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A.D. Westwood, R.A. Youngman, M.R. McCartney, A.N. Cormack, M.R. Notis

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**Oxygen Incorporation in Aluminum Nitride via Extended Defects.  
Part II: Structure of Curved Inversion Domain Boundaries and Defect  
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A.D. Westwood, R.A. Youngman, M.R. McCartney, A.N. Cormack,  
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## ABSTRACTS

### COMMUNICATIONS

**Influence of the Annealing Atmosphere on the Formation  
of Al-Cu-Fe Quasicrystals**

S-S. Kang, J-M. Dubois

(Laboratoire de Science et Génie des Matériaux Métalliques-  
CNRS URS 159)

Icosahedral samples of nominal composition Al<sub>63.5</sub>Cu<sub>24</sub>Fe<sub>12.5</sub> which normally grow a rhombohedral approximant crystal, were submitted to heat treatments of various duration. The atmosphere in contact with the surface of the specimens was either dry air or secondary vacuum in order to test whether the formation of the icosahedral phase is sensitive or not to the nature of the atmosphere. Materials annealed in air showed a very thin surface layer of oxide which contains hydrogen. Beneath this oxide layer, small quantities of oxygen and hydrogen were also detected within the underlying lattice. This contamination was sufficient to shift the composition of the samples out of the stability domain of the icosahedral phase and has induced formation of a significant amount of the cubic phase. In contrast, the rhombohedral compound no longer appeared. This behavior goes along with the great sensitivity of the icosahedral phase stability to a sharply defined electron to atom ratio.

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**Carbon in Diesel Particulate Matter: Structure, Microwave  
Absorption and Oxidation**

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(West Virginia University)

The structural, microwave absorption and oxidation characteristics of diesel particulate matter (DPM) collected from a CAT 3304 diesel engine are reported. The x-ray diffraction of DPM yields the characteristic peaks of pre-graphitic carbons (coke and pitches) and its modeling yields  $d_{(002)} \approx 3.429$  Å and crystallite size of about 20 Å. The real and imaginary parts of the dielectric constant  $\epsilon = \epsilon' + i\epsilon''$  are measured at 8.7 GHz using the cavity perturbation technique. The measured values for the DPM are  $\epsilon' = 8.6 \pm 1.7$  and  $\epsilon'' = 7.4 \pm 1.5$ , compared to  $\epsilon' \approx 1.0$  and  $\epsilon'' \approx 6 \times 10^{-5}$  for the ceramic trap material used for collecting DPM. The oxidation products of the DPM, analyzed by FTIR spectroscopy, are found to contain CO<sub>2</sub> and CO with a peak yield occurring around 500°C. Since microwave power absorption is proportional to  $\epsilon''$ , these results show that selective microwave heating of the DPM in the ceramic traps should be a very efficient process with CO<sub>2</sub> and CO as the main oxidation products.

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**Effect of Mn Valence on Crystal Structure of La-Mn-O Perovskite  
Oxides**

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(Kyoto University)

La:Mn=1:1 mixtures of lanthanum oxide and manganese carbonate were heat treated under various oxygen partial pressures at 1400°C or 1300°C. The Mn valence of the samples was measured by a chemical analysis and the crystal structures were refined by the powder x-ray diffraction and the Rietveld analysis. A novel orthorhombic perovskite phase, belonging to the space group *Pbnm* and containing Mn<sup>2+</sup> ions, was formed by the heat-treatment under low oxygen partial pressures. The structure was very close to a cubic symmetry. It is supposed that the micro Jahn-Teller effects of Mn<sup>3+</sup> ions were nearly cancelled by one another.

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**Low Temperature Synthesis of Thin Films of Carbon Nitride**

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(Indian Institute of Science)

Thin films of carbon nitride have been prepared using triode ion plating. It has been observed that the compound formation occurs at ambient substrate temperature itself. Though the films are completely amorphous at temperatures below 900°C, they are hard and transparent down to a wavelength of 200 nm. It has also been observed that the film transmission can be modulated using nitrogen ion flux and partial pressure. The IR transmission spectra clearly show the C-N stretch band around 2200 cm<sup>-1</sup>. Onset of crystallization, as evidenced from electron diffraction, occurs around 900°C substrate temperature.

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### ARTICLES

**Reduced Y-Stabilized ZrO<sub>2</sub> Deposition Temperatures  
for YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub>/YSZ Thin Films on Sapphire**

J.H. Kroese, A.J. Drehman, J.A. Horrigan

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Thin films of Y-Stabilized ZrO<sub>2</sub> (YSZ) were deposited by RF diode sputtering on R-plane sapphire as a buffer layer for the deposition of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> (YBCO). By increasing the partial pressure of oxygen in the sputter gas mixture from 20% to 50%, it was found that the substrate temperature required to obtain (100) oriented YSZ deposition could be lowered to 630°C from 800°C. This change is attributed to heating or mixing effects at the film surface, due to an increase in negative ion bombardment, which supplements the effects of external heating.

Increases in the partial pressure of oxygen beyond 50% were found to be counterproductive. YBCO films, deposited on the YSZ buffer layers via magnetron sputtering, showed c-axis orientation and transition temperatures of 82 K. Orientation of both the YSZ and YBCO films was confirmed by x-ray diffraction and SEM characterization.

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#### Oxygen Interaction with Ternary Chalcogenide: An ESCA Study of AgInTe<sub>2</sub>

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(National Chemical Laboratory)

X-ray photoelectron spectroscopic measurements of oxidation of AgInTe<sub>2</sub> surface at different temperatures are reported. The results are analyzed quantitatively. The oxidized surface was shown to have TeO<sub>2</sub>, Te and In<sub>2</sub>O<sub>3</sub>. Further, the presence of In<sub>2</sub>O is also identified. The experimental results are explained on the basis of heat of formation.

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#### Deposition of Epitaxial $\beta$ -SiC Films on Porous Si(100) from Methyltrichlorosilane in a Hot Wall LPCVD Reactor

C.C. Chiu, S.B. Desu, G. Chen, C.Y. Tsai, W.T. Reynolds, Jr.  
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Epitaxial  $\beta$ -SiC thin films were grown on modified Si(100) substrates from methyltrichlorosilane (MTS or CH<sub>3</sub>SiCl<sub>3</sub>) in a hot wall reactor by using low pressure chemical vapor deposition (LPCVD). At 1150°C, the growth rate of the  $\beta$ -SiC films was 120 Å/min. Epitaxial  $\beta$ -SiC(100) thin films were deposited after the deposition time of 12.5 min. However, the crystallinity of the deposited films was influenced by the deposition time. For example, the occurrence of rotational  $\beta$ -SiC(100) crystals and polycrystalline  $\beta$ -SiC with a highly preferred orientation of (100) planes was obtained for the deposition time of 50 min. XRD and TEM showed the appearance of polycrystalline  $\beta$ -SiC films with a preferred orientation of  $\beta$ -SiC(111) after further increasing the deposition times (time  $\geq$  75 min). At 1100°C, polycrystalline  $\beta$ -SiC films with poor surface morphology were observed even though the film had a preferred orientation of  $\beta$ -SiC(100) for short deposition time (e.g., 12.5 min). Polycrystalline  $\beta$ -SiC(111) film was obtained for the deposition time of 200 min at this temperature.

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#### Study of Diamond Growth from a Variety of Input Gases

K.L. Menningen, C.J. Erickson, M.A. Childs, L.W. Anderson, J.E. Lawler  
(University of Wisconsin-Madison)

The gas phase densities of CH<sub>3</sub> and CH and the hydrogen dissociation fraction are measured in a hot filament diamond deposition system for each of several different hydrocarbon input gases. The crystal growth rate and the appearance of the diamond grown from the different input gases are also examined. A comparison of the measurements indicates that the nature of the input hydrocarbon is relatively unimportant because fast gas phase reactions completely scramble the identities of the input carbon atoms. The addition of oxygen greatly alters the gas phase densities and other experimental factors such as the filament surface condition. Small concentrations of atomic impurities in the gas phase are also detected using high sensitivity absorption spectroscopy.

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#### Catalytic Properties of Nanocrystalline WO<sub>3-x</sub>, Pt/WO<sub>3-x</sub> and Pd/WO<sub>3-x</sub> Particles

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The gas-condensation technique is used to produce the nanocrystalline (NC) WO<sub>3-x</sub>, Pt/WO<sub>3-x</sub> and Pd/WO<sub>3-x</sub> powders under different atmosphere and pressure. HRTEM images show that coherently-bonded interface exists between Pt or Pd and WO<sub>3-x</sub>. The nanocrystal WO<sub>3-x</sub>, Pt/WO<sub>3-x</sub> and Pd/WO<sub>3-x</sub> grow into needle shape with plate inside when these as-evaporated powders are compacted and sintered

at 900°C for two hours. The plate grows preferentially in {220} plane along <001> direction. However, the mean particle size of nanophase Pt and Pd only increases from less than 10 nm to 30 nm and 50 nm, respectively. The results of CO oxidation show that nanophase Pt/WO<sub>3-x</sub> powders have better catalytic effects on the converting of CO to CO<sub>2</sub> than nanophase WO<sub>3-x</sub> and Pd/WO<sub>3-x</sub> powders.

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#### Synthesis of Low Density Carbon Crystal "Carbolite" by Quenching of Carbon Gas

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(Iwaki Meisei University)

A new carbon crystal named carbolite has been synthesized by quenching high-temperature carbon gas on a room temperature substrate. The crystal has a density as small as 1.46 g/cm<sup>3</sup> and is transparent for visible light. The crystal structure is hexagonal and the dimensions of the unit cell are 1.193 nm in the a-direction and 1.062 nm in the c-direction. Along the c-axis, a parallel array of carbon chains is proposed. The array is made of a triangular lattice in which the interchain distance is 0.344 nm and the mean interatomic distance within a carbon chain is 0.133 nm. The former value is close to the Van der Waals distance between the honeycomb planes of graphite and the latter value is close to the C-C bond length in the honeycomb plane. Intercalations of potassium, sodium, and iodine atoms are performed.

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#### Optimal Thickness for Si Interlayer as Diffusion Barrier at the Si<sub>3</sub>N<sub>4</sub>/GaAs Interface: A Transmission Electron Microscopy Study

T. Zheng, J.M. Gibson, D.S.L. Mui, H. Morkoç  
(University of Illinois at Urbana-Champaign)

Using transmission electron microscopy, we investigate Si<sub>3</sub>N<sub>4</sub> grown *in-situ* on GaAs metal-insulator-semiconductor (MIS) device structures with a Si interlayer, previously shown to improve the electrical properties of field-effect transistors with Si<sub>3</sub>N<sub>4</sub> gates on GaAs. We find that the primary role of the Si interlayer is to prevent the reaction between the nitride or nitrogen used for growth, and GaAs. The interlayer thickness dependence of this microstructure, and its relation to electrical properties, are discussed. The optimal thickness of the thin pseudomorphic Si interlayer appears to be around 0.4 nm. The growth temperature dependence of the critical thickness for morphological instability is demonstrated.

Order No.: JA505-011

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#### Analysis of the Diffusion Controlled Growth of Cobalt Silicides in Bulk and Thin Film Couples

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The solid state reaction between Co and Si has been studied in bulk diffusion couples between 850 and 1100°C. At the scale of the observations made, the three phases Co<sub>2</sub>Si, CoSi, and CoSi<sub>2</sub> are found to grow simultaneously, according to diffusion controlled kinetics. The results are analyzed in terms of the Nernst-Einstein equation that directly relates diffusion fluxes to the free energy changes driving the formation. The growth rates obtained for CoSi<sub>2</sub> at high temperatures, in the present bulk samples, are compared to those determined by others in thin films, at much lower temperatures. The comparison requires that attention should be paid to two factors. First is that the laws of growth are slightly different for a phase growing simultaneously with two other ones (bulk) and one phase growing alone (thin films). The second factor is that the grain size of the various samples varies with the temperature of reaction. Once this is done, excellent agreement is obtained between the two sets of measurements. Moreover, it is shown that by knowing the grain size, it is possible to calculate quite accurately the growth rate from the respective isotope diffusion coefficients, both for lattice and grain boundaries of Co and Si in CoSi<sub>2</sub>.

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### The Decagonal Quasicrystal Formed from an Al-Pd-Mn Icosahedral Quasicrystal

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(Tohoku University)

We present a detailed investigation on the decagonal quasicrystal (D-phase) formed from an Al-Pd-Mn icosahedral quasicrystal (I-phase) through a solid-state phase transformation, including its formation, compositional and crystallographical relationships with the matrix I-phase, growth mode and structural characteristics. The as-melt-spun  $\text{Al}_{70}\text{Pd}_{20}\text{Mn}_{10}$  alloy contains only I-phase. By annealing at  $800^\circ\text{C}$ , the D-phase is found to grow epitaxially from the I-phase to establish a D/I two-phase equilibrium with distinctly different compositions between them. The D-phase exhibits a stepped growth interface, which consists of a facet plane, formed by sharing the ten-fold plane with a five-fold plane of the matrix I-phase, and some ledges across it. The growth of the D-phase into the I-phase proceeds through lateral movement of the ledges along the ten-fold plane. High-resolution electron microscopy reveals that the structure of the D-phase is constructed by an aperiodic arrangement of decagonal atom clusters with definite linkages and long-range quasiperiodic correlation.

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### Analysis of Doped Rodlike Surface Fibers in Al-Al<sub>3</sub>Ni Eutectic Alloys

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Undoped and Cr doped rodlike Al-Al<sub>3</sub>Ni eutectics have been studied as initial alloys of Raney nickel catalysts. The aim was to localize the dopant at different stages of the catalyst life: in the initial alloy, after Al leaching out and after hydrogenation reaction. We have followed this dopant by STEM analysis and high resolution Auger spectroscopy. The outcome is that during Al leaching out, the dopant is also extracted from its initial phase and then forms a deposit on the Ni-crystallites of the catalyst. Moreover, during the hydrogenation, the quantity of Cr remains roughly constant.

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### Deformation and Fracture of a Directionally Solidified NiAl-28Cr-6Mo Eutectic Alloy

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(\*Shanghai Jiao Tong University, \*University of Tennessee, #NASA-Lewis Research Center)

A directionally solidified alloy based on the NiAl-(Cr,Mo) eutectic was examined by transmission and scanning electron microscopy to characterize the microstructure and room temperature deformation and fracture behavior. The microstructure consisted of a lamellar morphology with a  $\langle 111 \rangle$  growth direction for both the NiAl and (Cr,Mo) phases. The interphase boundary between the eutectic phases was semicoherent and composed of a well defined dislocation network. In addition, a fine array of coherent NiAl precipitates was dispersed throughout the (Cr,Mo) phase. The eutectic morphology was stable at 1300 K with only coarsening of the NiAl precipitates occurring after heat treatment for 1.8 ks (500 h). Fracture of the aligned eutectic is characterized primarily by a crack bridging/nucleation mechanism and is controlled by the strength of the semicoherent interface between the two phases. However, contributions to the toughness of the eutectic may arise from plastic deformation of the NiAl phase and the geometry associated with the fracture process.

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### Elevated Temperature Creep Properties of NiAl Cryomilled with and without Y<sub>2</sub>O<sub>3</sub>

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The creep properties of lots of NiAl cryomilled with and without Y<sub>2</sub>O<sub>3</sub> have been determined in compression and tension. Although identical cryomilling procedures were used, differences in composition were found between the lot ground with 0.5 vol.% yttria and the lot ground without Y<sub>2</sub>O<sub>3</sub>. Compression testing between 1000 to 1300 K

yielded similar creep strengths for both materials, while tensile creep rupture testing indicated that the yttria-containing alloy was slightly stronger than the Y<sub>2</sub>O<sub>3</sub>-free version. Both compression and tensile testing showed two deformation regimes; whereas the stress state did not affect the high stress exponent ( $n \approx 10$ ) mechanism, the low stress exponent regimes  $n$  was  $\sim 6$  in tension and  $\sim 2$  in compression. The strengths in tension were somewhat less than those measured in compression, but the estimated activation energies ( $Q$ ) of  $\sim 600$  kJ/mol for tensile testing were closer to the previously measured values ( $\sim 700$  kJ/mol) for NiAl-AlN and very different from the  $Q$ 's of 400 and 200 kJ/mol for compression tests in the high and low stress exponent regimes, respectively. A Larson-Miller comparison indicated that cryomilling can produce an alloy with long term, high-temperature strength at least equal to conventional superalloys.

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### Elastic Constants of Single Crystal $\gamma$ -TiAl

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The six independent second-order elastic stiffness coefficients of a  $\text{Ti}_{44}\text{Al}_{56}$  single crystal ( $L1_0$  structure) have been measured at room temperature for the first time using a resonant ultrasonic spectroscopy (RUS) technique. These data were used to calculate the orientation dependence of the Young's modulus and the shear modulus. The Young's modulus is found to reach a maximum near  $[111]$  direction, close to the normal to the most densely-packed planes. The elastic moduli and the Poisson's ratio for polycrystalline materials, calculated by the averaging scheme proposed by Hill, are in good agreement with experimental data and theoretical calculations.

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### Preparation of Stages 2-4 Ternary AlCl<sub>3</sub>-FeCl<sub>3</sub>-Graphite Intercalation Compounds

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(Kyoto University)

Intercalation of AlCl<sub>3</sub> into stage 2 FeCl<sub>3</sub>-graphite intercalation compound (GIC) using an ordinary two-bulb method has been studied by x-ray diffraction. Stages 2, 3, and 4 ternary AlCl<sub>3</sub>-FeCl<sub>3</sub>-GIC's are obtained when the temperatures of the stage 2 FeCl<sub>3</sub>-GIC were set at  $T(\text{GIC}) = 503, 523, \text{ and } 553$  K, respectively, for the AlCl<sub>3</sub> intercalate material at  $T(\text{AlCl}_3) = 473$  K, that is, the vapor pressure of  $(\text{AlCl}_3)_2(\text{g})$  of the main vapor species to be held at  $p((\text{AlCl}_3)_2) = 2.4 \times 10^5$  Pa. However, for the temperature of the stage 2 FeCl<sub>3</sub>-GIC at  $T(\text{GIC}) = 573$  K, the  $(\text{AlCl}_3)_2(\text{g})$  vapor is found to promote the decomposition of the stage 2 FeCl<sub>3</sub>-GIC, resulting in the formation of graphite. The decomposition of the stage 2 FeCl<sub>3</sub>-GIC is considered to take place because the complex  $\text{AlFeCl}_5(\text{g})$  in the gas phase, which is formed from both  $(\text{AlCl}_3)_2(\text{g})$  and FeCl<sub>3</sub> existing at the edge of the FeCl<sub>3</sub>-GIC, is thermodynamically more stable than the FeCl<sub>3</sub> and AlCl<sub>3</sub> intercalates in their GIC at  $p((\text{AlCl}_3)_2) = 2.4 \times 10^5$  Pa and  $T(\text{GIC}) = 573$  K.

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### Elastoplastic Deformation of Multi-Layered Materials During Thermal Cycling

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(Massachusetts Institute of Technology)

Analytical models are presented for the elastoplastic deformation of multilayered materials subjected to fluctuating temperatures. The layered structure comprises an elastic-perfectly plastic ductile material sandwiched between two elastic brittle materials. With creep, heat transfer and edge effects excluded, closed-form solutions for different characteristic temperatures for thermal cycling are presented as a function of the layer geometries and the thermal-mechanical properties of the constituent phases. The evolution of curvature, the generation of thermal residual stresses within each layer, and the onset and spread of plasticity in the ductile layer are also examined. It is theoretically shown that reversals of curvature in the layered solid can occur during monotonic changes in temperature, even when the thermomechanical

properties of the layer do not vary significantly with temperature. The predictions of the analytical model are seen to compare favorably with experimental observations of curvature during thermal cycling in the limiting case of a bi-layer composite with Al-Al<sub>2</sub>O<sub>3</sub> layers and Al-Si layers, and in a Si-Al-SiO<sub>2</sub> tri-layer system. Case studies of the effects of the relative variations in the geometry, elastic properties, and plastic response of the constituent phases on the overall deformation are examined for two practically significant layered systems: a Si-Al-SiO<sub>2</sub> layered solid with extensive applications in the electronics industry and a Cr<sub>2</sub>O<sub>3</sub>-coated steel with an interlayer of a Ni-Al alloy which is used in structural applications.

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#### Transmission Electron Microscopy Study of Al/Al<sub>2</sub>O<sub>3</sub> Composites Fabricated by Reactive Metal Infiltration

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The microstructure of Al/α-Al<sub>2</sub>O<sub>3</sub> composites made by infiltrating molten Al into dense mullite preforms has been characterized using transmission electron microscopy. The growth of the Al/Al<sub>2</sub>O<sub>3</sub> composites was found to proceed through three stages. Initially, Al infiltrates into a dense mullite preform through grain boundary diffusion, and reacts with mullite at grain boundaries to form a partial reaction zone. Then, a complete reaction takes place in the reaction region between the partial reaction zone and the full reaction zone to convert the dense mullite preform to a composite of α-Al<sub>2</sub>O<sub>3</sub> (matrix) and an Al-Si phase (thin channels). Finally, the reduced Si from the reaction diffuses out of the Al/Al<sub>2</sub>O<sub>3</sub> composite through the metal channels, whereas Al from the molten Al pool is continuously drawn to the reaction region until the mullite preform is consumed or the sample is removed from the molten Al pool. Based on the observed microstructure, infiltration mechanisms have been discussed, and a growth model of the composites is proposed in which the process involves repeated nucleation of Al<sub>2</sub>O<sub>3</sub> grains and grain growth.

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#### Combustion Synthesis of MoSi<sub>2</sub>-TiC Composites

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Stability of the binary phases in the quaternary system Mo-Ti-Si-C was examined for the combustion synthesis or self-propagating high-temperature synthesis (SHS) of MoSi<sub>2</sub>-TiC composites. Thermochemical calculations were carried out to obtain the adiabatic temperatures and the amounts of molten phases at the adiabatic temperature for the SHS of these composites. MoSi<sub>2</sub>-20 wt% TiC and MoSi<sub>2</sub>-40 wt% TiC composites were prepared by thermal explosion and SHS modes of combustion from elemental powders. The phase formation was confirmed by x-ray diffraction and the morphologies were observed under SEM. No general correlation was obtained between the phase formation or product morphology on the one hand, and the adiabatic temperature or the amount of molten phase formed at the adiabatic temperature on the other. MoSi<sub>2</sub>-40 wt% TiC composites obtained by SHS mode of combustion show a highly homogeneous distribution of the two phases with negligible amounts of ternary phases.

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#### Dynamic Modeling of the Interaction of Gas and Solid Phases in Multi-Step Reactive Micropyretic Synthesis

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A mathematical model of micropyretic synthesis including the consideration of pressure rise (due to gas evolution) in a porous compact is developed for a multistep reaction. The D'Arcy's law of gas flow, continuity equation and gas law are combined to obtain a relationship between the pressure and temperature of gas. This equation for the gas pressure is solved along with the energy equations of gas and

solid phase. The numerical analysis shows that the magnitude of pressure increase depends on the initial gas pressure, temperature and permeability. When gas evolution is considered, the pressure increase depends on the variables which determine the kinetics of the gas evolution reaction, such as, the activation energy and the pre-exponential factor. The pressure increase is maximum when the gas evolution takes place in the combustion reaction zone. The gas evolution is noted not to influence the combustion wave propagation.

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#### Formation and Characteristics of Nanocrystalline Composites γ-TiAl+Ti<sub>2</sub>AlN by Mechanical Alloying and Subsequent Annealing Treatment

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Formation of nanocrystalline composites γ-TiAl+Ti<sub>2</sub>AlN by mechanically alloying Ti<sub>50</sub>Al<sub>50</sub> in a N<sub>2</sub> atmosphere and subsequent annealing treatment are investigated. The development of the microstructure was monitored by x-ray diffraction, differential thermal and transmission electron microscopy. The amorphous phase could be obtained after milling for 30 h in a nitrogen atmosphere. The TEM results show that some nanocrystalline solid solution of Al in Ti also existed in the amorphous matrix. The result of annealing treatments at different temperatures for 0.5 h on the amorphous phase obtained by MA in N<sub>2</sub> gas for 30 h shows the formation of γ-phase (TiAl) and a nitride of titanium and aluminum (Ti<sub>2</sub>AlN). Available annealing treatments could produce nanocrystalline composites of γ-TiAl and Ti<sub>2</sub>AlN with grain size less than 20 nm. With increasing annealing temperature, the crystalline sizes of γ-TiAl and Ti<sub>2</sub>AlN increase but the values of microhardness increase slightly.

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#### The Low Temperature Hexagonal to Orthorhombic Transformation in Si<sub>3</sub>N<sub>4</sub> Reinforced BAS Matrix Composites

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The amount of Si<sub>3</sub>N<sub>4</sub> in a Si<sub>3</sub>N<sub>4</sub>-BaO·Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub> (BAS) composite and the time of sintering was examined in the context of the low temperature hexagonal to orthorhombic transformation in BAS. It was found that with increasing amounts of Si<sub>3</sub>N<sub>4</sub> in the composite the temperature of the hexagonal to orthorhombic transformation was decreased. As the sintering time was increased for a given composite composition, a drop in the temperature of the hexagonal to orthorhombic transformation was observed which was linked to an increase in the β-Si<sub>3</sub>N<sub>4</sub> content in the composite. In addition, as the Si<sub>3</sub>N<sub>4</sub> content in the composite was increased, a resultant drop in the coefficient of thermal expansion of the composite occurred. The extent of the linear dimensional change of the composite during the BAS hexagonal to orthorhombic transformation is reported.

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#### TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> Nanocomposites

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Nano-sized TiO<sub>2</sub> is synthesized in a nano-sized alumina matrix by a sol-gel procedure. The results of the study of the blue shift of the TiO<sub>2</sub> UV band edge is compared to the information obtained from transmission electron microscopy (TEM). As long as the atomic ratio Ti/Al remains smaller than 17.5%, no individualized TiO<sub>2</sub> particles are detected by TEM, in spite of the fact that no modification of the alumina structure occurs, as revealed by <sup>27</sup>Al MAS NMR. The shift of the UV band edge suggests the growth of homogeneous TiO<sub>2</sub> domains in the alumina matrix. Above a critical radius, on the order of 1.2 nm, individual and crystalline TiO<sub>2</sub> particles become visible in the TEM picture and the porosity of the material decreases markedly.

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### Oxygen Incorporation in Aluminum Nitride via Extended Defects. Part I: Refinement of the Structural Model for the Planar Inversion Domain Boundary

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The model proposed by Harris, et al. describing planar inversion domain boundaries in aluminum nitride, consists of a basal plane of aluminum atoms octahedrally coordinated with respect to oxygen, and with a translation of  $R = 1/3\langle 10\bar{1}0 \rangle + 1/3\langle 0001 \rangle$  or  $1/3\langle 10\bar{1}1 \rangle$ . This thin sandwich is inserted onto the basal plane of the wurtzite structure of aluminum nitride. This model does not take into consideration any interfacial relaxation phenomena, and is arguably electrically unstable. Therefore, this paper presents a refinement of the model of Harris, et al., by incorporating the structural relaxations arising from modifications in local chemistry. The interfacial structure was investigated through the use of conventional transmission electron microscopy, convergent electron diffraction, high-resolution transmission electron microscopy, analytical electron microscopy and atomistic computer simulations.

The refined planar inversion domain boundary model is closely based on the original model of Harris, et al.; however, the local chemistry is changed, with every fourth oxygen being replaced by a nitrogen. Atomistic computer simulation of these defects, using a classical Born model of ionic solids, verified the stability of these defects as arising from the adjustment in the local chemistry. The resulting structural relaxations take the form of a 0.3 mrad twist parallel to the interface, a contraction of the basal planes adjacent to the planar inversion domain boundary, and an expansion of the c-axis component of the displacement vector; the new displacement vector across the interface is  $R = 1/3\langle 10\bar{1}0 \rangle + \epsilon\langle 0001 \rangle$ , where  $\epsilon_{meas} = 0.387$  and  $\epsilon_{calc} = 0.394$ .

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### Oxygen Incorporation in Aluminum Nitride via Extended Defects. Part II: Structure of Curved Inversion Domain Boundaries and Defect Formation

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Three distinct morphologies of curved (curved, faceted and corrugated) inversion domain boundaries (IDBs), observed in aluminum nitride, have been investigated using conventional transmission electron microscopy, convergent beam electron diffraction, high-resolution transmission electron microscopy, analytical electron microscopy, and atomistic computer simulations. The interfacial structure and chemistry of the curved and faceted defects have been studied, and based upon the experimental evidence, a single model has been proposed for the curved IDB which is consistent with all three observed morphologies. The interface model comprises a continuous nitrogen sublattice, with the aluminum sublattice being displaced across a  $\{10\bar{1}1\}$  plane, and having a displacement vector  $R = 0.23\langle 0001 \rangle$ . This displacement translates the aluminum sublattice from upwardly pointing to downwardly pointing tetrahedral sites, or vice-versa, in the wurtzite structure. The measured value of the displacement vector is between  $0.05\langle 0001 \rangle$  and  $0.43\langle 0001 \rangle$ ; the variation is believed to be due to local changes in chemistry. This is supported by atomistic calculations which indicate that the interface is most stable when both aluminum vacancies and oxygen ions are present at the interface, and that the interface energy is independent of displacement vector in the range of  $0.05\langle 0001 \rangle$  to  $0.30\langle 0001 \rangle$ . The curved IDBs form as a result of non-stoichiometry within the crystal. The choice of curved IDB morphology is believed to be controlled by local changes in chemistry, non-stoichiometry at the interface, and proximity to other planar IDBs (the last reason is explained in paper III). A number of possible formation mechanisms are discussed for both planar and curved IDBs.

The Burgers vector for the dislocation present at the intersection of the planar and curved IDBs was determined to be  $b = 1/3\langle 10\bar{1}0 \rangle + t\langle 0001 \rangle$ , where  $t_{meas} = 0.157$  and  $t_{calc} = 0.164$ .

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### Investigation of Tetragonal Distortion in the PbTiO<sub>3</sub>-BiFeO<sub>3</sub> System by High-Temperature X-ray Diffraction

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Compositions in the  $(\text{Pb}_{1-x}\text{Bi}_x)(\text{Ti}_{1-x}\text{Fe}_x)\text{O}_3$  solid solution system for  $x \leq 0.7$  show unusually large tetragonal distortion. High-temperature x-ray diffraction was used to study the tetragonal distortion as a function of temperature (25–700°C) for compositions ( $x = 0-0.7$ ) using powders prepared by solid state reaction in the above system. Large changes in the lattice parameters were observed over a narrow temperature range near  $T_C$  for compositions near the morphotropic phase boundary (MPB) ( $x = 0.7$ ). Compositions near MPB showed a c/a ratio of 1.18 at room temperature. Polar plots of lattice constants at different temperatures indicated strong anisotropic thermal expansion with zero thermal expansion along [201] direction.

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### The Deposition of $\alpha\text{-Fe}_2\text{O}_3$ by Aerosol Chemical Vapor Deposition

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$\alpha\text{-Fe}_2\text{O}_3$  thin films have been deposited on silicon (111) substrates at high temperatures (600–800°C) by the spray pyrolysis method. Four different iron (III)  $\beta$ -diketonates have been used as precursors in order to obtain polycrystalline films of good adherence, which have been characterized by x-ray diffraction, scanning electron microscopy and magnetic measurements.

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### Devitrification Inhibitors in Borosilicate Glass and Binary Borosilicate Glass Composite

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Cristobalite is known to precipitate out of borosilicate glass (Corning 7740), and a binary glass mixture of borosilicate glass and high silica glass when these glasses are heated to elevated temperatures. To prevent cristobalite from forming in these glass systems, a devitrification inhibitor needs to be found. Among oxides selected for testing, both  $\text{Al}_2\text{O}_3$  and  $\text{Ga}_2\text{O}_3$  are found to prevent cristobalite from forming in these glass systems.

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### Densification Kinetics of Glass Films Constrained on Rigid Substrates

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The kinetics of constrained-film sintering were studied in a borosilicate glass (BSG)+silica system because of its applications in micro-electronic packaging technologies. Samples with silica content of 20% by volume were prepared from slurries of powder mixtures in a commercial polyvinyl butyral (PVB) binder solution. Constrained films about 0.2 mm thick were formed by doctor-blade casting the slurries on silicon wafers. Free standing films about 0.6 mm thick were also produced by casting the slurries on a treated mylar sheet for easy lift-off. Sintering experiments were carried out in a hot stage at temperatures between 715°C and 775°C. Shrinkage profiles of the free and constrained (shrinkage in thickness only) films were determined *in situ* using a custom-designed optical system. The densification rates measured in the constrained films were slower than those in the free films. However, the substrate constraint had no effect on the activation energy

of densification which was found equal to  $385 \pm 10$  kJ/mol, the same for both free and constrained films. A relation between the constrained-film and free-film densification profiles was derived using the viscous analogy for the constitutive equations of a porous sintering body.

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#### Coating of Uniform Inorganic Particles with Polymers.

##### Part III: Polypyrrole on Different Metal Oxides

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(Clarkson University)

Five kinds of uniform metal oxide particles ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, CeO<sub>2</sub>, CuO, NiO, and SiO<sub>2</sub>) were coated with polypyrrole by reacting the dispersed solids with pyrrole in a water/ethanol medium without the use of a soluble oxidant. When the process was carried out in air, all particles were coated with the polymer, although the thickness of the layer varied on different cores. In CuO dispersions, independent polypyrrole particles were produced in addition to coated spheres. While oxygen is the major oxidant that initiates the polymerization of pyrrole, some metal oxides may also affect the reaction both in terms of the amount and the composition of the shell. Thus,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> were found to be inactive in the polymerization, while CeO<sub>2</sub> and CuO react with the adsorbed pyrrole molecules through a reductive-dissolution process, in which the monomers are oxidized causing a release of reduced metal ions.

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