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## ABSTRACTS

## SPECIAL SECTION

## MATERIALS AND THE ENVIRONMENT: TOWARDS SUSTAINABLE DEVELOPMENT

**Characterization and cation exchange properties of zeolite synthesized from fly ashes**W. Ma, P.W. Brown, S. Komarneni  
(The Pennsylvania State University)

Zeolite P was formed from Class F fly ash under hydrothermal conditions. The fly ash was reacted with 2.8 or 5 M NaOH at 100°C. Zeolite of the above type was produced regardless of the NaOH concentration or the addition of quartz thus indicating its formation over a wide range of conditions. The zeolite produced was of the NaP-type with a fibrous morphology. Analysis of the pore structure of this zeolite by the BET method indicates a Type II isotherm. The specific surface areas of synthesized zeolites increased from 28.5 to 41.1 m<sup>2</sup>/g when the NaOH concentration was increased from 2.8 to 5.0 M. This zeolite showed a significant selectivity for the uptake of Cs and Sr. Cs uptake ranged from 7.67 to 8.61 meq/100g while that of Sr ranged from 9.8 to 10.54 meq/100g. The value obtained depended on the specific synthesis conditions. These values are higher than those observed for tobermorite prepared from the same fly ash even though the tobermorite exhibited a higher specific surface area.

Order No.: JA801-001

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**The effect of mixed modifiers on nuclear waste glass processing, leaching and Raman spectra**S.V. Raman  
(Idaho National Engineering Laboratory)

Borosilicate glasses with different waste loadings were prepared by ambient melting, quenching and annealing. Some melt compositions partially crystallize to durable phases of zircon and forsterite. The coexisting liquid quenches to glass and endures the leach tests. The waste loading dependent leach rate trends of these glasses are reminiscent of mixed alkali effect. Raman spectra suggest initial increase in durability with increases in the depolymerization of silicate species. Fluorine and hydroxyl ions also contribute to depolymerization. Tetraborate and metaborate rings are identified in the Raman spectra. The durability is enhanced when tetraborate bands are more intense than the metaborate bands.

Order No.: JA801-002

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**Characterization of a new Ca-Cd hydroxide hydrothermally synthesized and its implications for cement isolation of Cd**S. Goñi, A. Maclás, J. Madrid, J.M. Díez  
(CSIC)

Mixtures of CaO-CdO (1:1) were hydrothermally treated in a pressure reactor at 200°C and 200 psi of pressure during a period of 16 hours. The evolution of the reaction was followed by x-ray diffraction (XRD), infrared spectroscopy (IR) and thermogravimetric (TG and DTG) analysis. In addition the composition of the filtered solutions was analyzed to determine the

mechanism of the reaction as well as the thermodynamic solubility constant of the new compound formed. The results show that CaO and CdO react giving rise to a new CaCd(OH)<sub>4</sub> hydroxide whose thermodynamic solubility constant,  $15 \pm 0.4 \times 10^{-11} \text{ M}^2$ , is six orders of magnitude lower than those of both Ca(OH)<sub>2</sub> and β-Cd(OH)<sub>2</sub>. This low solubility constant justifies the Cd<sup>2+</sup> concentration measured in the pore solution of cement matrices used to immobilize cadmium containing wastes. The mechanism of the reaction proposed is via dissolution of both Ca(OH)<sub>2</sub> and β-Cd(OH)<sub>2</sub> being Ca<sup>2+</sup> and Cd(OH)<sub>4</sub><sup>2-</sup> the predominant species in solution.

Order No.: JA801-003

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**A <sup>133</sup>Cs magic angle spinning nuclear magnetic resonance study of cesium environments in barium hollandites and Synroc**J.S. Hartman\*, E.R. Vance\*, W.P. Power#, J.V. Hanna§  
(\*Brock University, \*Australian Nuclear Science and Technology Organization, #University of Waterloo, §CSIRO-North Ryde NMR Facility)

Barium aluminium hollandite is a major phase in Synroc, a ceramic designed for the immobilization of high-level waste (HLW) from nuclear fuel reprocessing. Radioactive cesium substitutes into the channel sites, and such hollandites give <sup>133</sup>Cs MAS nmr spectra consisting of a single peak at 211 ppm in the absence of paramagnetic ions. However the peak shifts to 640 ± 30 ppm and becomes extremely broad when Ti<sup>3+</sup> replaces Al<sup>3+</sup> in the channel walls of the hollandite structure, apparently because of Fermi contact interaction between the Cs nucleus and the unpaired electron of Ti<sup>3+</sup>. <sup>133</sup>Cs MAS nmr of Synroc and hollandites is very sensitive to the presence of water-soluble CsAlTiO<sub>4</sub> which would compromise the aqueous durability of Synroc. <sup>133</sup>Cs MAS nmr spectra of Synroc-C, hot-pressed in metal bellows at temperatures as high as 1325°C, do not indicate significant formation of CsAlTiO<sub>4</sub>. Synroc samples loaded with Cs and Sr only were shown by MAS nmr as well as electron microscopic techniques to be capable of incorporating nearly 10.wt% Cs before CsAlTiO<sub>4</sub> is formed.

Order No.: JA801-004

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**An analytical electron microscopy investigation of municipal solid waste incineration bottom ash**J.E. Krzanowski\*, T.T. Eighmy\*, B.J. Crannell\*, J.D. Eusden Jr.\*  
(\*University of New Hampshire, \*Bates College)

Incinerator bottom ash samples have been characterized using analytical electron microscopy (AEM) techniques, including electron diffraction, energy dispersive spectroscopy and electron energy loss spectroscopy. The samples were first separated by magnetic properties and density. Three resulting fractions were examined: the magnetic, high-density (MHD) fraction, the non-magnetic/high-density (NMHD) fraction and the non-magnetic, low-density (NMLD) fraction. Examination of these samples revealed a variety of sub-micron microstructural features. For the MHD fraction, metal oxides, iron silicates, aluminum silicates and calcium phosphate compounds were found in addition to amorphous material. The NMHD fraction contained elements similar to the MHD fraction but had

more amorphous material; crystalline silicates were less common. Compounds such as MgO and chloroapatite were also found. The NMLD fraction contained SiO<sub>2</sub> and numerous metal oxides. The result of some of these analyses were used to model leaching behavior of the ash. Based on the AEM results, three mineral phases were chosen as candidates for aqueous geochemical thermodynamic equilibrium modeling of pH-dependent leaching: chromite, chloroapatite and zincite. In two of these three cases (chromite, chloroapatite), the selected mineral phase provided excellent agreement with the experimentally observed leaching behavior. AEM was shown to be a useful tool for elucidating mineralogy of complex environmental samples.

Order No.: JA801-005

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#### Morphology of wetting reactions of SnPb alloys on Cu as a function of alloy composition

C.Y. Liu, K.N. Tu  
(University of California at Los Angeles)

We have investigated the wetting angle, side band growth, and intermetallic compound formation of seven SnPb alloys on Cu ranging from pure Sn to pure Pb. The wetting angle has a minimum near the middle composition and increases towards pure Sn and pure Pb, but the side band growth has a maximum near the middle composition. The intermetallic compounds formed are Cu<sub>6</sub>Sn<sub>5</sub> and Cu<sub>3</sub>Sn for the eutectic and high-Sn alloys, yet for the high-Pb alloys, only Cu<sub>3</sub>Sn can be detected. While no intermetallic compound forms between Cu and pure Pb, the latter nevertheless wets the former with an angle of 115°. The driving force of a wetting reaction, which may be affected by the free energy gain in compound formation, is discussed by assuming that rate of compound formation is fast.

Order No.: JA801-006

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#### VHF a-Si:H solar cells: A systematic material and cell study

W.G.J.H.M. van Sark, J. Bezemer, W.F. van der Weg  
(Utrecht University)

A systematic study of material quality has been performed for intrinsic a-Si:H layers deposited by plasma enhanced chemical vapor deposition at excitation frequencies between 30 and 80 MHz (VHF). The process conditions were optimized not only for device quality optoelectronic properties but also for a uniformity in layer thickness better than 5% over the 10 cm x 10 cm substrate area. We found optimized homogeneities at different pressures depending on the excitation frequency. The effect of frequency at these optimum conditions on the material quality is small. VHF-intrinsic layers have been used in a Si:H p<sup>+</sup>-i-n<sup>+</sup> solar cells, in which both the p<sup>+</sup>- and n<sup>+</sup>-layer were made using 13.56 MHz. There is a clear correlation between material quality and solar cell parameters. Material deposited at low power densities is of so-called "device quality," which is confirmed by demonstrating an initial efficiency of 10% for cells deposited at 65 MHz using a low power density. The depositin rate still is 2–3 times higher than the one at 13.56 MHz. Light-soaking of the cell leads to stabilization at 6% for the best cells, which compares well to conventional 13.56 MHz cells.

Order No.: JA801-007

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#### A novel approach to paint sludge recycling: Reclaiming of paint sludge components as ceramic composites and their applications in reinforcement of metals and polymers

S. Nakouzi, D. Mielewski, J. Ball, B.R. Kim, I.T. Salmeeen, D. Bauer, C.K. Narula  
(Ford Motor Company)

About 15 x 10<sup>6</sup> lbs. of paint sludge, produced every year at Ford plants, is disposed of in landfills. An economical alternative to this disposal method, that reclaims or recycles components of paint sludge, is highly desirable to preserve valuable natural resources. Here, we describe an alternative to landfill disposal whereby paint sludge is converted into ceramic composites that can be used as reinforcing materials. The conversion of paint sludge to ceramic composite, 1/N<sub>2</sub>/600 is achieved by pyrolysis under a nitrogen atmosphere. Two additional composites, labeled 1/N<sub>2</sub>/1000 and 1/NH<sub>3</sub>/1000, respectively, are prepared by sintering 1/600 at

1000°C under N<sub>2</sub> and NH<sub>3</sub>. All three composites contain crystalline CaTiO<sub>3</sub>, BaTiO<sub>3</sub>, TiO<sub>2</sub>, amorphous alumina and carbon. 1/NH<sub>3</sub>/1000, contains an additional crystalline phase of titanium nitride. The application of these composites as reinforcing materials is demonstrated in the fabrication of representative metal matrix composites (MMCs) and reinforced plastic components.

Order No.: JA801-008

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#### Role of rapid photothermal processing in environmentally conscious semiconductor manufacturing

R. Sharangpani, R. Singh  
(Clemson University)

Compliance with the increasingly stringent environmental laws will force the phasing out of conventional methods for the manufacture of all semiconductor devices. Development of processes with the highest possible material and energy efficiencies is the heart of the present ecological preservation efforts. Besides other benefits, rapid isothermal processing (RIP) also uses less energy than furnace processing. Further increase of material and energy efficiencies is possible by using rapid photothermal processing. Central to this method is the use of quantum photoeffects in conventional RIP systems. This approach can be consolidated with advanced chemical vapor deposition methods that further lower the wastages leading to systems that can give significant improvements over those presently used. Key experimental results and possible future directions are presented.

Order No.: JA801-009

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#### Alternative reagent to mercuric nitrate catalyst for dissolution of aluminum-clad nuclear fuels in nitric acid

P.A. Anderson, J.D. Christian  
(Lockheed Martin Idaho Technologies Company)

A substitute additive, HBF<sub>4</sub>, has been discovered that will replace Hg(NO<sub>3</sub>)<sub>2</sub> catalyst for dissolving aluminum spent nuclear fuels in nitric acid for recovery of useable materials. The catalyst or substitute is necessary to penetrate a protective oxide film that continuously forms on the Al surface in the oxidizing acid. A penetration rate of alloy Al-6061 T6 of 40 mg/cm<sup>2</sup>·h can be achieved in a continuous dissolution process at 100°C using a dissolvent of 0.15 M HBF<sub>4</sub> in 7 M HNO<sub>3</sub> that achieves a steady-state composition of 1.0 M Al<sup>3+</sup> and 3.5 M HNO<sub>3</sub>, while maintaining a corrosion rate of a Type 304L stainless steel dissolver vessel of 0.015 mm/mo. The penetration rate of aluminum is correlated with the equilibrium concentration of HF in the system. The postulated mechanism involves dissolution of the alumina film by approximately 0.006 M HF in equilibrium with the HBF<sub>4</sub> and complexed aluminum fluoride species in nitric acid, which provide a large semi-buffered supply of HF. This allows the HNO<sub>3</sub> to attack the aluminum metal. The small concentration of HF does not compete favorably with HNO<sub>3</sub> for reaction with and consumption by Al.

Order No.: JA801-010

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#### Polystyrene foams with dispersed catalyst for a design of recyclable plastics

T. Hirose\*, Y. Takai\*, N. Azuma\*, Y. Morioka\*, A. Ueno\*, M. Okada\*  
(\*Shizuoka University, \*Furukawa Electric Co.)

The present authors have already reported that barium oxide is the most effective catalyst for the conversion of waste polystyrene into styrene including both monomer and dimer. Around 85 wt.% of waste polystyrene was converted into styrene when it was degraded at 623 K in the presence of barium oxide as catalyst.

In this paper, the preparation and the thermal degradation of polystyrene films with dispersed catalyst are described for a design of recyclable plastics for better environments. More than 85 wt.% of the films was degraded into styrene at 623 K without addition of other catalytic compounds. Techniques for foaming the films using either nitrogen or carbon dioxide are discussed. It was found that the polystyrene foams with dispersed catalyst are well converted into styrene when they are simply degraded at 673 K.

Order No.: JA801-011

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**Evaluation of high resistance to environmental corrosion for excellent car coating treatment**K. Mori, T. Okada, K. Oguri, K. Sakamoto, Y. Nishi  
(Tokai University)

Using scanning laser microscope and mercury porosimeter, we evaluate effects of an excellent car coating treatment on resistance to environmental corrosion after aging for three years. The treatment maintains the clear surface. The effect can mainly be explained by the contact angle of sessile drops.

Order No.: JA801-012

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**COMMUNICATIONS****Influence of initial crystal structure and electrical pulsing on densification of nanocrystalline alumina powder**R.S. Mishra, S.H. Risbud, A.K. Mukherjee  
(University of California at Davis)

The effect of the crystal structure of starting alumina powder and electric pulsing on the initial stages of densification has been studied in the temperature range of 1200–1500°C. Multiple electric pulsing cycles enhance the densification significantly. The  $\alpha$ -alumina powders consolidate more readily in comparison to  $\gamma$ -alumina powders. A high density  $\alpha$ -alumina specimen (>98 % of theoretical density) was obtained at 1300°C in less than 10 minutes.

Order No.: JA801-013

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**Improvement of the morphological stability of Ag film on (001)Si with a thin interposing Au layer**Y.C. Peng, C.R. Chen, L.J. Chen  
(National Tsing Hua University)

The morphological stability of ultrahigh vacuum deposited Ag film on (001)Si has been drastically improved by the deposition of 1- to 5-nm thick interposing Au layers. In Ag/(001)Si samples, Ag islands were found to form after annealing at 200°C for 1 h. In contrast, continuous and uniform metal layer persisted after annealing at 500°C for 1 h in samples with a thin intermediate Au layer. The significantly improved morphological stability is attributed to the strong intermixing between Au and Ag atoms at the metal/Si interface. The intermixing lowers the interface energy between metal layer and (001)Si.

Order No.: JA801-014

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**REVIEW****Processing and properties of hydroxyapatite-based biomaterials for use as hard tissue replacement implants**W. Suchanek, M. Yoshimura  
(Tokyo Institute of Technology)

This paper reviews past, present and future of the hydroxyapatite (HAp)-based biomaterials from the point of view of preparation of hard tissue replacement implants. Properties of the hard tissues are also described. The mechanical reliability of the pure HAp ceramics is low, therefore it cannot be used as artificial teeth or bones. For these reasons, various HAp-based composites have been fabricated, but only the HAp-coated titanium alloys have found wide application. Among the others, the microstructurally controlled HAp ceramics such as fibers/whiskers-reinforced HAp, fibrous HAp-reinforced polymers, or biomimetically fabricated HAp/collagen composites seem to be the most suitable ceramic materials for the future hard tissue replacement implants.

Order No.: JA801-015

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**ARTICLES****Equilibrium tie-line in  $\text{PrO}_y\text{-BaO-CuO}$  ternary phase diagram around peritectic temperature of  $\text{Pr}_{1+x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$** M. Tagami\*, M. Kambara\*, T. Umeda\*, Y. Shiohara\*  
(\*SRL-ISTEC, \*University of Tokyo)

This paper presents tie-lines between  $\text{Pr}_{1+x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$  and liquid on a  $\text{PrO}_y\text{-BaO-CuO}$  ternary phase diagram at 965, 970, 975, 980 and 990°C in air atmosphere, for which knowledge is necessary to fabricate composition controlled  $\text{Pr}_{1+x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$  single crystals by the solution

growth method. Liquidus faces have been investigated by dipping MgO single crystal rods into the thermal equilibrium melt at various temperatures and analyzing the composition of the adhering melt by ICP. The compositions of  $\text{Pr}_{1+x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$  solid solution coexisting with various compositions of liquids were obtained by quantitative EPMA analysis of quenched melts. Tie-lines were calculated by applying the lever-rule to these experimental data for solid compositions and liquidus faces. Furthermore, the relationships between solid solubilities and peritectic temperatures of  $\text{Pr}_{1+x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$  are reported..

Order No.: JA801-016

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**Uniform and rapid nucleation of diamond via bias assisted hot filament chemical vapor deposition**Y. Chen, F. Chen, E.G. Wang  
(Chinese Academy of Sciences)

A new method was developed to obtain high density, uniform diamond nuclei via bias-assisted hot filament chemical vapor deposition. A negative bias was applied between a mesh (installed above the filament) and the substrate to produce abundant uniform ions at the growth surface. Raman spectroscopy, scanning electron microscopy, and Auger electron microscopy were used to analyze the films obtained. The results show that a layer of diamond film with a nucleation density of  $10^9/\text{cm}^2$  can be obtained after 10 min deposition under 1 Torr.

Order No.: JA801-017

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**Molecular orientation in single crystal thin films of N-(4-Nitrophenyl)-(L)-prolinol**L. Zhou, M. Thakur  
(Auburn University)

Thin single crystal films of N-(4-nitrophenyl)-(L)-prolinol (NPP) were prepared using the modified shear method. The surface orientation of the single crystal films was determined by x-ray diffraction and was found to be [101]. Polarized microscopy showed uniform birefringence and complete extinction when the thin film was rotated under crossed polarization, implying single crystal thin films with uniform surface were obtained. The molecular orientation in the NPP thin film was studied by polarized UV-visible and polarized micro-FTIR spectroscopy along with x-ray diffraction. The orientation of the NPP molecules was found to be almost parallel to the plane of the film. This parallel orientation is because of the polar (hydrogen bonding) interaction of the -(OH) and the N=O groups of the NPP molecule with the hydrophilic substrate surface. The results of the second harmonic generation (SHG) measurements are consistent with such a molecular orientation. These results show that the final molecular and crystallographic orientation in the film is determined by its initial molecule-substrate interaction.

Order No.: JA801-018

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**A micromechanistic model of the combustion synthesis process: Modes of ignition**C. He, G.C. Stangle  
(New York State College of Ceramics at Alfred University)

A theoretical model of the combustion synthesis process has been developed to study the ignition of a self propagating combustion synthesis process in the Nb-C system. Compared with most of the previously published theoretical work on this subject, this model provides a much more detailed description of the combustion synthesis process from a micro-scale point of view, due to the fact that it takes into consideration the various microprocesses, such as the melting of reactants, the diffusion and mixing of reactants, and the formation of products. Different ignition modes, including constant temperature ignition, constant-heat-flux ignition and variable-temperature ignition, are considered in this work. The key parameters which influence the ignition process are also discussed.

Order No.: JA801-019

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**A micromechanistic model of the combustion synthesis process: Mechanism of ignition**C. He, G.C. Stangle  
(New York State College of Ceramics at Alfred University)

A micromechanistic model of the combustion synthesis has been extended to study the detailed mechanism and influential parameters of a

combustion synthesis process, as well as the development of ignition criteria in the Nb-C system. The case of constant heat-flux ignition conditions has been used to illustrate the details of the ignition process. In order to elucidate the various physical and chemical processes that take place during the initial stages of the combustion synthesis process; however, the results of this study can be generally extended to the other modes of the ignition process. The results showed that the ignition criteria for Nb-C system corresponded to the establishment of a proper balance between the rates of enthalpy redistribution within the sample, and to the establishment of a kind of positive feedback loop during the ignition process that is necessary for self-propagation to occur. If the heat supplied from an external source to initiate the combustion synthesis process is less than a certain critical value, the combustion wave stops a certain short distance from the ignition surface. Otherwise, the reaction proceeds in a self-propagating manner.

Order No.: JA801-020

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#### Auto ignition synthesis and consolidation of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> nano/nano composite powders

S. Bhaduri, S.B. Bhaduri, E. Zhou  
(University of Idaho)

An "Auto Ignition" technique was utilized in synthesizing Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> powders with nano/nano microstructure. The process used the corresponding nitrates as oxidizers and urea as the fuel. The as-synthesized powders were characterized by x-ray diffraction and transmission electron microscopy. It was observed that the microstructure consisted of crystallites of Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub>, both of which were nanocrystalline. As opposed to the other nanocomposite ceramics, this feature of the microstructure classifies the present powders as nano/nano type. This nanocrystallinity of the microstructure (crystallite size less than 100 nm) was maintained even after a soaking at 1200°C for two hours. Since the microstructure is stable at high temperatures, it was possible to densify the powders by hot isostatic pressing at 1200°C. The product was 99% of the theoretical density and maintained nanocrystalline grain size. The average hardness and toughness values, as determined by an indentation technique, were 4.45 GPa and 8.38 MPa m<sup>1/2</sup> respectively. These values represent evidence of ductility in these composites since transformation toughening was ruled out in this case. The potential application of these results are expected to be in net shape deformation forming of ceramics.

Order No.: JA801-021

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#### Mechanism of the formation of β-Sialon by self-propagating high-temperature synthesis

Y. Wu\*, H. Zhuang\*, F. Wu\*, D. Dollimore\*, B. Zhang\*, S. Chen\*, W. Li\*  
(\*Chinese Academy of Sciences, \*The University of Toledo)

β-Sialon has been prepared by SHS (self-propagating high-temperature synthesis) with Si, Al, Al<sub>2</sub>O<sub>3</sub> and β-Si<sub>3</sub>N<sub>4</sub> under high nitrogen pressure. Rod-like crystals of β-Sialon with an aspect ratio of 4~15 and whiskers with various aspect ratios were obtained in the process. Considering the morphologies of the products, it is concluded that VLS (vapor-liquid solid) is the predominant mechanism of the formation of rod-like crystals and whiskers.

Order No.: JA801-022

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#### Metal-organic chemical vapor deposition of Sr-Co-Fe-O films on porous substrates

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(\*University of New Mexico, \*Sandia National Laboratories)

Aerosol-assisted chemical vapor deposition using the β-diketonate precursors Sr(tmhd)<sub>2</sub>·2H<sub>2</sub>O, Fe(tmhd)<sub>3</sub> and Co(tmhd)<sub>3</sub> was investigated for depositing thin films of the mixed-conducting ceramic SrCo<sub>y</sub>Fe<sub>1-y</sub>O<sub>3-δ</sub> onto porous α-Al<sub>2</sub>O<sub>3</sub> substrates. Single-phase SrCo<sub>y</sub>Fe<sub>1-y</sub>O<sub>3-δ</sub> perovskite films were obtained at a deposition temperature of 550°C and pressure of 15 mm Hg, whereas deposition at atmospheric pressure produced mixed-phase films. The Co/Fe elemental ratios in the films reflected those in the precursor solution, but the films were depleted in Sr. Reduced-pressure deposition provided a more uniform film morphology than atmospheric-pressure, and led to a supported film which was leak-tight to N<sub>2</sub> flow.

Order No.: JA801-023

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#### An attempt to prepare carbon nitride by thermal plasma chemical vapor deposition from graphite and nitrogen

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RF induction thermal plasma was applied to the chemical vapor deposition of carbon nitride from graphite powders and Ar-N<sub>2</sub> gas at about 1 atm. Low-density and fragile amorphous powder-like bulk deposits were obtained whose color is light yellow. Elementary analysis by a combustion method and x-ray photoelectron spectroscopy showed that the N/C ratio is higher than that of stoichiometric C<sub>3</sub>N<sub>4</sub>. Also, a large amount of hydrogen and oxygen are included, which seems to be due to the absorption of moisture and oxygen after exposure to air. Infrared absorption spectra suggest the presence of sp CN and sp<sup>2</sup> CN bonds, and nitrogen-containing polycondensed ring structures. Thermogravimetric analysis with mass spectroscopy shows that the deposits decompose almost completely at 800°C, suggesting that the polycondensed rings are not large and not well cross-linked.

Order No.: JA801-024

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#### Structural investigation of Ba<sub>6-3x</sub>Ln<sub>8+2x</sub>Ti<sub>18</sub>O<sub>54</sub> (x = 0.27, Ln = Sm) by single crystal x-ray diffraction in space group Pnma (No. 62)

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Single crystals of barium samarium titanium oxide Ba<sub>6-3x</sub>Sm<sub>8+2x</sub>Ti<sub>18</sub>O<sub>54</sub> (x = 0.27) have been synthesized and studied using x-ray diffraction. Superstructure reflections, that cause a doubling of the cell along the short axis, were taken into account and the refinement was conducted in the orthorhombic space group Pnma. Unit cell parameters from single crystal x-ray diffraction were a = 22.289(1), b = 7.642(1), and c = 12.133(1) Å. Refinement on F resulted in R1 = 5.37% for 1410 F<sub>o</sub> > 4σ with the thermal parameters of the Sm and Ba atoms refined anisotropically and the thermal parameters of the Ti and O atoms refined isotropically. The structure is made up of a network of corner sharing TiO<sub>6</sub><sup>-2</sup> octahedra creating rhombic (perovskite-like) and pentagonal channels. The two pentagonal channels are fully occupied by Ba atoms. The refinement suggests that one rhombic channel is fully occupied by Sm atoms (Sm3/Sm4), one rhombic channel is partially occupied by Sm atoms (100%Sm1/86.25%Sm5), and one rhombic channel is shared by Ba/Sm atoms (59.25%Ba3/40.75%Sm2), resulting in a formula of Ba<sub>10.38</sub>Sm<sub>17.08</sub>Ti<sub>36</sub>O<sub>108</sub> with Z = 1. The above site occupancies differ from the site occupancies previously reported in the literature for refinements conducted with the short axis approximately equal to 3.8 Å.

Order No.: JA801-025

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#### A study of barium strontium titanate thin films for use in bypass capacitors

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Physical and electrical characterization techniques have been applied to the problem of developing a lower temperature process for spin-on Ba<sub>0.7</sub>Sr<sub>0.3</sub>TiO<sub>3</sub> thin films and capacitors compatible with on-chip aluminum metallization. The films were prepared by spin-coating from carboxylate precursors and were processed at temperatures between 650°C and 450°C. Capacitors annealed at higher temperatures have a dielectric constant (κ) of 382, a C/A of 20 fF/μm<sup>2</sup>, and a leakage current density of 2 x 10<sup>-7</sup> A/cm<sup>2</sup> at 3.3 V. Those processed at 450°C show occasionally promising but inconsistent results, correlated using TEM images with locally variable crystallization into the perovskite phase. The kinetics of the spin on solution chemical decomposition and crystallization has been investigated through the use of x-ray diffraction (XRD), thermogravimetric analysis (TGA), and Raman spectroscopy.

Order No.: JA801-026

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**Dielectric properties of dielectrophoretically assembled particulate-polymer composites**C.P. Bowen, R.E. Newnham, C.A. Randall  
(The Pennsylvania State University)

The dielectrophoretic effect is a phenomenon in which dipole-dipole interactions are induced between particles in a suspension by an electric field. This dipole interaction leads to the formation of chains or fibrils parallel to the applied electric field. Recently, the dielectrophoretic effect has been shown to be a possible composite assembly technique permitting property changes to be induced with the appropriate electric fields. The results presented in this paper show that the dielectrophoretic assembly process can be used to engineer anisotropy into composite materials. Various filler materials are aligned in a thermoset polyurethane matrix and the dielectric properties are measured. Comparisons are drawn between the dielectrophoretically assembled composites and those processed conventionally in the absence of an electric field. Dielectric properties are modelled with modified mixing laws and discussed in relation to the composite microstructure and the alpha relaxations of the polymer phase.

Order No.: JA801-027

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**The preparation of metal-polymer composite materials using ultrasound radiation**S. Wizel, R. Prozorov, Y. Cohen, A. Doron, S. Margel, A. Gedanken  
(Bar-Ilan University)

Ultrasound radiation is used to prepare a composite material made of polymethylacrylate and amorphous iron nanoparticles. Two preparation methods are described, in which the monomer, methylacrylate, is the starting material. The magnetic properties of the composite material are measured and reveal a superparamagnetic behavior.

Order No.: JA801-028

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**Initial stages of silicate growth on and oxidation of graphite: A model for composites**D.A. Bonnell, D. Jiron, T. Flinn  
(The University of Pennsylvania)

The initial stages of silicate growth on graphite are characterized with atomic force microscopy. The morphological development indicates that decomposition of tetra ethyloxysilane at low pressure produces films of 3 nm clusters located at undercoordinated carbon sites. Clusters eventually cover the surface, at which point a second layer grows. In higher pressure deposition multiple layers of clusters grow simultaneously. A comparison of the oxidation behavior of surfaces with defects completely and incompletely terminated with SiO<sub>x</sub> shows that edge recession is the primary oxidation mechanism and that the site specificity of SiO<sub>x</sub> is effective in inhibiting oxidation.

Order No.: JA801-029

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**Nanometric powders and sintered ceramics studied by atomic force microscopy**A. Dias\*, R.L. Moreira\*, N.D.S. Mohallem\*, J.M.C. Vilela#, M.S. Andrade#  
(\*EE-UFMG, #ICEX-UFMG, #Setor de Tecnologia Metalúrgica-Fundação Centro Tecnológico de Minas Gerais-CETEC)

Atomic force microscopy, as well as the Brunauer, Emmett and Teller technique and x-ray diffraction, was used to analyze ultrafine NiZn ferrite powders hydrothermally synthesized at 200°C, for 5 hours. The particle sizes, measured through AFM images acquired from the surface of pressed powders were (52 ± 6 nm), which were higher than those obtained by the other techniques. The particles were monodispersed in size and approximately spherical, meeting the requirements for the production of high density sintered components. The observations performed on ceramic bodies sintered at different conditions (1100–1400°C, 5 to 240 minutes) showed necks characteristic of the early stages of sintering (1100°C) and the expected pore curvature evolution (1400°C) with sintering time in the final stage of the sintering process. Using a straightforward sample preparation technique, AFM proved to be a powerful tool for direct analysis of ceramic powder particles on the nanometric scale and sintered ceramics at different sintering stages.

Order No.: JA801-030

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**Preparation and properties of potassium-vermiculite films**C. Minker-Villemin, P. Bowen, J. Lamaître, T.A. Ring  
(Swiss Federal Institute of Technology-EPFL)

Flexible films of vermiculite have been prepared from aqueous suspensions after swelling by cation exchange and mechanical delamination. Two different swelling cations, lithium and butylammonium have been investigated. The degree of swelling and delamination during the suspension preparation was characterized by the percentage of water reabsorbed by dried clays and the adsorption of methylene blue. The vermiculite saturated with lithium ions is more easily delaminated but contains more water than those saturated with butylammonium. Good quality coherent flexible films could be prepared from both the lithium and butylammonium exchanged vermiculites but the high percentage of water found in the films has a detrimental effect on their dielectric properties. To reduce the amount of water in the exchanged vermiculites a second ion exchange with potassium, a less hydratable cation, was investigated. Films prepared after exchange with potassium showed significant improvements in their dielectric properties, with a dielectric constant around 10 and a dissipation factor tan δ around 0.06 at 25°C and a frequency of 1 kHz.

Order No.: JA801-031

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## The Optical Society of America and the Materials Research Society Invite Applications for their 1998–1999 Congressional Science and Engineering Fellowship

**PROGRAM:** The Fellow spends one year working as a special legislative assistant on the staff of a member of Congress or Congressional committee. Activities may involve conducting legislative or oversight work, assisting in Congressional hearings and debates, and preparing briefs and writing speeches. The Fellow also attends an orientation program on Congressional and executive branch operations, which includes guidance in the Congressional placement process, and a year-long seminar series on science and public policy issues. These aspects of the program are administered by the American Association for the Advancement of Science for the OSA/MRS Fellow, and those Fellows sponsored by nearly two dozen other scientific societies.

**PURPOSE:** To provide OSA and MRS members with an invaluable public policy learning experience, to contribute to the more effective use of optical and materials science knowledge in government, and to broaden awareness about the value of scientist and engineer-government interaction among OSA and MRS members and within the federal government.

**CRITERIA:** A prospective Fellow must demonstrate a record of success in research or scholarship, in a field relevant to optical and/or materials science and technology. The Fellow must also demonstrate sensitivity toward policy issues and have a strong interest in applying scientific and technical knowledge to public policy issues. The Fellow must be able to work quickly and communicate effectively on a wide variety of topics, and be able to work cooperatively with individuals having diverse viewpoints. An applicant is expected to be a Member of OSA or MRS (or an applicant for membership) and have a doctorate. Post-doctoral experience is preferred.

**AWARD:** The Fellow will have a one-year appointment beginning September 1, 1998. The Fellowship stipend will be \$40,000 to \$47,000, plus money for health insurance, and travel and relocation expenses to the Washington, DC area. Final selection of the Fellow will be made in early 1998.

**APPLICATION:** Candidates should submit the following materials by December 8, 1997: (1) a detailed vita providing information about educational background, professional employment and activities, professional publications and presentations, public policy and legislative experience, and committee and advisory group appointments; (2) a statement of approximately 1000 words addressing the applicant's interests in the fellowship, career goals, contributions the applicant believes he or she can make as an OSA/MRS Fellow to the legislative process, and what the applicant wants to learn from the experience; and (3) three letters of reference, specifically addressing the applicant's ability to work on Capitol Hill as a special legislative assistant, sent directly to the address below.

Application material should be sent to  
OSA/MRS Congressional Science and Engineering Fellow Program, c/o MRS, 506 Keystone Drive, Warrendale, PA 15086

The deadline for applications is **December 8, 1997**

For additional information contact MRS at (412) 779-3004 x501 ([oare@mrs.org](mailto:oare@mrs.org)) or OSA at (202) 416-1418 ([ebaldw@osa.org](mailto:ebaldw@osa.org))