

MANUFACTURE PROCESS OF (TiB₂-Al₂O₃)/Al COMPOSITES BY COMBUSTION SYNTHESIS AND HOT PRESSING

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ABSTRACT

(TiB₂-Al₂O₃)/Al Composites have been fabricated by SHS-HP (Self-propagating High-temperature Synthesis and Hot-pressing) from following chemistry reaction: $3\text{TiO}_2 + 5\text{Al} + (10+x)\text{Al} \rightarrow 3\text{TiB}_2 + 5\text{Al}_2\text{O}_3 + x\text{Al}$. Manufacture process, microstructure and mechanical property were studied in detail. Study results showed that the fabrication processes parameters (such as reactant stoichiometry, preheat temperature) have a great influence on the ignition temperature of synthesis reaction, reaction temperature, and the density of synthesized products. Three point bending testing and single edge notch bending tests (SENB) was used to measure the bending strength and fracture toughness of composites, the hardness was measured by micro-hardness measurement method. Mechanical property study show that the fracture toughness increases rapidly with increasing of Al content in composites, but the hardness decreases. The bending strength increases with increasing of Al content till about 30vol% and then decreases with the further increasing of Al content.

KEYWORDS

(TiB₂-Al₂O₃)/Al Composites, SHS-HP, manufacture process, microstructure.

EFFECT OF SINTERING AND POLING CONDITIONS ON THE PROPERTIES OF $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ CERAMIC DOPED WITH Pb-BASED GLASS FRIT

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ABSTRACT

The paper reports on the preparation of dense piezoceramics by way of reactive liquid phase sintering. Technique concerning a low-temperature sinterable process is developed by incorporating $4\text{PbO}\cdot\text{B}_2\text{O}_3$. The host system is a perovskite type piezoceramic, $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$. If small amounts of $4\text{PbO}\cdot\text{B}_2\text{O}_3$ glass powder are added to the calcined $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ ceramics, a liquid phase is formed during sintering. Hence, the piezoelectric and dielectric properties are enhanced and the sintering temperature can be reduced. The value of the k_p , K_{33}^T and Q_m is increased and dielectric loss tangent is decreased when small amounts of $4\text{PbO}\cdot\text{B}_2\text{O}_3$ dopants (i.e. 0.2 wt %) are added to the $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ system. With more $4\text{PbO}\cdot\text{B}_2\text{O}_3$ doping (i.e. 2 wt %), the values of k_p and K_{33}^T are decreased due to the glassy phase segregation at the grain boundaries.

KEYWORDS

Piezoceramics, dielectric and piezoelectric properties, sintering, poling.

STUDY ON CORUNDUM-SiC CONTAINING METAL PLASTIC PHASE REFRACTORY

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ABSTRACT

A novel refractory-corundum-SiC containing metal plastic phase composite refractory is introduced in the paper. The compositions, microstructure and properties of the material have been studied. The results show that the material is a high quality refractory for the lining of blast furnace. As the plastic metal mixing into hard corundum in the refractory, the refractory appears plastic characteristics, such as higher bulk density, lower porosity, and higher mechanical properties. When the active metal on the surface of brick, it is easy to react with the atmosphere of furnace to form silicon carbide, silicon nitride or Sialon, which possesses high performance of resistance to corrosion of molten slag and iron. It implies the refractory being a self-repaired capability, like a smart material. The refractory has been produced thousands tons and used in more than 30 blast furnaces.

KEYWORDS

Composite, refractory, metal plastic phase, corundum, silicon carbide.

UV-SHIELDING CERAMIC NANOPARTICLES SYNTHESIZED BY MECHANOCHEMICAL PROCESSING

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ABSTRACT

ZnO, TiO₂ and CeO₂ are known as UV-shielding ceramic materials that have advantages over organic UV absorbers for their photo-stability and non-hazardous nature to human bodies. However, they normally cause low transparency in the visible-light range due to light scattering by large particles, which is undesirable for many transparent UV-blocking applications in cosmetic and plastic industries. Light-scattering efficiency of particles can be drastically reduced by decreasing the particle sizes down below 100 nm. This paper reviews recent investigation on the synthesis of ZnO and CeO₂ nanoparticles by mechanochemical processing. The resulting particles had a significantly low degree of agglomeration, having mean particle sizes of ~ 25 nm and ~ 10 nm, respectively. The aqueous suspensions of the nanoparticles showed strong absorption in the UV-light range and high transmittance in the visible-light range. Mechanochemical processing offers the possibility of industrial-scale production of transparent UV-shielding ceramic particles for many applications.

KEYWORDS

Nanoparticles, mechanochemical processing, UV-shield, ultrafine powder, ZnO, CeO₂.

MOISTURE EXPANSION REVISITED: PHASE EQUILIBRIA

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ABSTRACT

Consensus has it that the post-fabrication moisture expansion of fired clay-based materials is governed by the presence of amorphous and/or vitreous phases. In this work, very simple calculations using the lever rule and the phase diagram of the $\text{Al}_2\text{O}_3\text{-SiO}_2\text{-K}_2\text{O}$ (A-S-K) system, were used to show how to select the compositions, among several commercial red-clay tiles of unspecified processing, that might be related and avoid an indiscriminate correlation of data points. In this way, the amount of liquid phase formed during firing, which remains in the fired body as a vitreous phase, can be used to dissect the experimentally observed dependencies between the A/S and K/A weight ratios and moisture expansion. This work also shows how that liquid phase first controls densification (porosity, water absorption, mechanical strength) and then affects moisture expansion. Bearing this in mind, an educated choice of processing conditions and additives can be made.

KEYWORDS

Moisture expansion, fired-clay ceramics, phase equilibrium.

GEL INDUCTION TIME OF ALUMINA GELCASTING SLIP

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ABSTRACT

The ball milling effect on gel induction time of alumina slip was investigated. The 55vol% of alumina gelcasting slip was prepared by ball milling overnight. Initiator, 1g/L of ammonium persulfate, was mixed for gelling of the alumina slip.

For a specific mechanochemical treatment of alumina slip, different gel induction time can be achieved by changing polymerization temperature, which results in obtaining the activation energy vs. milling time curve. As results, the gel induction time of 55vol% of alumina slip decreased significantly as increasing milling time. The activation energy of the alumina slip was much lower than that of the premix solution at the same composition of organics. As milling time increased the gel induction time of alumina slip decreased, which is referred to the free radical of acrylamide activated mechanochemically.

KEYWORDS

Gel induction time, working time for slip handling, dispersion, and alumina gelcasting.

EFFECT OF ALUMINA ADDITION ON TETRAGONAL-TO-MONOCLINIC MARTENSITIC TRANSFORMATION IN ZIRCONIA-YTTRIA SYSTEM

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ABSTRACT

The effect of alumina addition of 1.5~ 30 mol% on the tetragonal (T) - to - monoclinic (M) (martensitic) phase transformation in zirconia-2 and 3 mol% yttria ceramics has been studied by XRD and thermal dilatation measurement. Total expansion due to the T-to-M transformation during the cooling stage increases with increase in alumina additions, though the amount of M phase estimated from XRD at the ceramics surface decreases with these additions. The alumina addition introduces grain growth of T phase and a small increase in transformation rate in 3 mol% yttria. The ratio of XRD intensities of two M peaks, (111)_m/(11 $\bar{1}$)_m, increases from 0.2 to around 0.64, which is the theoretical value, with increasing alumina additions. This change from anisotropy to isotropy of the M phase can be attributed to relaxation phenomenon of the transformation stress caused by the alumina addition, and culminates in a large expansion during T-to-M transformation.

KEYWORDS

Martensitic transformation, zirconia-yttria, alumina addition, isothermal transformation, grain growth, XRD, thermal dilatation.

PROCESSING OF ALUMINIUM TITANATE USING FAST HEATING METHODS

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ABSTRACT

Two fast heating processes were evaluated for reaction sintering of Al₂TiO₅ (AT) with and without additives. One method is pulsed electric current sintering (PECS), a process similar to hot uniaxial pressing. In the other a powder is melted in a cold crucible (CCM) by induction heating at high frequencies. Both methods are discussed. Al₂O₃ and TiO₂ powders were reaction sintered at the 30 g scale to form AT at 1450°C or 1650°C by PECS with a dwell time of 5 min. The material processed at the higher temperature had a density and grain size of 94% (of true density) and 25-80 μm respectively and for the lower temperature material the corresponding values were 85% and 10-40 μm respectively. A ~1 kg similar powder mixture was melted in a CCM for 5 min and after cooling it consisted of AT with < 5% rutile. Similarly in the CCM, an Al₂O₃, TiO₂, Fe₂O₃ and SiO₂ powder blend was melted to form a material consisting of 95% AT-structured Al_{1.57}Fe_{0.43}TiO₅, 2% corundum and 3% aluminosilicate glass. Pellets formed from this finely ground melt were sintered at 1550°C for 4 h. The density of this material was 94% (of true density) and it maintained the same phase assemblage.

The total cycle time for processing by PECS was ~ 40 min, and melting by CCM took ~90 min. The sintered AT-rich materials were similar to those produced by conventional sintering. Both techniques offer fast processing and are suitable for fabricating AT materials.

KEYWORDS

Aluminium titanate, novel processing, pulsed electric current sintering, cold crucible melting, sintering.

SCRATCH RESISTANCE IMPROVEMENT OF SOL-GEL DERIVED ANTIREFLECTIVE COATINGS BY INCORPORATION OF POLYETHYLENE GLYCOL IN ACID CATALYSED SOLS

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ABSTRACT

Porous silica antireflective coatings were prepared by incorporation of polyethylene glycol (PEG) in acid catalysed sols. PEG decomposed during curing, leaving pores in the films, resulting in a decrease in refractive index and an increase in transmission. Properties of the films were analysed by SEM, AFM, FTIR, ellipsometry and a scratch test. Very uniform porous films were obtained by adding PEG into sols that contain highly condensed silica particles. After curing at 400°C, films exhibited transmissions of up to 99.7%. Scratch resistance of these films is considerably better than that of coatings prepared from the conventional alkali catalysed and mixed catalysed sols with comparable refractive indices. A further improvement in scratch resistance was achieved without degradation in transmission by increasing curing temperature to 550°C. Increased curing temperature results in further condensation of silanol groups, and greater cross-linking of Si-O-Si bonds within the individual island of silica particles, while maintaining the domain pores produced by the decomposition of PEG.

KEYWORDS

Scratch resistance, sol-gel, antireflective (AR) coating, polyethylene glycol (PEG), refractive index (n), transmittance.

STUDY ON ENGINEERING PROPERTIES OF FLY ASH-BASED GEOPOLYMER CONCRETE

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ABSTRACT

The necessity to develop a new technology to produce energy-efficient and environmentally friendly concretes is evident, as it is known that cement production not only consumes large amounts of energy but also contributes substantially to the greenhouse gas emissions. Inorganic materials containing mostly silicon (Si) and aluminium (Al) may be activated by alkaline solutions to form a geopolymer gel that binds the coarse and fine aggregates to form the concrete. The process needs significantly less energy and will not produce CO₂ during production.

This paper presents the results of a study on the engineering properties of geopolymer concrete utilising an industrial by-product, i.e. low calcium (class F) fly ash, as its base material. The results show that curing temperature, curing time, sodium silicate-to-sodium hydroxide (NaOH) liquid ratio, and sodium hydroxide (solid)-to-free water ratio play important roles in determining the geopolymer concrete compressive strength. The unit weight of geopolymer concrete is similar to that of the ordinary Portland cement concrete.

KEYWORDS

Geopolymer, fly ash, compressive strength, engineering properties, curing temperature, curing time, NaOH-free water ratio, sodium silicate/NaOH ratio.

SYNROC CERAMICS FOR NUCLEAR WASTE IMMOBILISATION

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ABSTRACT

The development of titanate ceramics for incorporation of high-level radioactive wastes is first reviewed, tracing the evolution of synroc-C designed for Purex-type waste through glass-ceramics for tank wastes and pyrochlore-rich ceramics for surplus impure plutonium. The remainder of the paper deals with scientific aspects of the waste ceramics with focus on actinides. The valences of actinides, notably U and Pu, incorporated in these ceramics depend on crystal field effects and the presence of charge compensators, as well as the prevailing redox conditions during equilibration of the ceramics at subsolidus temperatures. Experimental techniques used to investigate immobilisation include X-ray near-edge absorption, diffuse reflection and X-ray photoelectron spectroscopy, and electron microscopy. The occurrence of U⁴⁺ and U⁵⁺ in zirconolite, brannerite and pyrochlore, as well as the pyrochlore-rich titanate ceramics themselves, is detailed. Both Pu³⁺ and Pu⁴⁺ can be accommodated in zirconolite, pyrochlore and perovskite.

KEYWORDS

Titanate ceramics, nuclear wastes, actinides.

FACTORS AFFECTING CRYSTAL GROWTH IN HIGH-TEMPERATURE CERAMIC GLAZES

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ABSTRACT

Crystalline glazes were first developed commercially for the ceramic industry in France in the mid-nineteenth century. They became popular in Europe and America in the 1880s, and have been in use sporadically since then. With the developments in kiln technology over the past twenty years, there has been a renewed interest in them, especially within the studio pottery movement in the western world.

This paper describes some factors that affect the growth of crystals in high-temperature glazes and examines ways to manipulate these to develop a desired technical and aesthetic quality. In particular, the paper examines the influence of the glaze recipe and the firing cycle.

KEYWORDS

Glazes, crystalline, high-temperature.

**A REPORT ON A TRIAL TO USE TAILINGS FROM A ZINC MINE
AND DROSS FROM A LEAD RECLAMATION FACILITY TO
MAKE A CERAMIC BODY AND GLAZE**

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ABSTRACT

Waste avoidance is at the top of the hierarchy of options for waste management and is seen as the ideal situation for industrial activity. It is probable that waste avoidance will never be achieved for all industries. Waste reclamation (recycling) is an option that is seen to be more viable for a number of industries and there are some good examples of recycling as a waste management tool.

This paper argues that some hitherto little used industrial process wastes may yet have a future as raw material and discusses some examples. It concludes with an examination of a waste produced by a lead reclamation plant and tailings produced at a zinc mine and suggests a recycling option for each. It suggests that one may have value in low temperature glaze formulation and the other as a raw material in ceramic bodies.

KEYWORDS

Tailings, recycling, raw materials, waste reclamation, waste reuse, glaze, clay body, process wastes.

CLAY DEPOSITS OF WESTERN AUSTRALIA

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ABSTRACT

Western Australia has large resources of high-quality kaolin and ball clay as well as many established industries manufacturing clay pipes, high-quality bricks, and tiles. Also, there are many undeveloped deposits of high-grade clay of suitable quality for a range of other high-value ceramic products.

The known kaolin resource (including ball clay) in the State is approximately 300 Mt, and a significant proportion of this is suitable for the ceramic industry. Kaolin deposits at Jubuk, Ockley-Wickepin, Gabbin, Mount Gibson, Mullewa, and Kerrigan have formed as residual products of weathered granitic rocks in areas of mature drainage systems. The Greenbushes kaolin deposit was formed by weathering of numerous pegmatites. Mount Kokeby and Goomalling kaolin/ball clay deposits were developed in palaeochannels and lacustrine environments. These major ceramic clay deposits are situated between 150-300 km from the major ports of Fremantle, Bunbury, and Geraldton.

Many kaolin deposits in Western Australia contain kaolin of acceptable brightness and chemical grade for the ceramic industry, but more detailed testing on rheology, particle size distribution and other parameters for specific applications is required to evaluate fully many of these deposits.

The identified structural clay resource in the Perth metropolitan region is estimated at around 68.2 Mt. Clay producing localities in the Perth region include Muchea-Bullsbrook East, Swan Valley, Armadale-Cardup-Byford-Mundijong, Toodyay, Chittering, and Gidgegannup, all within a radius of 40 km from Perth city. Test results of construction industry clay samples collected from localities around Albany, Ravensthorpe, Kalgoorlie, and Roebourne, indicate that most material is suitable for use as structural clay. Western Australia produced 65 080 t of kaolin (valued at \$3 million) during the period 1939-2000. The State has also produced 715 355 t of ball clay (valued at \$4.8 million) from 1932 to 2000, and 6 Mt of variable grade fireclay (valued at \$5.8 million) from 1922 to 2000. The construction industry clay production in Western Australia is considered to be in excess of 2.3 Mtpa estimated at \$29 million.

Western Australia has enormous potential for the production of high-grade ceramic clays. Together with a well-established structural clay industry, Western Australia is on track to become a market leader through exports of quality ceramic-grade clays and added-value building materials. During 2001, Western Australia imported ceramic products valued at \$469 million. Value of exported ceramic products during the same year amounted to \$15 million.

KEYWORDS

Western Australia, Greenbushes, Jubuk, Ockley-Wickepin, Gabbin, Kerrigan, Mount Kokeby, kaolin, ball clay, fireclay, production, resource, granite, pegmatite, rheology, particle size fractionation, brightness.

THE EFFECT OF FILM MORPHOLOGY ON CRACK RESISTANCE IN PEROXO-TITANIA/PEG SYSTEM

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ABSTRACT

Porous crystalline (anatase) TiO₂ films were prepared using a peroxo titania (PT) sol that was produced by a combined sol gel hydrogen peroxide treatment. Polyethyleneglycol (PEG) was used to improve crack resistance as a pore creator. Film morphology was found to be a function of coating and drying conditions and it was an important factor in controlling crack resistance of the films. In order to improve crack resistance of the PT film, the film morphology was controlled using different spin conditions. The effect of film morphology on film properties such as porosity, stress, pore size and film hardness were investigated.

KEYWORDS

Spin coating, film porosity, film stress, film hardness, condensation, phase separation.

EFFECT OF β -SPODUMENE ON PHASE DEVELOPMENT IN ALUMINA /ALUMINIUM TITANATE CERAMICS

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ABSTRACT

The effect of β -spodumene additions on the in-situ phase formation and abundances in alumina/aluminium-titanate (AT) composites at temperatures in the range 1000-1400°C has been studied by neutron diffraction and differential thermal analysis. Results show that β -spodumene began to decompose by phase separation and partial melting at 1290°C, followed by complete melting at 1330°C. Formation of AT was observed to occur at 1310°C and its abundance increased with temperature. β -spodumene did not react with alumina or rutile to form additional phases. Addition of β -spodumene in excess of 5 wt% resulted in pronounced vitrification which partly recrystallised when cooled to room temperature. The temperatures of AT formation and melting of β -spodumene are consistent with the results of differential thermal analysis.

KEYWORDS

Neutron diffraction, β -spodumene, aluminium-titanate, alumina, phase abundance, Rietveld refinement.

COMPRESSIVE STRENGTH AND MICROSTRUCTURAL CHARACTER OF ALUMINOSILICATE GEOPOLYMERS

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ABSTRACT

Aluminosilicate inorganic polymers, known as geopolymers, were synthesised from dehydroxylated kaolinite activated with sodium silicate in an alkaline environment. The purpose of this study was to investigate the influence of the atomic ratio Si:Al and Na:Al as well as the inclusion of aggregate (in the form of α -quartz) on the compressive strength of geopolymers.

The X-ray diffraction patterns suggest that aluminosilicate geopolymers were essentially amorphous. The final 14-day compressive strength of the resulting materials was in the range 60 - 90 MPa. It was found that the addition of aggregate did not influence the strength of the materials. Scanning electron microscopy examination of the microstructure showed that the presence of aggregate has not reduced the number of pores or prevented the formation of cracks. It is well known that the presence of pores and cracks will significantly reduce the strength of geopolymer.

KEYWORDS

Aluminosilicate, inorganic polymer, geopolymer, crack, microstructure.

SAXS STUDIES ON LANTHANUM-DOPED MESOPOROUS ALUMINA

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ABSTRACT

SAXS was used as an effective and powerful technique for characterization of the structure of lanthanum doped mesoporous alumina powder. From XRD results it was originally thought that these powders exhibited ordered porosity and a primary d spacing of 7.9 nm. A combination of SAXS and TEM has shown that this powder does not exhibit an ordered porosity. TEM showed evidence of pores ranging in size from 2 - 10 nm, and the SAXS results indicate a cylindrical form factor. Using the approximation of monodisperse cylinders, analysis of the Guinier and Porod regions resulted in two estimates of a cylindrical pores average radii and height (r, h): (1.4, 2.6) and (1.2, 3.5) nm. Analysis of the scattering data in the high q Porod region (showing a slope of -4) and the low q Guinier region resulted in a calculated specific surface area of 550 m²g⁻¹. This is compared to 275 m²g⁻¹ determined from nitrogen adsorption results. The higher specific surface area determined by SAXS reflects the fact that the SAXS technique can access closed porosity, whereas the nitrogen absorption technique can only access open porosity.

KEYWORDS

Small angle x-ray scattering, mesoporous, alumina, lanthanum doped alumina, surface area, pore size.

TOWARDS THE DETERMINATION OF THE STRUCTURE OF γ -ALUMINA

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ABSTRACT

Gamma alumina (γ -Al₂O₃) is an industrially significant material. However, controversy remains over its structure. This paper details the examination of five single phase and two dual phase structure models of γ -Al₂O₃ using Rietveld analysis and nuclear magnetic resonance spectroscopy. Results show that the existing models do not provide a satisfactory fit to the data obtained here. However, if the distribution of aluminium is assumed to be isotropic, there is evidence that the structure could be either tetragonal, or consisting of two phases, as opposed to the traditional cubic spinel.

KEYWORDS

γ -alumina, Rietveld analysis, nuclear magnetic resonance, *ab initio*.