping University)

The slip system of TiN at room temperature has been determined to be {110}<110> by Burgers vector analysis using transmission electron microscopy and slip trace analysis of indents made on a TiN(001) film deposited on a MgO(001) substrate. Both small indents (0.4 mN maximum load) and large indents (40 mN maximum load) were used to study the dislocation structure in TiN. The nucleation of dislocations was investigated using small indents. Further development of the plastic zone was studied using large indents and microhardness indents (1.6 N). The critical resolved shear stress evaluated at the load when pop-in occurs was estimated to be 3.7 GPa, assuming a Herzian elastic contact. Indents made with a 0.4 mN maximum load show a complex dislocation pattern with loops and straight segments that belong to the same slip system. Dislocations of mixed screw and edge type are dominant. The cascade of dislocations generated during pop-in is likely to nucleate from loops. For larger indents, the plastic zone extends more than three times the diameter of the imprint. The straight dislocations outside the large imprint are arranged in arrays along the <100> and <110> directions. A scanning force microscopy study of the surface outside a microhardness indent revealed a raised surface along <110> and formation of troughs along <100>.
Order No.: JA708-023

Structural characterization of Fe_3O_4-NiO superlattices using high-resolution transmission electron microscopy
A. Rečnik*+, D.L. Carroll*, K.A. Shaw#, D.M. Lind#, M. Rühle*
*(*Max-Planck-Institut für Metallforschung, +Jozef Stefan Institute, #Florida State University)*

Superlattices of Fe_3O_4-NiO layers have been studied by high-resolution transmission electron microscopy (HRTEM). These superlattices are grown by oxygen-plasma-assisted molecular-beam epitaxy (MBE) on (001) oriented MgO substrates, and exhibit a high degree of ordering at the interfaces between the interlayers. The lack of misfit dislocations at the Fe_3O_4-NiO interfaces suggests that lattice strain is largely accommodated by changes in the lattice spacing. By quantitative HRTEM analysis of Fe3O4-NiO interfaces, possible atomic models are discussed, having implications in magnetic ordering and spin exchange mechanisms for such interlayer systems.
Order No.: JA708-024

Variations of structure and dielectric properties on substituting A-site cations for Sr^{2+} in $(Na_{1/2}Bi_{1/2})TiO_3$
S-E. Park, K.S. Hong
(Seoul National University)

Changes in structure and phase transition behavior of $(Na_{1/2}Bi_{1/2})_{1-x}Sr_xTiO_3$ were investigated using XRD analysis and dielectric measurements. A decrease in the degree of lattice distortion was observed when the structure was rhombohedral ($x<0.26$). Further substitution of Sr^{2+} ($x \geq 0.26$) caused $(Na_{1/2}Bi_{1/2})_{1-x}Sr_xTiO_3$ to show no macroscopic lattice distortion. However, all investigated samples ($0.1 \leq x \leq 0.5$) were ferroelectric at room temperature regardless of the lack of lattice distortion found in XRD patterns. Further XRD investigations revealed that non-uniform

strains caused by non-uniform polarized islands were responsible for this behavior. A-site cation substitution of Sr^{2+} also resulted in the decrease of T_{max} (the temperature where dielectric constant is maximum). Typical relaxor ferroelectric phase transition behavior, associated with the shift of T_{max}, with respect to the measuring frequencies, was found above x = 0.18. This could be also ascribed to the decrease of T_{max} - this reveals the increase in the shift of T_{max} with frequency, with associated decrease in ionic displacement and space charge contributions and associated increase in the relative contribution to the polarization by microdomains and domain walls at temperatures near T_{max}.

Order No.: JA708-025

Effects of (100)-textured $LaNiO_3$ electrode on the deposition and characteristics of $PbTiO_3$ thin films prepared by rf magnetron sputtering

C-M. Wu, T-J. Hong, T-B. Wu
(National Tsing Hua University)

Highly (100)-oriented thin films of $PbTiO_3$ were prepared on (100)-textured LNO/Pt/Ti/SiO_2/Si substrates by rf magnetron sputtering at temperature ≥ 480°C, while randomly oriented $PbTiO_3$ films were obtained on Pt/Ti/SiO_2/Si substrates. The textured LNO layer can help to control the orientation of $PbTiO_3$ thin films, and reduce their surface roughness quite significantly. The dielectric constant (ε_r) of $PbTiO_3$ films deposited on LNO was lower than that of films on Pt and the dielectric loss (tan δ) increased when a higher deposition temperature or longer time was used. The highly (100) textured $PbTiO_3$ films also showed a different ferroelectric hysteresis characteristics, i.e., a higher coercive field and a lower remanent polarization, from that of randomly-oriented films deposited on Pt.

Order No.: JA708-026

Structure development studies of $SrBi_2(Ta_{1-x}Nb_x)_2O_9$ thin films

T-C. Chen, T. Li, X. Zhang, S.B. Desu
(Virginia Polytechnic Institute and State University)

In this research, two tasks were pursued: (1) determination of the onset temperature of ferroelectric hysteresis properties of $SrBi_2Ta_2O_9$ thin films by structure development study, and (2) low temperature processing for thin film fabrication. For task (1), a non-destructive optical method using spectroscopic ellipsometry was utilized for characterizing the structure development of $SrBi_2Ta_2O_9$ thin films. The optical constants and film thickness were measured as a function of annealing temperature by spectroscopic ellipsometry. By observing the changes in refractive indices and film thickness, the temperatures of phase transformation and grain growth were determined. Consistent results were obtained from x-ray diffraction measurements. By comparing the results of the structure development study and ferroelectric hysteresis properties investigation, the onset temperature of the hysteresis curve of $SrBi_2Ta_2O_9$ with 50% excess Bi was determined to be about 700°C. The critical factor for the compound to exhibit a well-defined hysteresis curve was found to be the grain size. For task (2), the effects of excess Bi content and Nb/Ta ratio of $SrBi_2(Ta_{1-x}Nb_x)_2O_9$ on the ferroelectric hysteresis properties were studied. It was found that the onset temperature for obtaining well-defined hysteresis properties can be reduced by adding excess Bi or increasing Nb/Ta ratio. By choosing $SrBi_2Ta_2O_9$ with 50% excess Bi, the onset temperature of the hysteresis curve was reduced to about 650°C.

Order No.: JA708-027

Synthesis and magnetic properties of nanostructured maghemite

D. Vollath*, D.V. Szabó*, R.D. Taylor+, J.O. Willis+
*(*Forschungszentrum Karlsruhe, +Los Alamos National Laboratory)*

Nanocrystalline maghemite, γ-Fe_2O_3, can be synthesized in a microwave plasma using $FeCl_3$ or $Fe_3(CO)_{12}$ as the precursor. Electron microscopy revealed particle sizes in the range of 5 to 10 nm. In general, this material is superparamagnetic. The magnetic properties are strongly dependent on the precursor. In both cases the production process leads to a highly disordered material with the consequence of a low magnetization. The assumption of a disordered structured is also supported by EEL (electron energy loss) and Mössbauer spectroscopy. The structure of this material shows a nearly identical number of cations on tetrahedral and octahedral lattice sites.

Order No.: JA708-028

Electronic structures and host excitation of $LaPO_4$, La_2O_3 and $AlPO_4$

K.C. Mishra*, I. Osterloh+, H. Anton+, B. Hannebauer+, P.C. Schmidt+, K.H. Johnson#
*(*OSRAM Sylvania Inc., +Technische Hochschule Darmstadt, #Massachusetts Institute of Technology)*

We report the electronic structures and associated optical properties of three inorganic oxides, namely lanthanum oxide, aluminum phosphate and lanthanum phosphate calculated by the first principles augmented spherical wave (ASW) and full potential linear muffin tin orbital (FP-LMTO) band structure methods, and the self-consistent field Xα scattered wave (Xα SW) molecular orbital cluster approach. Our calculations indicate negligible effect of the choice of exchange correlation potentials on the position, shape and relative ordering of the energy bands. The ASW energy gaps in lanthanum phosphate and aluminum phosphate agree satisfactorily with the measured values. A comparison of the electronic density of states for an isolated phosphate group from molecular orbital calculation and that of the valence band from the band structure methods indicates that the nature of bonding within the phosphate groups do not change in aluminum and lanthanum phosphates. The states near the top of the valence band and bottom of the conduction band are mostly due to the phosphate bonding and antibonding orbitals indicating that optical absorption near the band edge involves excitation of electrons from the bonding levels to antibonding levels associated with phosphate groups. This explains why the optical gaps in many rare earth phosphates are nearly equal.

Order No.: JA708-029

The influence of binders on interfacial failure in sapphire fiber reinforced NiAl composites

J.I. Eldridge*, D.R. Wheeler*, R.R. Bowman*, A. Korenyi-Both+
*(*NASA Lewis Research Center, +Gilcrest)*

The influence of organic binders on fiber/matrix bonding during the powder metallurgy fabrication of sapphire fiber reinforced NiAl matrix composites (sapphire/NiAl) was investigated. One composite panel was fabricated using a poly(methyl methacrylate) (PMMA) fiber binder and a teflon matrix powder binder; another panel was fabricated by binderless powder metallurgy consolidation. The effect of the binders on fiber/matrix bonding was evaluated by fiber push-out testing from room temperature to 900°C. Examination of mating fiber and matrix-trough fracture surfaces by scanning electron microscopy (SEM) and Auger electron spectroscopy (AES) revealed differences in interfacial morphology and chemistry depending on the use of binders in fabrication. The primary difference between the two composites was the much higher concentration of carbon at the fiber/matrix interface in sapphire/NiAl fabricated with binders. This carbon residue from binder burnout prevented clean contact between the sapphire fiber and NiAl matrix surfaces, resulting in a weak, thermomechanically clamped fiber/matrix interface, in contrast to the stronger, less temperature dependent, interfacial bonding observed without binders.

Order No.: JA708-030

High refractive index materials of iron sulfides and poly(ethylene oxide)

T. Kyprianidou-Leodidou, H-J. Althaus, Y. Wyser, D. Vetter, M. Büchler, W. Caseri, U.W. Suter
*(*ETH-Zentrum, +Eidgenössische Materialprüfungs- und Forschungsanstalt)*

High refractive index composites of iron sulfides and poly(ethylene oxide) (PEO) have been prepared by co-precipitation from aqueous solution. Several reaction parameters were varied: inorganic reactants, reactant ratios, reaction temperatures, and reaction times. Selected samples were characterized with organic microelemental analysis, x-ray fluorescence spectroscopy, x-ray diffraction, DSC, and TEM. The nanocomposites with the highest refractive indices have been prepared using PEO, Mohr's salt and H_2S or NaHS. The analyses indicate that the iron sulfides in these materials consist of finely dispersed mackinawite and greigite ("amorphous" FeS) and, partially, also pyrite. The refractive index of the resulting composites are clearly above 2 at 632.8 and 1295 nm and can assume values between 2.5 and 2.8.

Order No.: JA708-031

Propagation of acoustic waves in periodic and random two-dimensional composite media

J.O. Vasseur*, P.A. Deymier+
*(*Université de Lille I, +University of Arizona)*

Transmission of acoustic waves in two-dimensional composite media composed of arrays of Duralumin cylindrical inclusions embedded in a PVC matrix is studied. Experimental and theoretical results for the transmission spectrum of a periodic array of cylinders organized on a square lattice are reported. Local gaps in the first two-dimensional Brillouin zone are predicted and observed. The experimental measurements of power spectra for a random array of inclusions show a weak correlation between disorder and acoustic absorptions up to a high degree of disorder. Only in the case of highly random arrangements does the transmission spectrum diverge from the one obtained with a periodic array of inclusions.

Order No.: JA708-032

REVIEW

Radiation effects in glasses used for immobilization of high-level waste and plutonium disposition

W.J. Weber*, R.C. Ewing+, C.A. Angell#, G.W. Arnold§, A.N. Cormack**, J.M. Delaye++, D.L. Griscom##, L.W. Hobbs§§, A. Navrotsky†, D.L. Price††, A.M. Stoneham¥, M.C. Weinberg[x]
*(*Pacific Northwest National Laboratory, +University of New Mexico, #Arizona State University, §Consultants International, **Alfred University, ++DTA/SRMP, ##Naval Research Laboratory, §§Massachusetts Institute of Technology, †Princeton University, ††Argonne National Laboratory, ¥University College of London, [x]University of Arizona)*

This paper is a comprehensive review of the state-of-knowledge in the field of radiation effects in glasses that are to be used for the immobilization of high-level nuclear waste and plutonium disposition. The current status and issues in the area of radiation damage processes, defect generation, microstructure development, theoretical methods and experimental methods are reviewed. Questions of fundamental and technological interest that offer opportunities for research are identified.

Order No.: JA708-033

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