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## ABSTRACTS

### COMMUNICATIONS

**Current-voltage hysteresis in  $GdBa_2Cu_3O_{7-\delta}$  ceramics in air at room temperature**

Y. Kurihara, T. Okamoto, B. Huybrechts, M. Takata  
(Nagaoka University of Technology)

We report here the appearance of a hysteresis in the current-voltage characteristic, when a voltage is applied to a  $GdBa_2Cu_3O_{7-\delta}$  rod at room temperature. This hysteresis was caused by the appearance and disappearance of a hot spot. When a gradually increasing dc voltage (0-8 V) was applied to a  $GdBa_2Cu_3O_{7-\delta}$  rod, a hot spot appeared at a given voltage,  $V_a$ . Further increasing the voltage increased the spot size, while the current stayed constant. When a voltage of 8 V was reached the voltage was gradually decreased. The spot did not disappear at  $V_a$  but at a voltage 0.2 to 1.3 V lower than  $V_a$ . The current remained constant until the spot disappeared. By the authors' best knowledge, this is the first report of a hysteresis in the current-voltage characteristic at room temperature for a high- $T_c$  superconductor.

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**Scanning electron microscopy observations of misfit dislocations in epitaxial  $In_{0.25}Ga_{0.75}As$  on GaAs(001)**

R.R. Keller, J.M. Phelps  
(National Institute of Standards and Technology)

Dislocations in the misfit epitaxial film system  $In_{0.25}Ga_{0.75}As$  on GaAs(001) were imaged using a modified electron channeling contrast technique in a LaB<sub>6</sub> SEM. We obtained images at an incident beam energy of 30 keV, a beam divergence of less than 1 mrad and a specimen tilt of 70° in conjunction with a movable scintillator detector mounted at a take-off angle of approximately 3° to 5°. We achieved a spatial resolution of approximately 80 to 100 nm with this technique. Such resolution allowed rapid imaging of clusters consisting of only a few closely-spaced dislocations in a 55 nm thick film. At such small film thicknesses, we did not require accurate knowledge of the incident beam direction in order to obtain sufficiently strong channeling contrast for qualitative characterization. The observed defect arrangements included features which we believe represent clustered threading segments.

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### ARTICLES

**Controlled decomposition and reformation of the 2223 phase in Ag-clad  $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$  tapes and its influence on the microstructure and critical current density**

J.A. Parrelli\*, Y. Feng\*, S.E. Dorris\*, D.C. Larbalestier\*  
(\*University of Wisconsin-Madison, \*Argonne National Laboratory)

The decomposition of almost fully reacted  $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$  (BSCCO 2223) tapes caused by heating in 1 atmosphere of pure O<sub>2</sub> at

825°C has been studied. It was found that partially decomposing 2223 tapes to a mixture of  $Bi_2Sr_2Ca_1Cu_2O_y$ ,  $(Ca,Sr)_2PbO_4$ , and other secondary phases reduced the critical current density (77 K, 0 T) from ~20 kA/cm<sup>2</sup> to nearly zero. Reheating the tapes in 7.5% O<sub>2</sub> restored the 2223 phase and, while there was some degradation of the 2223 grain alignment due to residual secondary phase growth, the critical current density was also restored to nearly its original value. We hypothesize that such a decomposition/reformation process can be useful in increasing the connectivity of polycrystalline 2223, by encouraging the formation of a liquid phase which heals residual cracks in the BSCCO core.

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**The demonstration of  $Y_2BaCuO_5$  particle segregation in melt-processed  $YBa_2Cu_3O_{7-x}$  through a computer visualization model**

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(University of Notre Dame)

In melt-processed  $YBa_2Cu_3O_x$  (123) microstructures, often unreacted  $Y_2BaCuO_5$  (211) particles are observed to be present in an inhomogeneous manner delineating distinguishable patterns. The presence of these patterns in 123 is more clearly observed in the case of low 211 volume concentration and also when the 211 particle size is small. The observed patterns are believed to be due to 211 particle segregation in 123 domains in specific planes during melt texture growth. In the present paper a software program is used to draw a three-dimensional visualization model to demonstrate a possible structure of 211 particle segregation in 123 domains and to explain the presence of observed patterns in the microstructures. The formation of such a 211 particle segregation is explained in light of previously proposed 123 growth mechanisms.

Order No.: JA603-004

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**Rapidly quenched Y-Pd-B-C borocarbides: Identification of the superconducting metastable phases**

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Rapidly quenched melt-spun ribbons of palladium-based borocarbides with large fractions of the superconducting phase ( $T_c > 20$  K) have been obtained. Superconductivity in the Pd-based alloys seems to be metastable and exists only in the rapid quenched state. Thus, while in the as cast state the  $YPd_2B_2C$  ingot is found to be paramagnetic, the rapidly quenched ribbon superconducts and has a simple XRD-pattern characteristic for a face-centered cubic lattice with a lattice parameter of 4.15 Å. A systematic study in an annealing sequence shows decay of the superconducting phase above 750°C. The reduction in the integral intensities of XRD fcc-peaks correlates well with the loss of the superconducting fraction on successive annealing. The recently reported tetragonal phase, attributed to the superconducting phase in this system, is not found in any of our rapid quenched samples. We present the

phase diagrams indicating the optimum composition regimes for superconductivity in the Y-Ni-B-C and Y-Pd-B-C systems. From our magnetic data the values for the upper critical field  $H_{c2}(0)$ , and the coherence length  $\xi(0)$  are estimated to be 10 Tesla and 57 Å, respectively, for the rapid quenched YPd<sub>2</sub>B<sub>2</sub>C superconducting ribbons.

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#### Growth defects in GaN films on sapphire: The probable origin of threading dislocations

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Single crystal GaN films with a wurtzite structure were grown on the basal plane of sapphire. A high density of threading dislocations parallel to the c-axis crossed the film from the interface to the film surface. They were found to have a predominantly edge character with a  $1/3\langle 11\bar{2}0 \rangle$  Burgers vector. In addition, dislocation half-loops, elongated along the c-axis of GaN, were also found on the prism planes. These dislocations had a mostly screw character with a  $[0001]$  Burgers vector. Substrate surface steps with a height of  $1/6C_{Al_2O_3}$ , were found to be accommodated by localized elastic bending of GaN  $(0001)_{GaN}$  planes in the vicinity of the film/substrate interface. Observations show that the region of the film, with a thickness of ~100 nm, adjacent to the interface is highly defective. This region is thought to correspond to the low-temperature GaN "buffer" layer which is initially grown on the sapphire substrate. Based on the experimental observations, a model for the formation of the majority threading dislocations in the film is proposed. The analysis of the results leads us to conclude that the film is under residual biaxial compression.

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#### Microstructural changes in welded Zn-Al alloy

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Extruded eutectoid Zn-Al alloy was welded by a melt of the same eutectoid alloy. Two different microstructures were observed in the joint part and in the bulk of the welded alloy. Typical dendritic structure of as cast Zn-Al alloy was observed in the joint part of the welded alloy. The bulk of the welded Zn-Al alloy appeared as fine grain structure. Two different metastable phases  $\eta'_T$  decomposed from  $\eta'_S$  of chilled as cast state and  $\eta'_E$  of extruded state were found to be unstable during early stage of aging. A four phase transformation occurred after the decompositions of these two metastable phases of  $\eta'_T$ . Microstructures of both joint part and bulk of the welded alloy were investigated during the aging processes.

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#### A study of solid-state amorphization in Zr-30 at.% Al by mechanical attrition

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Elemental powders of zirconium and aluminum in the atomic ratio of 70:30 were mechanically alloyed in an attritor under argon atmosphere using zirconia balls as milling media. Samples have been taken out for characterization after different durations of milling. The process of alloying and resultant amorphization had been studied using x-ray diffraction (XRD) and transmission electron microscopy (TEM). Scanning electron microscopy (SEM) was carried out to study the morphological changes occurring during repeated cold welding and breaking of the particles. Samples for TEM study were prepared by dispersing the mechanically attrited particles in the nickel foil by electrochemical co-deposition. TEM study of the initial stages of milling revealed that a localized structural change precedes the bulk amorphization process during mechanical alloying (MA). The sequence of phase evolution has been identified as:

(1) the formation of nanocrystalline supersaturated solid solution of aluminum in  $\alpha$ -zirconium, (2) amorphization of localized regions at powder interfaces, (3) ordering of aluminum-rich regions in the metastable Zr<sub>3</sub>Al (DO<sub>19</sub>) phase, and (4) bulk amorphization of the powders.

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#### Low-temperature structures of the second stage cesium graphitide and effect of trace impurities

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Stage 2 CsC<sub>24</sub> graphite-cesium derivatives were synthesized and characterized using x-ray diffraction. Pure CsC<sub>24</sub> specimens are single phase stage 2 in the range 77 K-300 K. From the 00/*l* scans we observed on samples slightly polluted during the transfer in the glove box, the tridimensional segregation of stage 2 in a main CsC<sub>26</sub> structure and an additional dense CsC<sub>20</sub> structure. The 2D Laue diffraction photographs are the same as already reported, but are interpreted as a mixture of  $2 \times 2$  R 0° lattice commensurate and  $2.54 \times 2.54$  R 14.5° lattice incommensurate structures. In oxidized compounds, we identified another  $2.23 \times 2.23$  R 25.5° 2D additional incommensurate structure corresponding to the composition CsC<sub>20</sub> already evidenced by analysis of the 00/*l* diffractograms. We suggest that oxygen impurities are at the origin of this particular stiff and dense structure at low temperature.

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#### Effects of lanthanum modification on rhombohedral Pb(Zr<sub>1-x</sub>Ti<sub>x</sub>)O<sub>3</sub> ceramics: Part I. Transformation from normal to relaxor ferroelectric behaviors

X. Dai, Z. Xu, J-F. Li, D. Viehland  
(University of Illinois)

The interruption of long-range polar order in rhombohedral ferroelectric Pb(Zr<sub>1-x</sub>Ti<sub>x</sub>)O<sub>3</sub> (PZT) ceramics has been systematically studied by incorporating La onto the A-site of the perovskite (ABO<sub>3</sub>) structure for Zr/Ti-ratios of 65/35 and 80/20 and various La-contents. Studies have been performed by hot-stage transmission electron microscopy, dielectric spectroscopy, and Sawyer-Tower polarization (P-E) techniques. The evolution of a polar nanodomain state from a normal micron-sized domain state with increasing La-content was observed. The emergence of this polar cluster state was characterized by the onset of strong frequency dispersion in the dielectric response, indicative of relaxor behavior. The La-content which drives the structure into the relaxor state was found to be related to the lattice distortion of the undoped base composition.

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#### Effects of lanthanum modification on rhombohedral Pb(Zr<sub>1-x</sub>Ti<sub>x</sub>)O<sub>3</sub> ceramics: Part II. Relaxor behavior vs. enhanced antiferroelectric stability

X. Dai, Z. Xu, J-F. Li, D. Viehland  
(University of Illinois)

Lanthanum modified lead zirconate titanate ceramics Pb<sub>1-3/2x</sub>La<sub>x</sub>(Zr<sub>1-y</sub>Ti<sub>y</sub>)O<sub>3</sub> (PLZT 100x/100(1-y)/100y) with Zr/Ti ratios close to the antiferroelectric-ferroelectric (AFE-FE) phase boundary were investigated by dielectric spectroscopy, Sawyer-Tower polarization techniques, and electron microscopy. An AFE<sub>in</sub> phase was found to be stabilized from the rhombohedral FE state in the compositional series 100x/90/10 for  $x \geq 0.02$ . The La<sub>3</sub>-content required to induce the AFE<sub>in</sub> state increased as the Ti-content was increased. For 100x/85/15, a state with relaxor-like dielectric behavior and nanodomains was observed to develop with increasing La-content, however the double-loop like P-E curves were suggestive of antiferroelectric behavior. Investigations for the composition 6/85/15 revealed the formation of nanodomains from the AFE<sub>in</sub> modulation, where the size of the nanodomains equaled the value of the

$AFE_n$  modulation wavelength. For this composition, P-E studies revealed double hysteresis characteristics, whereas dielectric investigations revealed relaxor-like behavior. It is suggested that the order within the nanodomain state may be antipolar over a range of compositions in high La-content rhombohedral PLZT ceramics.

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#### Sol-gel synthesis of microcrystalline rare earth orthophosphates

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Studies of the reactions between rare earth salts and phosphoric acid in aqueous or ethanolic media have shown that in both cases stable gels can be formed. Upon drying, gels prepared in aqueous environments yield macrocrystalline  $REPO_4$  products similar to those produced by conventional precipitation and drying. Gels prepared in ethanol, on the other hand, undergo dehydration to form dense microcrystalline products. This observation is based on optical and scanning electron microscopy, as well as on x-ray diffraction studies and infrared spectroscopy. These techniques, as well as differential thermal analysis, indicate that crystal growth of these products takes place around 600-700°C. The composition of the dehydrated gels produced in both the aqueous and ethanolic systems corresponds to an orthophosphate structure. Other characteristics of the non-crystalline  $REPO_4$  products include high resistance to attack by water, absence of coloration upon exposure to gamma rays, and a high index of refraction.

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#### The role of ordered $A_1$ -site vacancies in belt nano-domains of $Pb_{1-x}Ba_xNb_2O_6$ (PBN) solid solution

X. Xiao, Y. Xu, Z. Zeng, Z. Gui, L. Li, X. Zhang  
(Tsinghua University)

The order-disorder states of the A-site vacancy of PBN solid solution affected by different thermal treatments were studied with the aid of high resolution electron microscopy (HREM). PBN ceramics around the morphotropic phase boundary were prepared through two routes to control ordering degree of the A-site vacancy: (1) samples through quenching processes resulted in chaotic states of the A-site vacancies and misfit anti-phase boundary; (2) samples through slowly cooling led to an ordered structure of the vacancies in the  $A_1$ -site. The ordered  $A_1$ -site vacancies were modulated by interchanges of the sublattices of the ordered vacancies and the  $Pb^{2+}$  cations both in the  $A_1$ -sites along  $<-1/2\ 0\ 0>_p$  and  $<0\ -1/2\ 0>_p$  orientations, forming a narrow discommensurate wall between two anti-phase domains. The anti-phase domains were observed as a regular belt structure with dimensions of about 45 nm x ( $\geq 120$ ) nm. The belt nano-domain structures were a result of quasi-equilibrium thermodynamic processes.

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#### Elevated temperature deformation of fine-grained $La_{0.9}Sr_{0.1}MnO_3$

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Compressive creep behavior of fine-grained (5  $\mu$ m)  $La_{0.9}Sr_{0.1}MnO_3$  with a relative theoretical density between 85-90% was investigated over the temperature range 1150-1300°C in air. The fine grain size, brief creep transients, stress exponent close to unity and absence of deformation induced dislocations suggested that the deformation was controlled by a diffusional creep mechanism. The activation energy for creep of  $La_{0.9}Sr_{0.1}MnO_3$  was 490 kJ/mole. A comparison of the activation energy for creep of  $La_{0.9}Sr_{0.1}MnO_3$  with existing diffusion and creep data for perovskite oxides suggested that the diffusional creep of  $La_{0.9}Sr_{0.1}MnO_3$

was controlled by lattice diffusion of the cations, either lanthanum or manganese.

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#### Preparation of $NiFe_2O_4$ powder by spray pyrolysis of nitrate aerosols in $NH_3$

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To avoid the formation of hollow spheres during spray pyrolysis,  $NH_3$  was employed to change the mechanism of forming  $NiFe_2O_4$  from aerosols, containing Ni(II) and Fe(III) nitrates in the required stoichiometric ratio. Nearly spherical, solid submicron  $NiFe_2O_4$  particles with narrow size distribution were produced in one step using a dilute aqueous solution at pyrolysis temperatures as low as 823 K. However, higher pyrolysis temperatures ( $\geq 1023$  K) reduced the oxides to metallic alloy of Ni and Fe due to dissociation of  $NH_3$ . The forming steps and possible reaction mechanisms for aerosol droplets involved in the process were discussed.

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#### Microstructural development of ZnO using a rate controlled sintering dilatometer

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Rate controlled sintering (RCS) of isostatically pressed particulate compacts of ZnO showed lower average grain sizes and intragranular pore densities than constant heating rate temperature controlled sintering. Valid comparisons of this form could only be made after corrections to hardware and software which reduced specimen creep under dilatometer pushrod load, non-uniform pushrod expansion, reproducible specimen temperature determination, thermal expansion during sintering, and instantaneous termination of sintering at the specified end of RCS. The improved microstructures from RCS were attributed to maximized efficiency of densification, optimizing the time and temperatures permitted for grain growth.

Order No.: JA603-016

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#### Hydroxyapatite coatings with a bond coat of biomedical implants by plasma projection

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The deposition of hydroxyapatite coatings on titanium substrates, such as those used in biomedical implants, was studied by the technique of plasma projection. The results obtained with two types of precursor powder, without a bond coat and with a calcium silicate bond coat, are presented and compared.

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#### Effective optical constants $n$ and $\kappa$ and extinction coefficient of silica aerogel

J.S.Q. Zeng, R. Greif, P. Stevens, M. Ayers, A. Hunt  
(University of California-Berkeley)

In this work the normal reflectance,  $R$ , at a planar silica aerogel interface and the normal transmittance,  $T$ , of a silica aerogel slab were measured using a Fourier transform infrared spectrometer. Two procedures were used to obtain the effective optical constants, i.e., the refractive index  $n$  and the absorption index  $\kappa$ , of silica aerogel. One procedure determined  $\kappa$  from the measured transmittance  $T$  and then determined  $n$  from the results for  $\kappa$  and from the measured reflectance  $R$  using the Kramers-Kronig relation; the other procedure determined  $n$  and  $\kappa$  of silica aerogel from  $n$  and  $\kappa$  of fully dense silica glass by using the Clausius-Mossotti equation, Maxwell Garnett formula and Bruggeman formula. The first procedure has a relatively large error due to the

inaccuracy of the transmission and reflection measurements. The second procedure, especially the Clausius-Mossotti equation, yields values of  $n$  that are consistent with experiments and may be used for the calculation of the effective optical constants and the extinction coefficient of silica aerogel.

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#### On the optimization of a DC arcjet diamond chemical vapor deposition reactor

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Based on results from chemical kinetic model calculations, a method to improve diamond film growth in a DC arcjet chemical vapor deposition reactor has been developed. Introducing the carbon source gas ( $\text{CH}_4$ ) into an Ar/ $\text{H}_2$  plasma in close proximity to the substrate produced diamond films exhibiting simultaneous improvements in quality and mass deposition rates. These improvements result from a reduced residence time of the methane in the plasma which inhibits the hydro-carbon chemistry in the gas from proceeding significantly beyond methyl radical production prior to encountering the substrate. Improvements in growth rate were modest, increasing by only a factor of two. Optical emission actinometry measurements indicate that the flux of atomic hydrogen across the stagnation layer to the substrate is mass diffusion limited. Since diamond growth depends upon the flux of atomic H to the substrate, these results suggest that under the conditions examined here, a low atomic H flux to the substrate poses an upper limit on the attainable diamond growth rate.

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#### Synthesis of stoichiometric lead molybdate $\text{PbMoO}_4$ : An x-ray diffraction, Fourier transform infrared spectroscopy and differential thermal analysis study

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(National University of Singapore)

$\text{PbO}/\text{MoO}_3$  system with 47%:53%, 53%:47% and 50%:50% molar ratios at various processing temperatures has been studied with XRD, FTIR, and DTA methods. It is found that in addition to the crystallization of primary  $\text{PbMoO}_4$  phase, sub-phases such as  $\text{Pb}_2\text{MoO}_5$  and  $\text{PbMo}_2\text{O}_7$  are also formed. The remaining PbO and  $\text{MoO}_3$  are detected at certain stages of the thermal process due to localized powder inhomogeneity. Physical processes, such as sublimation, eutectic melting, solid to liquid, and liquid to vapor transformations are also investigated. In particular, evaporations of excessive PbO or  $\text{MoO}_3$  in the non-stoichiometric  $\text{PbO}/\text{MoO}_3$  can be correlated to thermal processing parameters. The current study has led to the following three processing guidelines to obtain stoichiometric  $\text{PbMoO}_4$ : (1) for high-temperature application, such as the Czochralski melt growth, it is suggested an excessive  $\text{MoO}_3$  (a few mol.%) must be included and a slow heating rate should be employed; (2) for low-temperature synthesis, the stoichiometric  $\text{PbO}-\text{MoO}_3$  can be used but with a fast heating rate; and (3) PbO rich  $\text{PbO}/\text{MoO}_3$  system is not recommended in  $\text{PbMoO}_4$  synthesis.

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#### Space filling by nucleation and growth in chemical vapor deposition of diamond

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Phase transformations, including chemical vapor deposition (CVD) of diamond, taking place by nucleation and growth are commonly described by Avrami or Johnson-Mehl type models. In order to avoid the restrictions of such models with respect to assumptions concerning nucleation rates and growth velocities, the variation with time of nucleation and growth of diamond particles during the deposition by micro-

wave plasma-assisted CVD was studied. The size distributions obtained from image analysis enabled us to trace back details of the nucleation and growth history. Three sources of particle formation were operating during deposition. A general growth law suitable for all particles did not exist. These observations limited the applicability of Avrami-type models to describe space filling. Computer simulation of surface coverage and particle growth was successful because one particular mode of particle formation and growth dominated surface coverage. Based on image analysis and the determination of the film growth rate, the evolution of the diamond volume fraction with time, starting from three-dimensional particle growth followed by a continuous transition to one-dimensional film growth, was described.

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#### The extent of solid solubility in the $\text{RuO}_2$ - $\text{TiO}_2$ system

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$\text{RuO}_2$  single crystals were obtained by evaporation of PbO from  $\text{Pb}_2\text{Ru}_2\text{O}_{6.5}$  at high temperatures and were verified as good standards for WDS analysis. They were used for the investigation of phase equilibria and the extent of solid solubility in the  $\text{RuO}_2$ - $\text{TiO}_2$  system by WDS quantitative microanalysis. The solid solubility at 1350°C was determined to be 16.5%  $\text{TiO}_2$  in  $\text{RuO}_2$  and 13.5%  $\text{RuO}_2$  in  $\text{TiO}_2$ .

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#### Amorphization of graphite induced by mechanical milling and subsequent crystallization of the amorphous carbon upon heat treating

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The effects of mechanical milling (MM) on the phase transformation of graphite carbon were investigated using high resolution electron microscopy (HREM), x-ray diffraction and differential thermal analysis (DTA). Amorphization of graphite as a result of prolonged high energy ball milling was directly observed with HREM. The exothermic peak in the DTA trace of the ~200 hour ball milled sample indicated a crystallization onset temperature of about 670°C and crystallization activation energy of 234 kJ/mole.

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#### Electrically conductive CuS-poly(acrylic acid) composite coatings

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Copper sulfide (CuS) powder precipitated from chemical bath containing copper(II) chloride and thiourea and annealed in air at 150°C for 1 h was dispersed in poly(acrylic acid) aqueous solution (with additional water or propylene glycol as a dispersive agent) and cast on glass slides. Upon evaporation of the solvent, coatings of ~50 nm in thickness of a CuS poly(acrylic acid) composite is formed. Measurement of sheet resistance ( $R_{\square}$ ) indicates a percolation threshold of electrical conduction at a weight fraction (wf-% weight of CuS to poly(acrylic acid)+CuS) of about 40%: the composite undergoes a transition from insulator ( $R_{\square} \sim 10^{13}\Omega$ ) to conductive state ( $R_{\square} \sim 10^2\Omega$ ). The morphology and thermal stability of the composite depend on the choice of the dispersive agent for the CuS powder: smoother and thermally stable (up to a temperature of 250°C) coatings are obtained when propylene glycol is used. The results on x-ray diffraction, thermogravimetric analysis, and Fourier transform infrared spectroscopy studies are given to indicate the structure and bonding mechanisms and their dependence on temperature and dispersive agent.

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**Characterization of SiC fiber (SCS-6) reinforced-reaction formed silicon carbide matrix composites**M. Singh, R.M. Dickerson  
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Silicon carbide fiber (SCS-6) reinforced-reaction formed silicon carbide matrix composites were fabricated using reaction forming process. Silicon-2 at.% niobium alloy was used as an infiltrant instead of pure silicon to reduce the amount of free silicon in the matrix after reaction forming. The matrix primarily consists of silicon carbide with a bi-modal grain size distribution. Minority phases dispersed within the matrix are niobium disilicide ( $\text{NbSi}_2$ ), carbon and silicon. Fiber push-out tests on these composites determined a debond stress of ~67 MPa and a frictional stress of ~60 MPa. A typical four point flexural strength of the composite is 297 MPa (43.1 KSi). This composite shows tough behavior through fiber pull-out.

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**Influences of stress on the measurement of mechanical properties using nanoindentation: Part I. Experimental studies in an aluminum alloy**

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The influence of applied stress on the measurement of hardness and elastic modulus using nanoindentation methods has been experimentally investigated using special specimens of aluminum alloy 8009 to which controlled stresses could be applied by bending. When analyzed according to standard methods, the nanoindentation data reveal changes in hardness with stress similar to those observed in conventional hardness tests. However, the same analysis shows that the elastic modulus changes with stress by as much as 10%, thus suggesting that the analysis procedure is somehow deficient. Comparison of the real indentation contact areas measured optically to those determined from the nano-indentation data shows that the apparent stress dependence of the modulus results from an underestimation of the contact area by the nanoindentation procedures.

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**Influences of stress on the measurement of mechanical properties using nanoindentation: Part II. Finite element simulations**

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The finite element method has been used to study the behavior of aluminum alloy 8009 during elastic-plastic indentation to establish how the indentation process is influenced by applied or residual stress. The

study was motivated by the experiments of the preceding paper which show that nanoindentation data analysis procedures underestimate indentation contact areas and therefore overestimate hardness and elastic modulus in stressed specimens. The NIKE2D finite element code was used to simulate indentation contact by a rigid, conical indenter in a cylindrical specimen to which biaxial stresses were applied as boundary conditions. Indentation load-displacement curves were generated and analyzed according to standard methods for determining hardness and elastic modulus. The simulations show that the properties measured in this way are inaccurate because pile-up is not accounted for in the contact area determination. When the proper contact area is used, the hardness and elastic modulus are not significantly affected by the applied stress.

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**Microtexture of highly crystallized graphite as studied by galvanomagnetic properties and electron channeling contrast effect**

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Relationship between microtexture and crystallinity of highly crystallized graphites with the residual resistivity ratio  $\rho_{300\text{K}}/\rho_{4.2\text{K}}$  of 3.45-5.50 was investigated. The graphite crystals studied were kish graphite (KG), highly oriented pyrolytic graphite (HOPG) and highly crystallized graphite films prepared from carbonized aromatic polyimide films. The study was made by the observations of electron channeling pattern and electron channeling contrast image (ECI) under scanning electron microscope and the measurements of x-ray diffraction, magnetoresistance and Hall coefficient. The values of the mean free path of the carriers  $\lambda$ , which approximates the mean crystal grain size, were estimated to be 2.6-6.1  $\mu\text{m}$  from the magnetoresistance at 4.2 K for the highly crystallized graphites. The values of the average crystal grain diameter  $D$  in the basal plane evaluated from ECI were several hundred microns or more for KG, 60  $\mu\text{m}$  for HOPG and 6 and 12  $\mu\text{m}$  for the graphite films. The difference between the values of  $\lambda$  and  $D$  for each crystallized graphite was discussed in relation to other results obtained.

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