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ABSTRACTS

COMMUNICATIONS

Thermal stresses in multilevel interconnections: Aluminum lines at different levels

Y-L. Shen
(The University of New Mexico)

Numerical results on the evolution of thermal stresses in multilevel interconnects are presented. Two levels of aluminum lines with an aspect ratio of unity, aligned vertically or arranged in a staggered manner, are considered by recourse to the finite element analysis. The stresses are found to be significantly higher in the lower-level lines than in the upper-level lines, for both the aligned and staggered arrangements. The stress magnitudes are generally smaller in lines of staggered arrangement, compared to the case of aligned lines. Implications of the present findings are discussed, with directions of future studies highlighted.

Order No.: JA709-001

Effects of gravity on processing heavy metal fluoride fibers

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The effects of gravity on the crystal nucleation of heavy metal fluoride fibers have been studied in preliminary experiments utilizing NASA's KC-135 reduced gravity aircraft and a microgravity sounding rocket flight. Commercially produced fibers were heated to the crystallization temperature in normal and reduced gravity. The fibers processed in normal gravity showed complete crystallization while the fibers processed in reduced gravity did not show signs of crystallization.

Order No.: JA709-002

ARTICLES

ac irreversibility line of bismuth-based high-temperature superconductors

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We discuss the magnetic properties of lead doped Bi-2223 bulk samples obtained through combined magnetic melt texturing and hot pressing (MMTHP). The ac complex susceptibility measurements are achieved over a broad ac field range ($1\ Oe < h_{ac} < 100\ Oe$) and show highly anisotropic properties. The intergranular coupling is improved in the direction perpendicular to the applied stress and magnetic field direction and an intragranular loss peak is observed for the first time. A comparison is made with other bismuth-based compounds and it is shown that the MMTHP process shifts the ac irreversibility line (ac IL) toward higher fields. It is also shown that all the ac IL's for quasi 2D bismuth-based compounds show a nearly quadratic temperature dependence and deviate, therefore, strongly from the linear behavior observed in quasi 3D compounds and expected from a critical state model.

Order No.: JA709-003

Two interferometric methods for the mechanical characterization of thin films by bulging tests. Application to silicon single crystal

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Two optical methods are presented for the mechanical characterization of thin films, namely real time holographic interferometry and a fringe projection method called "contouring." These two methods are coupled to the interferometry by the phase measurements, thus allowing the displacement field to be measured at all points on the membrane. We discuss the solutions retained in terms of their precision and sensitivity. These methods are then applied to membrane bulging tests, a type of test which is widely used in micro-mechanical studies. The measurements are performed on silicon single crystal and the results are compared to the solutions calculated by finite element methods. In both cases, the good agreement between theory and experiments allows the experimental apparatus to be validated.

Order No.: JA709-004

Microstructure, electrical properties, and thermal stability of Au-based ohmic contacts to p-GaN

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The work described in this paper is part of a systematic study of ohmic contact strategies for GaN-based semiconductors. Au contacts exhibited ohmic behavior on p-GaN when annealed at high temperature. The specific contact resistivity (ρ_c) calculated from TLM measurements on Au/p-GaN contacts were 53 $\Omega \cdot cm^2$ after annealing at 800°C. Multilayer Au/Mg/Au/p-GaN contacts exhibited linear, ohmic current-voltage (I-V) behavior in the as-deposited condition with $\rho_c \approx 214\ \Omega \cdot cm^2$. The specific contact resistivity of the multilayer contact increased significantly after rapid thermal annealing (RTA) through 725°C. Cross-sectional microstructural characterization of the Au/p-GaN contact system via high-resolution electron microscopy (HREM) revealed that interfacial secondary phase formation occurred during high-temperature

treatments, which coincided with the improvement of contact performance. In the as-deposited multilayer Au/Mg/Au/p-GaN contact, the initial 320 Å Au layer was found to be continuous. However, Mg metal was found in direct contact with the GaN in many places in the sample after annealing at 725°C for 15 s. The resultant increase in contact resistance is believed to be due to the barrier effect increased by the presence of the low work function Mg metal.

Order No.: JA709-005

Annealing induced interdiffusion and crystallization in sputtered amorphous Si/Ge multilayers

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The intermixing and crystallization of amorphous Si/Ge multilayers (with individual layer thickness between 1.5 and 20 nm) and SiGe alloys produced by DC magnetron sputtering have been studied by cross-sectional electron microscopy and x-ray diffraction.

Measurement of the crystallization temperature as a function of the Si content showed that multilayers and alloys with equal composition crystallized at the same temperature. This implies that intermixing precedes crystallization in the multilayers.

Close to the crystallization temperature, formation of Kirkendall voids was observed in the short-period Si/Ge multilayers. These voids were found at positions corresponding to the original Si layers indicating that Si diffuses faster in amorphous Ge than Ge in amorphous Si.

The Ge layers in short-period Si/Ge multilayers retained their amorphous state to much higher temperatures than thick amorphous Ge layers. This is shown to be due to inhibition of nucleation by the presence of the layer interfaces.

A lower estimate for the Si diffusion constant in crystalline Ge is also determined.

Order No.: JA709-006

Micro-Raman analysis of residual stresses and phase transformations in crystalline silicon under microindentation

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Vickers microindentations obtained with loads comprised between 0.05N and 2N were performed on crystalline (100) silicon. The residual stress field and the different structural states induced by loading were studied by mapping the indented zones by their micro-Raman response. A Raman signature of amorphous silicon is found in the center of the impression. The energy of the Γ_{25} zone center phonon is found to vary from 522 cm^{-1} when probing the silicon at a distance of 80 μm from the center of the indentation up to 527 cm^{-1} when probing the pile-up region of the impression. When probing cracked zones in the vicinity of the pile-up region, wavenumbers as high as 536 cm^{-1} are measured. The stress components induced by a point indentation (1N) have been calculated from analytical expressions given in the literature. For an average conversion factor of 3.2 cm^{-1}/GPa, the residual local stresses after unloading are found of the same order of magnitude or even larger than the calculated stresses which are generated during loading. A tentative explanation is proposed. Finally, a systematic laser induced thermal treatment of the central area and of the pile-up region of indentations was performed. It is shown that the amorphous silicon in the center can partly recrystallize but that the residual stress state in the pile-up region cannot be completely relaxed by local laser heating.

Order No.: JA709-007

The growth of decagonal Al-Co-Ni single crystals as a function of chemical composition

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Decaprismatic single crystals taken from a series of alloys of nominal compositions within $Al_{65-77}Co_{3-22}Ni_{3-22}$ have been studied by means of x-ray diffraction techniques. The substitution of Co by Ni in increasing amounts changes the (pseudo)decagonal diffraction patterns drastically and indicates structural changes which range from a single-crystalline approximant via orientationally ordered nanodomain structures and quasiperiodic phases with different types of ordering phenomena, to a basic decagonal phase. A quantum phase diagram analysis shows a clear separation of the stability regions of the ternary systems described in this study and other decagonal phases.

Order No.: JA709-008

Correlation between energy transfers and solid state reactions induced by mechanical alloying on $Mo_{33}Si_{66}$ system

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Phase transformations of $Mo_{33}Si_{66}$ powder mixture under different milling conditions have been systematically investigated by x-ray diffraction, scanning electron microscopy, and transmission electron microscopy. The effect of the milling conditions on the Mo/Si solid state reactions (SSR) has been examined in detail. The energy transfer from the milling tools to the powder under processing has been quantified by an already assessed collision model. It has been found that the higher energetic input favors the formation of the room temperature stable phase α-$MoSi_2$, while the lower energetic input promotes the formation of the metastable phase β-$MoSi_2$. In addition, if the energy transfer is high enough, the Mo/Si reaction proceeds in a form of self-propagating high-temperature synthesis (SHS). Thermodynamics and kinetics aspects related to the different SSRs have been discussed.

Order No.: JA709-009

Thermal cycling effects in high-temperature Cu-Al-Ni-Mn-B shape memory alloys

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The effects of thermal cycling through the martensitic transformation have been studied in three Cu-Al-Ni-Mn-B high-temperature shape memory alloys. An increase of the martensitic transformation temperatures with the number of cycles (up to ~7 K after 60 cycles) has been generally observed by DSC measurements. The microstructure of these alloys is rather complicated, with the presence of big manganese or aluminium boride particles and small boron precipitates, as well as the formation of dislocations during thermal cycling. By means of aging experiments, it has been shown that the evolution of transformation temperatures during cycling is mainly due to the step-by-step aging in parent phase accompanying the thermal cycling, and that the dislocations formed during cycling have only a very small effect, at least up to 60 cycles.

Order No.: JA709-010

Aging of the Inconel 718 alloy between 500 and 750°C

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The aging of the NC 19 Fe Nb alloy (Inconel 718), previously quenched from 990°C, is characterized by a hardness peak at 650°C, then a maximum in hardness at about 750°C. Over this temperature, the hardness progressively decreases. In the 550–650°C temperature range, TEM observations have revealed that β (Ni_3Nb) precipitates are formed as long platelets parallel between them within the same grain, as well as extremely fine γ′[Ni_3(Ti,Al)] particles responsible for the observed improvement in hardness. For a tempering temperature higher than 650°C, a first hardening occurs after a 4 hour treatment, which has been associated with the γ′ phase precipitation, with a more or less spherical shape. Beyond this time, a second hardening takes place linked to the γ″ phase precipitation (Ni_3Nb, bct DO_{22} structure), as thin platelet shaped, perfectly coherent with the matrix. The misfit between the γ′ and γ″ phases is about 3% in the <001>γ″ direction and lower than 1% in the <100>γ″ and <010>γ″ directions. During a longer aging at 750°C, the γ″ platelets progressively dissolve while β precipitates are growing.

Order No.: JA709-011

Superplastic behavior of a kappa carbide material (Fe_3AlC_x)
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Fine-grained kappa carbide (Fe_3AlC_x) materials, containing 12.5 and 14%Al, and 3.5%C, were prepared by powder processing and hipping procedures. The creep behavior of the kappa materials was shown to be identical to that observed in superplastic iron carbide, and was shown to follow a grain-boundary-diffusion controlled grain boundary sliding relation. The tensile fracture strains in kappa, however, were shown to be considerably less than in iron carbide with a maximum elongation of 92% noted. This difference is attributed to either a low stress intensity factor or to contamination of the powder surface in the kappa material. The compression creep strength, at a given strain rate, was shown to be about two times higher than the tension creep strength.
Order No.: JA709-012

Electrical and microstructural characteristics of Ge/Cu ohmic contacts to n-type GaAs
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It is shown that Cu-Ge alloys prepared by depositing sequentially Cu and Ge layers onto GaAs substrates at room temperature, followed by annealing at 400°C, form a low-resistance ohmic contact to n-type GaAs over a wide range of Ge concentration that extends from 15 to 40 at.%. The contacts exhibit a specific contact resistivity of 7×10^{-7} Ω cm^2 on n-type GaAs with doping concentrations of 1×10^{17} cm^{-3}. The contact resistivity is unaffected by varying the Ge concentration in the range studied and is not influenced by the deposition sequence of the Cu and Ge layers. Cross-sectional high-resolution transmission electron microscopy results show that the addition of Ge to Cu in this concentration range causes Cu to react only with Ge forming the ξ and ε_1-Cu_3Ge phases which correlate with the low contact resistivity. The ξ and ε_1-Cu_3Ge phases have a planar and structurally abrupt interface with the GaAs substrate without any interfacial transition layer. It is suggested that Ge is incorporated into the GaAs as an n-type impurity creating a highly doped n+-GaAs surface layer which is responsible for the ohmic behavior. n-channel GaAs metal-semiconductor field-effect transistors using ohmic contacts formed with the ξ and ε_1-Cu_3Ge phases demonstrate a higher transconductance compared to devices with AuGeNi contacts.
Order No.: JA709-013

Critical assessments of accommodation process by liquid phase for superplastic flow in Si_3N_4/Al-Mg-Si metal matrix composites
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A liquid phase serves to relax stress concentrations caused by sliding at interfaces and grain boundaries in high strain rate superplasticity for aluminum matrix composites. However, the presence of a liquid phase does not always lead to high strain rate superplasticity because too much liquid causes decohesion at a liquid phase. The critical conditions of the optimum distribution, thickness and volume in a liquid phase are discussed based on the observation results by differential scanning calorimetry and transmission electron microscopy. As a result, a very thin and discontinuous liquid phase is required both to assist relaxation of the stress concentrations and to limit decohesion at a liquid phase.
Order No.: JA709-014

Fabrication of multilaminated Si_3N_4-Si_3N_4/TiN composites and its anisotropic fracture behavior
J-L. Huang, Y-L. Chang, H-H. Lu
(National Cheng-Kung University)

Laminated composites containing alternate layers of Si_3N_4 and TiN/Si_3N_4 materials were used as model material for investigating the crack behaviors and mechanical properties.

Results indicated that both strength and toughness in laminated composites were higher than that of monolithic silicon nitride.

The failure profiles were affected by the stored strain energy prior to failure and the stress gradient in each layer. Cracks deviated successively from one layer to the other due to periodic stress distribution. Samples with better strength and toughness also had longer crack propagation path and higher amplitude of crack deviation.

The periodic stress distribution in laminated composites was confirmed by the measurements of indentation crack length. Results also suggested a tensile stress in Si_3N_4 layer, and compressive stress in TiN/Si_3N_4 layer, in directions normal to the free sample interface.
Order No.: JA709-015

A study of the frequency dependence of the dielectrophoretic effect in thermoset polymers
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Ceramic-polymer composites with a 1–3 connectivity can be created via a novel process called dielectrophoretic assembly. The process involves an electric field which is applied to a suspension of ceramic particles in an uncured thermoset polymer matrix. Under appropriate conditions, the applied electric field acts to induce a spatial redistribution of the particles into a chained or fibril structure. It was shown previously that the electrorheological response and fibril microstructure are dependent on both the frequency and magnitude of the applied alternating electric field. This paper will show that the frequency dependence of the uncured thermoset polymer suspensions results from the complex electrical phenomena specific to each thermoset system. Specifically, it will be shown through low field dielectric measurements and high field current-voltage analysis that the dielectrophoretic effect can be limited by electrode polarization, ionic conductivity and space charge relaxation. It is the frequency dependence of these limiting phenomena which give rise to the observed frequency dependence in the dielectrophoretic force of attraction being utilized to drive particulate assembly.
Order No.: JA709-016

Investigation of Si_3N_4-TiN/Si_3N_4-Si_3N_4 trilayer composites with residual surface compression
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(National Cheng Kung University)

The present study involved the fabrication of three-layered composites consisting of outer layers that contained Si_3N_4, and an inner layer that contained TiN in a Si_3N_4 matrix. Surface compressive stresses were developed upon cooling due to the relatively higher thermal expansion coefficient (CTE) in the inner layer. The flexural strength of layered Si_3N_4 composites was substantially greater than that of monolithic Si_3N_4. This was attributed to the surface compressive stress.

The effects of TiN composition and inner layer thickness on the mechanical properties were investigated. Layered samples containing 20 vol% TiN had lower flexural strength than Si_3N_4-10%TiN/Si_3N_4-Si_3N_4 due to the formation of microcracks in the inner layer.

Crack behaviors in layered samples were affected by the residual stress, interface and free sample surface.

Both theoretical and experimental results indicated that the strength and toughness of layered composites were substantially greater than those of monolithic materials. The determination of fracture toughness in three-layered materials by surface indentation technique should be done carefully due to the influence of residual stress.
Order No.: JA709-017

Domain pattern formation in ferroelastic $Pb_3(PO_4)_2$ by computer simulation
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A model of lead phosphate, which describes the rhombohedral-monoclinic phase transition, is used to form domain patterns in the annealing process. The obtained domain structures show *W* and *W′* types of domain walls in agreement with the stress-free laws proposed in the Sapriel's theory. The observed *W* domain walls are parallel to the ternary symmetry axis, while the *W′* ones are tilted with respect to the

same axis. The antiphase domain walls take no preferential orientations, and remain parallel to the ternary axis. The calculated density of the potential energy of the domain wall of type W is estimated to be E_{dw} = 49 K/Å^2 at T = 300 K.

Order No.: JA709-018

Electrical properties of ultrafine-grained yttria-stabilized zirconia ceramics

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Nanoparticles of yttria-doped tetragonal zirconia polycrystalline ceramics (Y-TZP) with an average crystallite size of less than 9 nm were prepared by a combustion synthesis process. Dense and fine-grained (< 200 nm) Y-TZP ceramics were obtained by fast-firing using temperatures lower than 1400°C and dwell times of less than 2 minutes. Impedance spectroscopy was employed to measure conductivities of oxygen vacancies in the grain and the grain boundary of the fine-grained Y-TZP. The relationships between the concentration of the oxygen vacancies in the grain boundary and measurable physical parameters were determined semi-quantitatively. The oxygen vacancy concentrations and activation energies for the oxygen-ion conduction in the grain and the grain boundary of the fine-grained Y-TZP were found to be independent of the average grain size in the average grain-size range of 90~200 nm. These experimental results suggest that, in order to retain the abnormally high oxygen vacancy concentrations of the Y-TZP nanoparticles and thus enhance the oxygen-ion conductivity, it may be necessary to decrease the average grain size to approximately 10 nm.

Order No.: JA709-019

Thermal diffusivity maps: Case studies in ceramics

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A new methodology for mapping thermal diffusivity using a photothermal deflection method is introduced. Two case studies are made: fiber-reinforced composite structures, and contact damage zones in alumina. In the former, characterization of thermal microstructural features is demonstrated; in the latter, microcrack density is quantified. Experimental data are analyzed and compared with literature results. Advantages and limitations of the technique are discussed.

Order No.: JA709-020

X-ray photoelectron spectroscopy characterization of barium titanate ceramics prepared by the citric route. Residual carbon study

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Powder and ceramics of barium titanate prepared by the citric process were studied by x-ray photoelectron spectroscopy (XPS). Spectra of C_{1s}, O_{1s}, Ti_{2p}, Ba_{3d} and Ba_{4d} levels are analyzed in powder and ceramics immediately after the sintering step and after several months of exposure in the air. Ar-ion etching allowed characterization of the material intrinsic carbon. The results are discussed in comparison with works previously published on oxide single crystals.

Order No.: JA709-021

Characteristics of TiN film deposited on stellite using reactive magnetron sputter ion plating

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TiN films were deposited onto stellite 6B alloy (Co base) by the reactive magnetron sputter ion plating. As the substrate bias increases, TiN film changes from columnar structure to dense structure with great hardness and smooth surface due to densification and resputtering by ion bombardment. The content of oxygen and carbon impurities in the TiN film decreases greatly when the substrate bias is applied. The preferred orientation of the TiN films changes from (200) to (111) with a decrease of the N_2/Ar ratio and from (200) to (111) and then (220) with an increase of the substrate bias. The change of the preferred orientation is discussed in terms of surface energy and strain energy which are related with the impurity contents and the ion bombardment damage. The hardness of the TiN film increases with increasing compressive stress generated in the film by virtue of ion bombardment. It becomes as high as up to 3500 kgf/mm^2 when an appropriate substrate bias is applied.

Order No.: JA709-022

Deposition of mullite and mullite-like coatings on silicon carbide by dual-source metal plasma immersion

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Mullite and mullite-like coatings on silicon carbide have been produced by a metal plasma immersion ion implantation and deposition technique based on two cathodic vacuum arc sources and concurrent pulse biasing of the substrate in an oxygen atmosphere. The deposition was carried out at oxygen partial pressures of between 0.66 and 3.33 Pa. The Al:Si ratio in the films varied from 1:1 to 8:1 and was controlled by varying the pulse duration of the separate plasma guns. High bias voltage was used early in the deposition process in order to produce atomic mixing at the film-substrate interface, while lower bias voltage was used later in the deposition; low ion energy allows control of the physical properties of the film as well as faster deposition rates. The as-deposited films were amorphous, and crystalline mullite was formed by subsequent annealing at 1100°C for 2 hours in air. Strong adhesion between the mullite and the SiC was achieved, in some cases exceeding the 70 MPa instrumental limit of our pull-tester.

Order No.: JA709-023

Bonding behavior of Cu/CuO thick film on a low-firing ceramic substrate

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The adhesion strength between a low-firing substrate consisting of an alumina/glass composite and a copper thick film was affected by the addition of cupric oxide and glass frit to the copper paste in a new co-firing process. An interlayer, 3~4 μm in thickness, was produced in the metal-ceramic interface during the new co-firing process due to the diffusion of copper. At the same time, the adhesion strength was improved by controlling the cupric oxide content.

The addition of about 3 wt.% glass frit (softening point = 670°C, based on the calcium-barium borosilicate glass composition) to the metal paste resulted in highest adhesion strength of 3 kg/mm^2 with a shift of the debonding site toward the ceramic substrate within the interlayer. The shift of the debonding site could be observed by comparing the ratios of Al_2O_3/Cu and Ca concentration at the test pad areas on the substrate after debonding. The shift of the debonding site is attributed to the migration of glass frit into the interfacial region. The migration of glass frit occurred easily when the softening point of the glass frit was compatible with the new co-firing process, regardless of how much frit was used.

Order No.: JA709-024

Carbothermal synthesis of TaC whiskers via a vapor-liquid-solid growth mechanism

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Tantalum carbide whiskers have been synthesized via a vapor-liquid-solid (VLS) growth mechanism in the temperature region 1200–1300°C in nitrogen or argon. The starting materials consisted of Ta_2O_5, C, Ni and NaCl. Carbon was added to reduce tantalum pentoxide, via a carbothermal reduction process, and Ni was used to catalyze the whisker growth. Thermodynamic calculations showed that tantalum is transported in the vapor phase as an oxochloride rather than as a chloride. An alkali metal chloride such as NaCl can be used as a source of Cl.

The formation of TaC whiskers was found to be strongly dependent on the processing conditions used, on the choice of precursor materials (e.g. their particle sizes), and on the mixing procedure. So far we have obtained TaC-whisker in a yield of 75–90 vol%. These whiskers are

0.1–0.6 μm in diameter and 10–30 μm in length, and they are straight and exhibit smooth surfaces. The main impurities are TaC particles, minor amounts of unreacted carbon and remnants of the Ni catalyst.
Order No.: JA709-025

Computer simulation of ferroelastic phase transition in $LaNbO_4$
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A model of lanthanum orthoniobate which possesses a ferroelastic tetragonal-monoclinic phase transition is proposed. It contains only one particle per unit cell, but it is constructed consistently with symmetry changes at the phase transition. The model parameters are chosen to reproduce the bare soft mode, degree of deformation of the tetragonal unit cell to monoclinic one, and the phase transition temperature. The ferroelastic system with free boundary conditions was simulated by the molecular dynamics technique and the second order phase transition was reproduced. The studied annealing process shows formation of the stripe lenticular domain pattern, which has been interrupted by the appearance of temporary bands of perpendicularly-oriented lenticular domains. The maps contain only *W*'-type domain walls, which orientations are fixed by interplay of potential parameters and not by symmetry elements. The simulated domain pattern has the same features as those observed by transmission electron microscope.
Order No.: JA709-026

Analytical electron microscopy of planar faults in SrO-doped $CaTiO_3$
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Oxide-rich planar faults within a perovskite matrix are the prevailing type of extended defects in polycrystalline SrO-doped $CaTiO_3$. These defects form, depending on the temperature of sintering, random networks or ordered structures. The chemistry of the polytypoid, the isolated planar faults and the perovskite phase have been studied by spatially resolved electron energy loss and energy-dispersive x-ray spectroscopies using a dedicated scanning transmission electron microscope. We have found that Sr ions from SrO additions preferably substitute Ca in the $CaTiO_3$ lattice, thus forming a solid solution $(Ca_{1-x}Sr_x)TiO_3$. The surplus of Ca ions form single and ordered CaO-rich planar faults in the host $(Ca_{1-x}Sr_x)TiO_3$ phase. Whereas the excess Ca segregates in a form of single planar faults at lower temperatures, it forms a stable polytypoidic phase at higher temperatures. For materials having up to 25 mol% of SrO additions, this phase has $(Ca_{1-x}Sr_x)_4Ti_3O_{10}$ composition, comprising a sequence of CaO faults followed by three $(Ca_{1-x}Sr_x)TiO_3$ perovskite layers. Analytical electron microscopy revealed that the composition of the single planar faults, formed at lower temperatures, is identical to that of polytypoids, which are stable at higher sintering temperatures.
Order No.: JA709-027

Effect of rate-controlled sintering on microstructure and electrical properties of ZnO doped with bismuth and antimony oxides
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Various rate-controlled sintering (RCS) schedules were used on isostatically pressed particulate compacts of ZnO with Bi_2O_3 and Sb_2O_3 additives. For low additive content, smaller average grain sizes with more rapid RCS schedules were attributable to thermal schedules which minimized the time at elevated temperatures where grain growth could occur. β-Bi_2O_3, $Zn_7Sb_2O_{12}$, and $Zn_2Sb_3Bi_3O_{14}$ phases formed during/after sintering. Elevated heat treatment temperatures favored the formation of $Zn_7Sb_2O_{12}$ and additional β-Bi_2O_3, while $Zn_2Sb_3Bi_3O_{14}$ was dominant in sintered samples where the RCS schedule did not result in temperatures in excess of 1100°C. $Zn_2Sb_3Bi_3O_{14}$ precipitated during sintering, functioning as grain boundary pinning sites which impeded ZnO grain growth. Bismuth and antimony oxide-based liquid facilitated sintering at lower temperatures, which in turn resulted in decreased average grain size. Rapid RCS schedules for samples with low dopant content resulted in lower sintering temperatures, since time was not allowed for $Zn_2Sb_3Bi_3O_{14}$ precipitation to deplete the liquid phase. For higher dopant contents, liquid phase was adequately plentiful, wherein longer RCS schedules resulted in lower sintering temperatures. Increasing concentration of second phase generally fostered decreased grain size and attenuated the effect of thermal schedule on the microstructure. Electrical resistance and breakdown voltage increased consistent with decreasing ZnO average grain size.
Order No.: JA709-028

An alternative method for penetration depth determination in nanoindentation measurements
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The information provided by the shape of the unloading portion of indentation curves is used to calculate the area of contact between the indentor and the material. Results obtained in fused silica and nickel, including Young's modulus and hardness values, are presented to illustrate the validity of the approach. It is shown that errors of only a few percent are introduced when fitting unloading curves with power laws. The present method is especially useful when direct specimen stiffness measurements can be performed.
Order No.: JA709-029

Determination of Young's modulus by spherical indentation
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In this paper we consider elastic-plastic materials which are tested by spherical indentation. Finite element calculations, which take into account nonlinear geometry properties, are carried out in order to determine the influence of the plastic history on the unloading response of the material. Two different iterative methods are proposed for determining Young's modulus under the assumption of a bilinear plasticity law. The first method deals with loading and unloading parts of the indentation test, whereas the second one deals with unloading parts of the indentation test only.
Order No.: JA709-030

Determination of equilibrium cations for the $KTiOPO_4$ structure
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Single crystals with the structure of $KTiOPO_4$ (KTP) were grown from mixtures containing equal concentrations of (i) $KSnOPO_4$ and $KGeOPO_4$, (ii) $KTiOPO_4$ and $KGeOPO_4$, and (iii) $KTiOPO_4$ and $KTiOAsO_4$, respectively. The comparison of the lattice parameters measured and calculated from Vegard's rule shows that structural stability of $KTiOAsO_4$ is higher in comparison with $KTiOPO_4$. It was found that the addition of GeO_2 to the KTP containing flux is accompanied by increasing all lattice parameters of KTP that correspond to substitution of As^{5+} by Ge^{4+}.
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Determination of elastic modulus of thin layers using nanoindentation
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Elastic modulus of thin homogeneous films can be determined by indenting the specimen to various depths, and extrapolating the measured (apparent) *E*-values to zero penetration The paper shows the application of five approximation functions for this purpose: linear, exponential, reciprocal exponential, Gao's, and Doerner and Nix's function. The comparison of the results for twenty-six film/substrate combinations has shown that the indentation response of film/substrate composites can, in general, be described by the Gao analytical function. In determining the thin film modulus from experimental data, satisfactory results can also be obtained with the exponential function, while linear function may be used only for thick films where the relative depths of penetration are small. The article explains the pertinent procedures and gives practical recommendations for the testing.
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Friction and wear performance of diamond-like carbon, boron carbide, and titanium carbide coatings against glass

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Protection of glass substrates by direct ion beam deposited diamond-like carbon (DLC) coatings was observed using a commercial pin-on-disk instrument at ambient conditions without lubrication. Ion beam sputter-deposited titanium carbide and boron carbide coatings reduced sliding friction, and provided tribological protection of silicon substrates, but the improvement factor was less than that found for DLC. Observations of unlubricated sliding of hemispherical glass pins at ambient conditions on uncoated glass and silicon substrates, and ion beam deposited coatings showed decreased wear in the order: uncoated glass > uncoated silicon > boron carbide > titanium carbide > DLC > uncoated sapphire. Failure mechanisms varied widely and are discussed. Generally, the amount of wear decreased as the sliding friction decreased, with the exception of uncoated sapphire substrates, for which the wear was low despite very high friction. There is clear evidence that DLC coatings continue to protect the underlying substrate long after the damage first penetrates through the coating. The test results correlate with field use data on commercial products which have shown that the DLC coatings provide substantial extension of the useful lifetime of glass and other substrates.

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Thermal stresses in carbon-coated optical fibers at low temperature

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The thermal stresses in carbon-coated optical fibers at low temperature have been analyzed. The thermally induced lateral pressure in the glass fiber would produce microbending loss. In order to minimize such a microbending loss, the thickness, Young's modulus and Poisson's ratio of the carbon coating should be decreased. On the other hand, the maximum thermal stress is the tangential stress in the carbon coating which occurs at the interface of the carbon coating and glass fiber. It was experimentally observed that if the maximum thermal stress is larger than the tensile strength of the carbon coating, the carbon coating will be broken along the axial direction. In order to minimize such a maximum thermal stress, the thickness of the carbon coating should be increased, but the Young's modulus, thermal expansion coefficient and Poisson's ratio of the carbon coating should be decreased. Finally, an optimal selection of the carbon coating for optical fiber is discussed.

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