

## **DEPTH PROFILING OF PHASE COMPOSITIONS IN FUNCTIONALLY-GRADED ALUMINA/CALCIUM HEXALUMINATE COMPOSITES USING X-RAY DIFFRACTION AND THE RIETVELD METHOD**

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### **ABSTRACT**

Composites of alumina/ calcium-hexaluminate (A/CA<sub>6</sub>) synthesized via novel infiltration processing of a porous alumina preform with calcium acetate to yield a homogeneous layer of alumina and a heterogeneous graded layer of alumina/CA<sub>6</sub> have been developed. The depth profiling of phase compositions of this system has been determined by x-ray diffraction (XRD). Graded compositions with XRD Rietveld refinement analysis showed that the concentration of CA<sub>6</sub> decreases with depth, whereas  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> content increases with depth.

### **KEYWORDS**

Depth-profiling, phase abundance, x-ray diffraction, Rietveld method, functionally-graded materials, alumina/calcium-hexaluminate.

## **A COMPARISON OF BORIDES FORMED ON THE AISI 316L AND AISI 304 STAINLESS STEELS**

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### **ABSTRACT**

The present study compares some of the properties of borides formed on the AISI 316L and AISI 304, stainless steels. Boronizing, thermochemical surface hardening was performed in a solid medium consisting of Ekabor powders at 950°C for 2-8 h. It was observed that the hardness of borides formed on the surface of AISI 316L is higher than that of AISI 304 stainless steel while the depth of layer is lower. The presence of borides was verified by means of XRD analysis. The depth of coating layer formed on AISI 316L ranged from 12 to 40 µm while the depth same type of layer on AISI 304 stainless steel was between 12 to 50 µm. The hardnesses of borides formed on 316L and 304 stainless steels were about 1700 DPN and 1360 DPN respectively. Borides formed on both steel substrates have a smooth morphology in nature. EDS studies revealed that, nickel concentrates in the base metal beneath coating, chromium and manganese preferentially enter the coatings by substituting for iron in the Fe<sub>2</sub>B and FeB.

## **(Ce,Y)-TZP/Al<sub>2</sub>O<sub>3</sub> CERAMIC COMPOSITES**

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### **ABSTRACT**

An extensive experimental study was conducted to examine the mechanical properties, sintering behavior and toughening mechanisms in (Ce,Y)- TZP/Al<sub>2</sub>O<sub>3</sub> dual-phase ceramic composites. Results indicated that (Ce,Y)-TZP/Al<sub>2</sub>O<sub>3</sub> composites maintain high bending strength and fracture toughness over a wide range of Al<sub>2</sub>O<sub>3</sub> content, 10vol%-50vol%, rather than decrease sharply when the addition of Al<sub>2</sub>O<sub>3</sub> exceeds 10-20vol% as reported in other literature. The Vickers hardness increases with increasing Al<sub>2</sub>O<sub>3</sub> content. Densification and grain growth were investigated and explained in detail as a function of Al<sub>2</sub>O<sub>3</sub> volume fraction. The existence of second phase, Al<sub>2</sub>O<sub>3</sub> or ZrO<sub>2</sub>, improves sintering behavior and microstructure by inhibiting the movement of grain boundaries. Various toughening mechanisms that existed simultaneously in this binary system showed different contributions to fracture toughness at different compositions.

### **KEYWORDS**

Zirconia, alumina, mechanical properties, toughening.

## **DIFFRACTION STUDIES OF A NOVEL Ti<sub>3</sub>SiC<sub>2</sub>-TiC SYSTEM WITH GRADED INTERFACES**

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### **ABSTRACT**

A high-temperature vacuum heat-treatment process has been proposed for the designing of Ti<sub>3</sub>SiC<sub>2</sub>-TiC composites with graded interfaces. The phase evolution and the graded nature of this system have been characterised by x-ray diffraction (XRD), synchrotron radiation diffraction (SRD) and neutron diffraction (ND). Results of SRD and ND in the temperature range 1000-1500°C show that the TiC layer commenced to form near the surface at 1200°C and grew rapidly in thickness with rising temperature. Depth-profiling of the TiC layer by XRD and SRD has revealed a distinct gradation in phase composition.

### **KEYWORDS**

TiC, Ti<sub>3</sub>SiC<sub>2</sub>, x-ray diffraction, neutron diffraction, synchrotron radiation diffraction.

## **IMPROVEMENTS IN MICROSTRUCTURE AND PROPERTIES OF SrO DOPED CALCIA-STABILIZED ZIRCONIA**

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### **ABSTRACT**

Improvements in density and fracture toughness of calcia-stabilized zirconia are obtained by the addition of 3 mol% SrO. Evidence is presented which indicates that the added SrO effectively neutralizes the accumulation of SiO<sub>2</sub> containing grain boundary phases. It was also noted retention of tetragonal phase after sintering was high in the sintered body, which results in material with optimum properties.

### **KEYWORDS**

Strontia, calcia, zirconia, tetragonal, monoclinic, fracture toughness, grain boundary.

## **MACHINING TECHNOLOGY OF INSULATING Si<sub>3</sub>N<sub>4</sub> CERAMICS BY WIRE EDM**

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### **ABSTRACT**

Complex shape machining of insulating ceramics was tried by the wire cut electrical discharge machining process. Using the assisting electrode method developed by the authors, the insulating material has been machined by the sinking EDM method<sup>(1)</sup>. The Wire cut EDM method is one of the most useful methods to machine complex shapes on hard materials but it has been recognized as a more difficult process to machine insulating materials. In this paper, machining of the insulating Si<sub>3</sub>N<sub>4</sub> ceramics by WEDM was tried by considering different wire electrode materials and electrical machining conditions.

### **KEYWORDS**

Insulating ceramics, electrical discharge machining, wire electrode, electrical conductive layer.

## **A NEW SURFACE MODIFICATION TECHNOLOGY ON STEEL USING EDM MACHINE**

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### **ABSTRACT**

Many surface modification technologies have been proposed and practically carried out by CVD, PVD etc. In the EDM method, a carbonized layer is usually generated for the purpose of surface modification. In this paper, to make a nitride layer by EDM some new trial experiments were carried out using a titanium electrode in liquid nitrogen. TiN could be obtained on the EDMed surface. To confirm the formation mechanism of the nitride layer on the steel workpiece, the following experimental parameters were investigated: (1) working atmosphere, (2) electrode polarity, (3) electrode material, (4) electrical machining conditions (peak current, discharge duration and duty factor). Characteristics of the modified surface were investigated by SEM, EPMA, X-ray diffraction analysis.

### **KEYWORDS**

Electrical discharge machining, surface modification, liquid nitrogen, titanium nitride, electrode.

## **DECOLORATION OF WASTE COLORED GLASS THROUGH PHASE-SEPARATION AND ITS MECHANISM**

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### **ABSTRACT**

We propose a recycling process for waste colored glass based on the property of glass phase separation. The soda-lime-silicate glass with heavy metal ions such as Cr ions was re-melted with B<sub>2</sub>O<sub>3</sub>, and the glass thus obtained heat-treated and leached by hot acid solution. All the alkali and heavy metal ions were successfully removed from the glass and nearly pure SiO<sub>2</sub> was obtained. The decoloration mechanism was investigated. The glass structural change with composition and heat treatment was measured by <sup>11</sup>B and <sup>27</sup>Al NMR spectra. The valence and site of the Cr ions in the glasses before and after the heat and acid treatments were investigated by leaching rate, ESR and UV/VIS spectra. It was found that the process of removing the heavy metal ions from the waste colored glass is related to the composition and heat treatment dependent structural change of the glass.

### **KEYWORDS**

Waste colored glass, a recycling process, phase separation, NMR, ESR, decoloration mechanism.

## **CHARACTERISATION OF TWO KAOLIN DEPOSITS FROM SARAWAK, MALAYSIA**

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### **ABSTRACT**

Two new kaolin deposits from the state of Sarawak in Malaysia (located on the island of Borneo) were characterised and compared to a currently-worked kaolin deposit from the Tapah-Bidor area on peninsular Malaysia. The Sarawak deposit from Telaga Air is reported to derive from an intrusive weathering of dacitic sills or dykes whilst the Telagus deposit is believed to derive from sediments of the Sadong Formation. The deposit from peninsular Malaysia is reported to derive from the hydrothermal alteration of schist. Chemical analyses indicate significantly lower Fe<sub>2</sub>O<sub>3</sub> content and a higher K<sub>2</sub>O content in both the Sarawak deposits as compared to the Tapah-Bidor deposit. Mineralogical analyses establish that all three deposits are kaolinitic with substantial quartz contents and trace amounts of micaceous minerals, feldspathic minerals and gibbsite. Brightness tests also indicate higher values ( $\geq 80\%$ ) for both Sarawak deposits as compared to a value of  $\sim 74\%$  for the Tapah-Bidor deposit. Electron microscopic studies clearly show the difference in morphology of the kaolinitic minerals for all three kaolin samples. These results indicate the influence of geological origins on the characteristics of the kaolin deposits from Sarawak and that from peninsular Malaysia. Consequently, the applications of these raw materials should also be different.

### **KEYWORDS**

Kaolin, quartz, chemical composition, mineralogy, microscopy, brightness, weathering, hydrothermal.

## **SLIP CHARACTERISATION AND TAPE CASTING OF CERIUM OXIDE NANOPOWDER**

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### **ABSTRACT**

The utility of mechanochemically synthesized samarium doped cerium oxide nanoparticles in a tape casting process has been investigated using 25nm mechanochemically synthesized CeO<sub>2</sub> as a model compound. In ethanol the optimum amount of acidic copolymer dispersant was determined using rheometry. Thermal analyses allowed the determination of an optimum heating profile to obtain tape cast ceramic articles whose grains were examined using electron microscopy. Sintered densities between 53% and 67% of theoretical were observed in ceramic bodies having grain sizes from 450nm to 144nm. To obtain a sub-micron grain size it was found necessary to keep the sintering temperature below 1100°C.

### **KEYWORDS**

Milling, nanopowder, dispersants, tape casting, sintering, dilatometry.

## **FABRICATION OF MgAlON BONDED REFRACTORY BY REACTION SINTERING ON Al-Al<sub>2</sub>O<sub>3</sub>-MgO SYSTEM**

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### **ABSTRACT**

MgAlON bounded refractory was fabricated by reaction sintering, using aluminum, alumina and magnisite powder mixture as matrix materials and magnesia or magnesia-alumina spinel as aggregate materials. The results showed that sintering temperature, the Al/Al<sub>2</sub>O<sub>3</sub> in the matrix were noticeably influenced on the formation of MgAlON phase and the sintering properties of specimens. By means of selecting suitable materials and correct composition of matrix, the MgAlON bonded composite refractories prepared directly have been shown to exhibit good properties.

### **KEYWORDS**

MgAlON, nitridation, reaction sintering, MgAlON bonded refractory.

## **COMPARISON OF THE OXIDATION BEHAVIOUR OF SURFACE AND INNER BULK MATERIAL OF A MoSi<sub>2</sub> BASED COMPOSITE**

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### **ABSTRACT**

In this work a MoSi<sub>2</sub>-based heating element composite was studied. The influence of phase composition and morphology of surface and inner bulk material on the oxidation behavior was examined using XRD, ESEM, SEM and EDX. It is shown that the surface material forms a thick yellowish oxide layer (amorphous SiO<sub>2</sub>/crystalline MoO<sub>3</sub>) when oxidized at 490°C in O<sub>2</sub>, whereas no such oxide is formed on the inner bulk material. It is also shown that the surface curvature does not contribute to the formation of the yellowish oxide. The results show that heat treatment to form a SiO<sub>2</sub> scale also causes formation of Mo<sub>5</sub>Si<sub>3</sub> at the MoSi<sub>2</sub>/SiO<sub>2</sub> interface. At a higher heat-treatment temperature a larger amount of SiO<sub>2</sub> and Mo<sub>5</sub>Si<sub>3</sub> is formed. When the SiO<sub>2</sub> scale is mechanically removed the exposed Mo<sub>5</sub>Si<sub>3</sub> triggers the formation of a yellowish oxide during oxidation. In contrast, the inner bulk material that contains less Mo<sub>5</sub>Si<sub>3</sub> does not form this yellowish oxide.

### **KEYWORDS**

Molybdenum disilicide (MoSi<sub>2</sub>), pest, oxidation, Mo<sub>5</sub>Si<sub>3</sub>, Molybdenum trioxide (MoO<sub>3</sub>), micro-structure, SiO<sub>2</sub>.

## **SOLID SOLUTIONS OF HYDROXYFLUORAPATITE. INFLUENCE OF THE AMOUNT OF FLUORIDE ON MECHANICAL PROPERTIES**

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### **ABSTRACT**

Wet synthesis at room temperature was employed to produce hydroxyfluorapatite solid solutions and further processed to investigate the mechanical properties of sintered compacts. Calcined powders showed a decrease in the width of the unit cell with increasing fluoride content. Powders were densified by uniaxial and cold isostatic pressing and then sintered at 1150°C in an atmosphere of air with water vapour. Sintered pellets revealed a decrease in sintered pellet density for compositions containing intermediate fluoride compositions. This is thought to be attributed to decrease in diffusion from the hydrogen bonding between the hydroxide and fluoride ions within the structure. The linear decrease in the hardness of partially substituted hydroxyapatite compositions, despite being linked to increasing fluoride contents, is more closely related to the decrease in density. Hardness was greater for fully substituted apatites, namely hydroxyapatite and fluorapatite. Fracture toughness remained at the same value for low fluoride contents until decreasing for fluorapatite.

### **KEYWORDS**

Hydroxyfluorapatite, Solid solutions, Fluorapatite, Hydroxyapatite, Bioceramics, Sintering, Mechanical properties.

## **VALIDATION OF NOVEL EVALUATION METHOD FOR UNIFORMITY OF POWDER COMPACT**

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### **ABSTRACT**

Novel evaluation method was validated to examine uniformity of powder compacts using a confocal laser scanning microscope (CLSM) with fluorescence mode. In this method, internal structure of the specimen was evaluated as the change of the fluorescence intensity, which corresponds to two-dimensional powder packing of the region. Observation depth or time of scan also affected the fluorescence intensity detected. To compare the various specimens using this technique, optimal condition for these factors was found out. Good reproducibility was obtained for images of internal structure.

### **KEYWORDS**

Alumina, density, fluorescence, immersion liquid, microstructure, pore, powder compact, uniformity.

## **KINETICS OF SURFACE CARBONITRIDING OF YTTRIA-DOPED TETRAGONAL ZIRCONIA POLYCRYSTALS**

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### **ABSTRACT**

The low-temperature (200°C) environmental degradation in water of tetragonal zirconia polycrystals doped with 3mol% yttria (3Y-TZP) was effectively prevented by a carbonitriding heat treatment process in which the sintered samples of 3Y-TZP were buried in a uniformly mixed powder of ZrN and graphite at 1200°C to 1600°C for 2 to 8 hours. The effect on phase stability of the introduction of both nitrogen and carbon ions into tetragonal zirconia was compared with the individual effects of nitrogen or carbon ions. The stronger surface stability is attributed to the simultaneous effects of nitrogen and carbon ions since nitrogen and carbon ions occupy totally different sites in the zirconia lattice. The thickness of surface transformed layer was observed to increase by a parabolic rate law which shows that the carbonitriding process is controlled by diffusion. The combined diffusivity of carbon and nitrogen was evaluated from the temperature dependence of carbonitriding rate.

### **KEYWORDS**

Tetragonal zirconia, carbonitriding, low temperature degradation, phase stability, interstitial sites, anion stabilizers.

## **RHEOLOGICAL PROPERTIES OF GUNNING REFRACTORY**

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### **ABSTRACT**

This paper studies the factors influencing the rheological properties of gunning refractory suspension such as shear rate, shear time, water content, temperature, particle composition of refractory and dispersion reagent. The rheological and adhesive properties of gunning refractory are determined from the experiment. The results of rheological tests indicate that: the rheological curve of gunning refractory bonded by high-alumina cement is Bingham type; the rheological curve of low-cement gunning refractory bonded by ultrafine powder is characterized by pseudo-plasticity fluid with yielding stress; and the rheological property of low-cement gunning refractory bonded by ultrafine powder is proven to be better than that of gunning refractory bonded by cement. Meanwhile as indicated by the adhesive tests: the better the rheological properties of gunning refractory suspension, the better the repair effect has.

## **NANOSTRUCTURED SnO<sub>2</sub>:TiO<sub>2</sub> SOLAR CELLS SENSITIZED BY RUTHENIUM DYE**

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### **ABSTRACT**

The photoelectrochemical properties of nanostructured SnO<sub>2</sub>/TiO<sub>2</sub> bilayered and SnO<sub>2</sub>-TiO<sub>2</sub> composite solar cells sensitized by RuL<sub>2</sub>(NCS)<sub>2</sub> dye were studied. The bilayered system (cells) shows higher IPCE (incident photon-to-current conversion efficiency) value than the composite system. A maximum IPCE value attained was 88.1% at 540 nm wavelength in the bilayered system with SnO<sub>2</sub> of 3.5 μm and TiO<sub>2</sub> of 7 μm in the film thickness. The IPCE difference in the bilayered and composite systems is discussed.

### **KEYWORDS**

Nanostructure, solar cell, photoelectrochemical, IPCE, dye.

## **A NEW TECHNIQUE TO REMOVE HYDROGEN CHLORIDE GAS AT HIGH TEMPERATURE USING MAYENITE**

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### **SUMMARY**

Mayenite [Ca<sub>12</sub>Al<sub>10</sub>Si<sub>4</sub>O<sub>32</sub>(OH)<sub>6</sub>] is capable of reducing HCl in the temperature range 400-950°C. Chlorine ions are fixed as wadalite [Ca<sub>12</sub>Al<sub>10</sub>Si<sub>4</sub>O<sub>32</sub>Cl<sub>6</sub>]. Wadalite is formed by substituting between OH<sup>-</sup> and Cl<sup>-</sup> in cavity of three-dimensional framework. The crystal structure of the product phase Ca<sub>12</sub>Al<sub>9.9</sub>Si<sub>4.05</sub>O<sub>32</sub>Cl<sub>5.9</sub> was cubic, space group *I43d*, with unit cell  $a=12.0173(1)$  Å, and was similar to that of wadalite. The structure consists of a framework of (Al,Si)O<sub>4</sub> tetrahedra. A large cavity in the framework accommodates Ca-Cl-Ca linearly coordinated atoms.

### **KEYWORDS**

Mayenite, wadalite, high temperature.

## **THE EFFECT OF THE CaO TO P<sub>2</sub>O<sub>5</sub> MOLE RATIO ON THE CRYSTALLISATION OF CALCIUM PHOSPHATE GLASS**

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### **ABSTRACT**

The effect of the CaO to P<sub>2</sub>O<sub>5</sub> mole ratio on the crystallisation of calcium phosphate glass is studied based on the CaO to P<sub>2</sub>O<sub>5</sub> glass system. The investigation on this system is of interest due to its capability to be used as a bioceramic material. Phosphate glass with theoretical composition of 0.85, 0.95, 1.10 and 1.20 mole ratios were produced. Then, they were heat treated at 600°C with a heating rate of 10°C/min for 48 hours to produce calcium phosphate glass ceramic. The percentage of crystallisation, the crystal phases and microstructure of the glass ceramic produced was then determined. It has been found that the binary system of calcium phosphate glass undergoes surface crystallisation and the mole ratio of CaO to P<sub>2</sub>O<sub>5</sub> is the main factor in determining the end result of the crystallisation process. This parameter affects the percentage of crystallisation, types of crystal present and the crystal microstructure. It also ensures the chemical stability and the mechanical properties of the glass ceramic product.

### **KEYWORDS**

Phosphate glasses, CaO/ P<sub>2</sub>O<sub>5</sub> ratio, crystallisation, stability, microstructure, hardness.