

## **IMMOBILISATION OF RADIOACTIVE $^{129}\text{I}$ IN A LEAD VANADATE MATRIX BY IN-CAN HOT ISOSTATIC PRESSING**

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### **ABSTRACT**

One method of capturing radioactive  $^{129}\text{I}$  is to adsorb it from a gas stream heated to  $\sim 200^\circ\text{C}$  onto  $\text{AgNO}_3$ -impregnated porous alumina or silica sorbents, with the I forming principally  $\text{AgI}$ . A lead vanadate matrix was selected to examine its suitability for encapsulating non-radioactive I-loaded sorbents by hot isostatic pressing (HIPing) in a stainless steel can at  $700^\circ\text{C}$ . After HIPing, the  $\text{AgI}$  was shown to be contained within the sorbent by scanning electron microscopy and energy dispersive X-ray analysis. There was no observable reaction of the  $\text{AgI}$  or the alumina sorbent with the matrix. No elemental iodine was detected on the inside of the can or in the matrix, showing iodine had not volatilised. However, although limited aqueous durability tests showed good leach resistance for iodine, this was not so for the Ag and Pb and more work on this aspect is necessary.

### **KEYWORDS**

Iodine-129, hot isostatic pressing, radioactive waste,  $\text{AgI}$ , lead vanadate.

## **AN EMPIRICAL RELATIONSHIP BETWEEN NITROGEN DEPOSITION PRESSURE AND HARDNESS OF MAGNETRON CO-SPUTTERED TERNARY NITRIDE COATINGS**

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### **ABSTRACT**

An empirical equation of the hardness of ternary nitride coatings and the nitrogen deposition pressure was formulated in the present study. A linear relationship was established between the coating hardness and the reciprocal of the square root of nitrogen pressure. The primary sputter yield of the coating materials was found to contain a linear relationship with the minimum hardness value,  $H_0$ , of the empirical equation but showed no effect on the rate of hardness increase,  $m_H$ . With defined discharge conditions and sputter yields of the target materials, the values of  $m_H$  and  $H_0$  can be calculated and the hardness of a coating produced at a specified nitrogen deposition pressure can be theoretically determined.

### **KEYWORDS**

Ternary nitride coatings, magnetron sputtering, nitrogen deposition pressure.

**EFFECT OF SINTERING AND POLING CONDITIONS ON THE  
PROPERTIES OF 0.875Pb(Ti<sub>0.5</sub>Zr<sub>0.5</sub>)O<sub>3</sub>-0.125Pb(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub>  
CERAMIC WITH PbO-BASED GLASS FRIT**

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**ABSTRACT**

The influence of sintering and poling conditions on the dielectric and piezoelectric properties of the 0.875Pb(Ti,Zr)O<sub>3</sub>-0.125Pb(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub> system with PbO-based glass frit was investigated. Specimens were prepared by the conventional mixed-oxide technique. If small amounts of 4PbO·B<sub>2</sub>O<sub>3</sub> glass powder are added to the calcined 0.875PZT-0.125PMN ceramics, the liquid phase is formed during sintering. Hence, the piezoelectric and dielectric properties are enhanced and the sintering temperature can be reduced. Depending on the doping amount, the 4PbO·B<sub>2</sub>O<sub>3</sub> appears not only to be a grain growth promoter within the solubility limit, but also to be a grain growth inhibitor above the solubility limit. For the poling process, poling efficiency is a main consideration. The optimum poling conditions were 3 kV/mm for 30 min at 90°C. The values of the  $k_p$ ,  $K_{33}^T$  and  $Q_m$  are increased when small amounts of 4PbO·B<sub>2</sub>O<sub>3</sub> dopants (i.e. 0.2 wt %) are added to the 0.875PZT-0.125PMN system. With more 4PbO·B<sub>2</sub>O<sub>3</sub> doping (i.e. 1 wt %), the values of  $k_p$  and  $K_{33}^T$  are decreased due to the glassy phase segregation at the grain boundaries.

**KEYWORDS**

Dielectric and piezoelectric properties, domain reorientation, poling, sintering, pyrochlore.

## **CHARACTERISATION OF PHASE RELATIONS IN AIR- OXIDIZED TITANIUM SILICON CARBIDE (Ti<sub>3</sub>SiC<sub>2</sub>)**

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### **ABSTRACT**

The nature and processes of oxidation of Ti<sub>3</sub>SiC<sub>2</sub> in air from 25-1500°C have been characterised by differential thermal analysis, x-ray diffraction and neutron diffraction. Diffraction results show that rutile formed at ~750°C and a glassy phase formed at <1000°C which subsequently crystallized to cristobalite at ~1300°C. The oxides formed at the near-surface are layered and exhibit a duplex microstructure with an outer layer of TiO<sub>2</sub> and an inner layer consisting of SiO<sub>2</sub> and TiO<sub>2</sub>.

### **KEYWORDS**

Ti<sub>3</sub>SiC<sub>2</sub>, rutile, cristobalite, oxidation, x-ray diffraction, neutron diffraction.

## **TITANIUM ISOPROPOXIDE POST-TREATMENT OF TITANIUM DIOXIDE ELECTRODES FOR USE IN DYE-SENSITISED SOLAR CELLS**

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### **ABSTRACT**

Surface modification of titanium dioxide (TiO<sub>2</sub>) electrodes for application in dye-sensitised solar cells (DSSCs) using 0.1M and 1M solutions and neat titanium isopropoxide (TIP) was performed. The modified electrodes were characterised by X-ray diffraction (XRD) and scanning electron microscopy (SEM) and DSSCs incorporating these electrodes were constructed and tested. The homogeneity and electrical connectivity of the TiO<sub>2</sub> layer appeared to improve in the case of the 0.1M TIP post-treatment, and the cell efficiency was found to increase from 0.8% to 1%. This rise in cell efficiency is proposed to be due to the increased necking in the semiconductor, which is critical to electron conduction. However, if TIP is used at a higher concentration (1M or neat) a monolithic sol-gel monolithic layer was formed on top of the TiO<sub>2</sub>. This had a much greater effect on cell efficiencies resulting, in an increase from below 1% up to 3%.

### **KEYWORDS**

Dye-sensitised solar cells, TiO<sub>2</sub> working electrodes, nanostructured TiO<sub>2</sub> network, electrode post-treatment.

## **OXIDATION BEHAVIOUR OF ZIRCONIUM DIBORIDE**

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### **ABSTRACT**

Oxidation behaviour of dense zirconium diboride ( $ZrB_2$ ) was investigated over a temperature range of 873 to 1273K in static air. Pressureless sintering of fine powder at 2223K produced the dense  $ZrB_2$  product. Both isothermal and non-isothermal studies were carried out. It has been seen that during oxidation two types of oxides (boron oxide and zirconia) were formed on the surface of the sample. Formation temperatures of both the boron oxide ( $B_2O_3$ ) and zirconia ( $ZrO_2$ ) were detected by Differential Thermal Analysis (DTA) technique. The activation energy for oxidation was estimated to be approximately 8.23 kJ/mol. A boron oxide layer was formed at lower temperatures. At higher temperatures  $B_2O_3$  vaporized and only a crystalline  $ZrO_2$  layer was observed on the surface of the sample.

### **KEYWORDS**

Oxidation, Sintering,  $ZrB_2$ , Fracture Surface, Crystalline, Evaporation, XRD, SEM.

## **MICROWAVE HEATING OF CERAMICS – A REVIEW**

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### **ABSTRACT**

A brief review of microwave heating of ceramics is presented. The basis of microwave heating of ceramics is explained and its advantages compared with conventional ceramic heating processes are discussed. A comprehensive tabulated summary of published microwave heating studies for which non-thermal heating effects leading to the enhancement of diffusion-based processes, such as densification, are either claimed or discounted is presented. The experimental limitations of microwave heating studies and their significance in the interpretation of microwave heating data also are considered.

### **KEYWORDS**

Microwave heating, thermal gradients, sintering.

## NEAR-EQUILIBRIUM PROCESSING OF CERAMICS FOR ACTINIDE DISPOSITION

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### ABSTRACT

A baseline pyrochlore-rich ceramic designed for Pu-immobilisation, nominally 95 wt%  $\text{Ca}_{0.89}\text{Gd}_{0.22}\text{Hf}_{0.23}\text{Pu}_{0.22}\text{U}_{0.44}\text{Ti}_2\text{O}_7$  plus 5 wt%  $\text{Ti}_{0.9}\text{Hf}_{0.1}\text{O}_2$ , has been studied. The samples were prepared via by alkoxide-route to give samples having near-equilibrium mineralogy and via oxide-routes using wet or dry milling to give samples similar to those of production scale. Samples containing Pu and Th as a Pu analogue were studied. Samples were sintered in oxidizing, neutral (Ar) and reducing conditions (3.5%  $\text{H}_2$  in Ar). Related ceramics were prepared based around “zirconolite-rich” and “brannerite-rich” formulations, plus a formulation originally designed to contain ~ 10 % perovskite (or “Ca-rich”). Pyrochlore was the main actinide host phase and brannerite and zirconolite were the secondary actinide-bearing phases found in the materials, with a small amount of perovskite forming in most of the samples sintered in hydrogenous atmospheres. The ceramics were found to be extremely flexible with respect to processing conditions and composition, with the composition changing slightly to accommodate variations in the valency of the U and Ti with sintering atmosphere.

### KEYWORDS

Plutonium immobilisation, waste forms, pyrochlore, zirconolite.

## **CHEMICALLY-MIXED POWDERS FOR SOLID OXIDE FUEL CELLS**

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### **ABSTRACT**

Yttria-stabilised zirconia is commonly used as both the electrolyte and as an anode support structure in Solid Oxide Fuel Cell (SOFC) manufacture. To allow for ionic conduction, the zirconia and yttria must be in solid solution with a consistent distribution allowing control of the zirconia polymorphs. The addition of alumina to the stabilised zirconia facilitates the scavenging of any contaminant silica that may be present as well as allowing for the control of microstructure through grain pinning. This addition also acts as a sintering aid. For consistent performance, the three elements need to be intimately mixed with a homogenous distribution.

Mapping of the distribution of elements in 10 mol % yttria-stabilised zirconia doped with alumina by energy dispersive spectroscopy shows that the elements Y, Al and Zr are homogeneously mixed at the nanometre scale. The sintered ceramic produced from the precursor has a homogenous mixture of yttria and zirconia. The alumina separates and forms deposits, some crystalline, through the ceramic matrix. Amorphous alumina tends to form in the grain boundaries and at triple points.

The 10 mol% yttria-stabilised zirconia doped with alumina was produced by Millennium Chemicals using a process that is of a proprietary nature.

### **KEYWORDS**

Solid Oxide Fuel Cells (SOFC), Yttria-stabilised zirconia, SEM, TEM, EDS elemental mapping.

## **CAN COMPUTER-INTEGRATED DESIGN AND FINITE ELEMENT ANALYSIS BE APPLIED TO THE TRADITIONAL CERAMICS? AN INDUSTRIAL DESIGNER'S VIEW**

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### **ABSTRACT**

Integrated computer-aided design, finite element analysis, and manufacturing are currently applied in high-tech ceramic production areas. With the recent improvement of computer technology and the development of more powerful but compact and user-friendly programs, the cost of production can be further reduced. This technique can be applied to the traditional ceramics. This paper provides a short review of this integrated technology and gives an industrial designer's view on applying and improving the production quality of traditional ceramics and reducing extensive experimentation involved in the trial-and-error approach.