

***In Vitro* BIOMIMETIC DEPOSITION OF APATITE ON CHEMICALLY AND ELECTROCHEMICALLY TREATED TITANIUM**

A. OSAKA*, S. HAYAKAWA, K. TSURU, S. TAKEMOTO, Y. KAWABE and S. IWATANI

*Biomaterials Laboratory, Faculty of Engineering, Okayama University, Tsushima,
Okayama-shi 700-8530, Japan*

**email: osaka@cc.okayama-u.ac.jp*

ABSTRACT

Titanium was oxidized with H₂O₂ aqueous solutions, simple heating in the air, and anode polarization to yield titanium oxide layers. Ability of those layers to induce biomimetic apatite deposition in a simulated fluid was evaluated by X-ray diffractometry and scanning electron microscopy. Heating in air at temperatures in the range 200° to 700°C yielded the oxide layers with little ability. The treatment with 30 mass% H₂O₂ solution was similarly almost ineffective for biomimetic apatite deposition, but the subsequent heating at 300° ~ 700°C in air significantly improved the ability. Such enhancement was attributed to an epitaxial induction of apatite nucleation caused by the precipitation of anatase on heating. Addition of Ta(V) in a few mmol/l caused a moderate positive effect. The oxide layers derived from anode oxidation at 9.5 V vs. Ag/AgCl electrode in an aqueous calcium nitrate solution behaved similarly, i.e., subsequent heating was effective to stimulate nucleation of apatite. More significant enhancement was achieved by additional cathode polarization at -2.0 V vs. Ag/AgCl followed by the anode oxidation. The cathode polarization deposited calcium ions as calcium hydroxide. Calcium carbonate was yielded from the hydroxide when soaked in the simulated body fluid, and provided the sites of apatite nucleation as well as it was exhausted by the formation of apatite.

KEYWORDS

Titanium, biomimetic deposition, apatite, hydrated titania gel, H₂O₂ treatment, anode oxidation, mechanism of apatite deposition.

COMPARISON OF CHEMICAL SOLUTION DEPOSITION SYSTEMS FOR THE FABRICATION OF LEAD ZIRCONATE TITANATE THIN FILMS

FREDERIC LECARPENTIER, MARC DAGLISH and TIM KEMMITT

Industrial Research Ltd, PO Box 31-310, Lower Hutt, New Zealand

ABSTRACT

Ferroelectric thin films of lead zirconate titanate $\text{Pb}(\text{Zr}_x\text{Ti}_{1-x})\text{O}_3$ (PZT) were prepared from five chemical solution deposition (CSD) systems, namely methoxyethanol, citrate, diol, acetic acid and triethanolamine. Physical characteristics of the solutions, processing parameters and physical and electrical properties of the films were used to assess the relative advantages and disadvantages of the different chemical systems. All the CSD systems decomposed to produce single phase perovskite PZT at temperatures above 650°C. Thin film deposition was influenced by the specific characteristics of each system such as wetting on the substrate and viscosity. Distinct precursor effects on the thin film crystallinity and electrical performance were revealed. The diol route yielded films with the highest crystallite size, highest permittivity and lowest loss tangent. The relative permittivities exhibited by films made by the other routes were 25% to 35% lower at equivalent thicknesses.

KEYWORDS

Chemical solution deposition, thin film, PZT.

PREPARATION AND CHARACTERIZATION OF SOL-GEL DERIVED TiO₂ FILMS

Y. J. HONG¹, M. P. BRUNGS¹, R. P. CHAPLIN¹ and E. SIZGEK²

*¹School of Chemical Engineering and Industrial Chemistry, University of New South Wales,
Sydney, 2052, Australia*

*²Materials Division, Australian Nuclear Science & Technology Organisation, New Illawarra Road,
Lucas Height, NSW 2234, Australia*

ABSTRACT

Crack-free transparent titania films have been prepared through a new sol-gel process combined with hydrogen peroxide treatment. Hydrogen peroxide and HCl were used to dissolve amorphous titania powder which is obtained by drying common sol-gel titania sol. The peroxy-titania sol produced a thicker film than the common sol-gel titania sol due to a higher degree of condensation. Film thickness could be further increased by controlled drying conditions of the amorphous titania powder or "aging" the sol. Polyethylene glycol (PEG) and chemical additives were effective in controlling condensation rate by preventing rapid condensation during curing of the film. When these two components were incorporated, it was possible to create a 0.5µm transparent film and PEG could also control the porosity of the film.

KEYWORDS

Transparent thick TiO₂ film, sol-gel process, hydrogen peroxide, crack resistance.

STUDY OF REACTIVE MAGNETRON SPUTTERED TITANIUM DIOXIDE FILMS FOR USE IN THE GRATZEL PHOTOELECTROCHEMICAL CELL

M. G. WHEATLEY^{1*}, M. P. BRUNGS¹, R. P. CHAPLIN¹ and E. SIZGEK²

¹*School of Chemical Engineering and Industrial Chemistry University of New South Wales 2052,
Sydney, Australia*

²*Materials Division, Australian Nuclear Science & Technology Organization, New Illawarra Road,
Lucas Heights, NSW 2234, Australia*

**email: michaelw@engineer.com*

ABSTRACT

The Gratzel photoelectrochemical cell is a relatively new device that has the advantage of being significantly cheaper to produce than a bulk silicon solar cell. This paper examines sputtered titanium dioxide Gratzel devices specifically to discuss the practical implementation of such a device. In addition to the sputtered titanium dioxide coating, an electrical model is presented to aid in the design of components for a complete photovoltaic power system. The Gratzel cell exhibits a significant capacitance that can cause misleading measurements when tested on conventional equipment.

The capacitance and the reverse bias conditions are presented as an electrical model for use in circuit simulators such as SPICE or MICROCAP.

KEYWORDS

Gratzel cell, modelling, titanium dioxide, sputtering, photoelectrochemical.

J. Aust. Ceram. Soc., [37], 1, 2001, pages 35-38

MODELLING OF CREEP IN THE BLADE-FIT AREA OF A TURBINE ROTOR

M. LAW, P. BENDEICH and W. PAYTEN

Australian Nuclear Science and Technology Organisation (ANSTO)
email: mlx@ansto.gov.au

ABSTRACT

Where creep and contact occur in a high temperature rotor, finite element analysis is required to model the material and geometric non-linearities present.

KEYWORDS

Finite Element Analysis, creep, contact, high temperature, rotor.

EROSION BEHAVIOUR OF ADVANCED CERAMICS

Y. ZHANG¹, Y.-B. CHENG¹ and S. LATHABAI²

¹*Department of Materials Engineering, Monash University, Clayton, Victoria 3800, Australia*

²*CSIRO Manufacturing Science and Technology, Woodville, South Australia 5011, Australia*

ABSTRACT

Three kinds of advanced ceramics, namely alumina, zirconia, and sialon, were subjected to airborne erosion tests using garnet and SiC erodents. The effect of angle of impact on the erosion rate of these materials has been studied. A marked difference in erosion rate corresponding to shallow angle and normal impact was observed. Such a difference could be accounted for in terms of the erosion mechanism in the light of SEM micrographs of eroded surfaces. The effect of erodent particle properties on the erosion rate of these materials has also been examined. A large difference in erosion rate corresponding to various erodent materials was observed. Such a difference could be attributed to the variations in the shape, as well as the hardness and toughness of the erodent materials. The erosive wear behaviours of these ceramics were also different. Such differences could be explained by the various microstructures of these materials.

KEYWORDS

Airborne erosion; advanced ceramics; erosion rate; impact angle; erodent properties; microstructure; erosion mechanism.

PREPARATION OF Mg- α SiAlON POWDER BY CARBOTHERMAL REDUCTION-NITRIDATION OF TALC AND HALLOYSITE

J. Y. QIU¹, C. ZHANG¹, K. KOMEYA^{1*}, T. MEGURO¹, J. TATAMI¹ and Y.-B. CHENG²

¹*Graduate School of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya-Ku, Yokohama-shi 240-8501, Japan*

²*Department of Materials Engineering, Monash University, Clayton, Melbourne, Victoria 3168, Australia*

**email: komeya@ynu.ac.jp*

ABSTRACT

Carbothermal reduction-nitridation (CRN) of talc ($\text{Mg}_3(\text{Si}_2\text{O}_5)_2(\text{OH})_2$) and halloysite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) clay provides a useful route for preparing low-cost Mg- α sialon powder. In this study, the chosen molecular ratios of talc to halloysite were 0.1:1, 0.2:1, 1.5:1.0 and 2.0:1. The CRN reaction was conducted at 1450 to 1520°C and 2 to 6h holding time using carbon black as a reducing agent in flowing N_2 (gas). The results showed the synthesized powder was composed of α -sialon, β -sialon and small amounts of SiC, 15R and AlN phases that greatly depended on the ratio of talc to halloysite, the reaction temperature and holding time. The highest content of Mg- α sialon, as much as 90wt%, was achieved at 1480°C for a holding time of 4h at a talc to halloysite ratio of 1.5:1.0. SiC was considered as an intermediate compound. We also discuss the evaporation loss of Mg caused by talc decomposing into MgSiN_2 intermediate phase in the CRN reaction.

KEYWORDS

Mg- α sialon, halloysite, talc, carbothermal reduction-nitridation.

PREPARATION OF SOL-GEL DERIVED BaTi(Hf,Zr)O₃ THIN FILMS AND THEIR PROPERTIES

J. THONGRUENG¹, K. NISHIO¹, Y. WATANABE¹, K. NAGATA² and T. TSUCHIYA¹

¹*Department of Materials Science and Technology, Science University of Tokyo, 2641, Yamazaki, Noda 278-8510, Japan*

²*Department of Electronic Engineering, National Defense Academy, 1-10-20, Hashirimizu, Yokosuka 239-8686, Japan*
email: tjforce@hotmail.com

ABSTRACT

Sol-gel derived BaTi_{0.91}(Hf_{0.5}Zr_{0.5})_{0.09}O₃ (BTHZ-9) thin films were successfully prepared onto Pt substrate by spin-coating technique and sintered at various temperatures in range of 650 to 950°C. Barium acetate, titanium(IV)tetrabutoxide monomer, zirconium(IV) propoxide and hafnium oxychloride octahydrate were used as raw materials. Acetic acid, 2-methoxyethanol and water were used as solvents. Acetylacetone as a chelating agent to increase stability of BTHZ sol against ambient humidity and solution aging with time was used. Microstructure and electrical properties were investigated. The grain size of the films ranged from 70 to 200nm, depending on the sintering temperature, from 650 to 850°C. BTHZ-9 with rhombohedral phase at room temperature exhibited well-saturated *P-E* hysteresis loop. The Curie point of the BTHZ-9 films was determined to be 87°C.

KEYWORDS

BaTiO₃, BTHZ-9, sol-gel, coercive field, remanent polarization, thin film, hysteresis loop, ferroelectric.

INHOMOGENEOUS GRAIN GROWTH AND ELONGATION OF Dy- α SiAlON CERAMICS

C. ZHANG, K. KOMEYA*, J. TATAMI and T. MEGURO

*Graduate School of Engineering, Yokohama National University,
79-5 Tokiwadai, Hodogayaku, Yokohama 240-8501, Japan
email: komeya@ynu.ac.jp

ABSTRACT

Dysprosium α -sialon ceramic has been fabricated from α -Si₃N₄, AlN, Al₂O₃ and Dy₂O₃ starting powders, using gas pressure sintering at 1800°C or higher temperatures under a relatively low gas pressure of 0.9MPa N₂. The resultant α -sialon grains showed significantly different features, such as equiaxed, elongated, and even a few extraordinarily large with high aspect ratio grains in the fine matrix, which has rarely been observed in α' ceramics. It is suggested that the temperature strongly influences the grain morphologies of α -sialon, playing an important role particularly in the latter stage of the “nucleation-growth” process. Such a microstructural morphology of mixed with equiaxed and elongated grains toughens the α -sialon ceramics and leaves them still hard. Overly high temperature treatment leads to the preferential growth of a few α' grains which become excessively large and long, but does not significantly improve the material's toughness.

KEYWORDS

α -sialon, sintering, GPS, grain size, elongation, mechanical properties.

A STUDY ON THE CHARACTERISATION AND BIOSTABILITY OF USED AND VIRGIN DIALYSIS MEMBRANES AND BIOCOMPATIBILITY OF THE COMPOSITE BIOMATERIALS

B. A. KONDUK and A. H. UCISIK

*Bogaziçi University, Department of Protheses, Materials and Artificial Organs,
Institute of Biomedical Engineering, 80815 Bebek-Istanbul, Turkey*

ABSTRACT

In this study two composite systems consisted of polymers and ceramics respectively were investigated in terms of biocompatibility and biostability simultaneously. The selected polymers were polysulfone and cellulose acetate. Morphology, supramolecular and molecular structure, and mechanical properties of the virgin polymeric membranes used in hemodialysis and their variations after use were analyzed with respect to degradation and aging due to corrosion provoked by blood and by surrounding aggressive environment. The wall thicknesses and diameters of the polymeric hollow fibers were measured under a microscope. These measurements were entered to run mechanical tests. In virgin and used hollow fibers belonging to polysulfone and cellulose acetate, the morphology of the support structure and geometry of the pores together with fracture surfaces have been comparatively evaluated by SEM and AFM studies. Mechanical studies have revealed that ultimate tensile strength, strain to fracture and yield strength values have been reduced after blood-contact. These results coupled with fractographic evidences prove degradation and ductile to brittle transition after use. X-ray diffraction studies implied that internal, supramolecular structure of the hollow fibers altered in a way that the strong bonding of crystalline and amorphous regions into a composite structure giving rise to good mechanical properties has been impaired after use. The results were compared in an attempt to find out alternative methods to improve the quality of the membranes. On the other hand, alumina-zircon composites were investigated in parallel to the polymer studies. The alumina used had a purity of 99.7 % with 0.1 % Na₂O by weight and dimensions of 7 mm diameter and 2 mm thickness. After sintering, the density of the alumina-zircon ranged from 3.2 to 3.6 g/cm³ respectively. Tissue reactions of test materials were performed using rats for 2 months duration. X-ray diffraction studies showed that alumina exists only in the form of corundum. As the amount of zircon increased, mullite formations became visible on the particle boundaries. Animal studies revealed that these ceramic composites do not have any adverse effect on the tissues investigated histologically. Herein it is postulated that composite systems consisting of basic polymers either, polysulfone or cellulose, and additives (ceramic) may improve the biostability and biocompatibility of the membranes used for hemodialysis or their respective degraded products as explained to different degrees after use, or may serve as new biomaterials. Hence, this study is partly aiming at improving the quality of membranes and formation of new biomaterials for biomedical applications. In future polymers mentioned such as cellulosic or synthetic ones reinforced with ceramics may create new benefits in the area of nephrology, whereas alumina-zircon reinforced by polymers may do the same in dentistry and orthopedics.

KEYWORDS

Hemodialysis, dialysis membrane, polysulfone, hemophan, cuprophan, cellulose acetate, polymer, composite, biostability, biocompatibility, reuse, bioceramic.

BORIDE COATING ON IRON BASED ALLOYS

A. H. UCISIK^{1*}, S. ZEYTIN² and C. BINDAL²

¹*Bogaziçi University, Department of Protheses, Materials and Artificial Organs, Institute of Biomedical Engineering, 80815 Bebek-Istanbul, Turkey*

²*Sakarya University, Engineering Faculty, Department of Metallurgical and Materials Engineering, 54187Esentepe Campus, Sakarya, Turkey*

**email: ucisik@hotmail.com*

ABSTRACT

Surface hardening treatments (i.e. carburizing, nitriding) by diffusion of interstitial elements such as carbon and nitrogen have been applied for a long time in industry. While studies on carburizing have decreased considerably, nitriding treatment is still widely used, developing in different ways such as plasma nitriding. However, surface treatment by boron diffusion has been interest to many researchers since the 70's. Boriding can be applied to most steels, cast iron, titanium and nickel alloys. Boriding, like carburizing and nitriding, has been applied to improve wear resistance of substrate materials due to its high hardness. Borides formed on plain carbon steel have columnar-needle shaped morphology, while addition of alloying elements results in smooth, compact or monolithic morphology. As the amount of carbon and alloying elements increase, the growth rate of the layer decreases because of difficulty of diffusion. However, hardness of the layer increases due to formation of complex borides. During boriding, two distinct regions form, which are boride rich surface layer and a transition zone from surface to inner regions. The hardness of the transition zone, an iron-boron solid solution, is higher than that of the matrix, but lower than that of the surface layer which has a hardness of 1500 HV. The fracture toughness of the surface boride layer formed on steel can be compared to those of many ceramic materials e.g. Si₃N₄ and Al₂O₃. The friction coefficient of the boride layer formed on briefly annealed low alloy steels drops to 0.03-0.05 because of formation of a slippery layer, whereas it is about 0.4-0.5 in the unannealed condition. This result promises that this kind of coating can be utilized in many engineering areas.

KEYWORDS

Boride coating, fracture toughness, hardness, steel.

INDENTATION RESPONSES, WEAR AND DAMAGE CHARACTERISTICS OF HOT-ISOSTATICALLY-PRESSED Ti₃SiC₂

Y. KURODA¹, I. M. LOW¹, M. W. BARSOUM² and T. EL-RAGHY²

¹*Materials Research Group, Department of Applied Physics, Curtin University of Technology,
GPO Box U1987, Perth, WA, 6845, Australia*

²*Department of Materials Engineering, Drexel University, Philadelphia PA 19104 U.S.A.*

ABSTRACT

The influence of grain size on the indentation responses, wear and damage characteristics of hot-isostatically pressed Ti₃SiC₂ (312) ceramics have been investigated. "Bonded-interface" sections are used to characterise the degree of subsurface damage accumulation from Vickers contacts, wear and drilling. When compared to coarse-grained (CG) 312, fine-grained (FG) samples are harder, more difficult to machine, and have better resistance to contact wear and damage. However, the load-dependent hardness or indentation size effect is observed for both FG and CG samples when measured at 80 and 300 K. In contrast to FG 312, the CG sample exhibits a pronounced shear deformation during indentation at both 80 and 300 K, indicating microscale dislocation-induced plasticity which can be associated with intragrain *multiple* basal-plane slip between micro-lamellae, intergrain sliding, lamellae or grain push-out, and microfailures at the ends of the constrained shear-slips.

KEYWORDS

Ti₃SiC₂, indentation responses, damage, bonded-interface, dislocations, grain-size, wear.

UNDERSTANDING FACTORS INFLUENCING THE PHASE DEVELOPMENTS IN HYDROTHERMALLY CURED CEMENT-BASED SYSTEMS: AN OVERVIEW

D. S. KLIMESCH

*Centre for Built Infrastructure Research, Faculty of Engineering, University of Technology, Sydney,
P.O. Box 123 Broadway, Sydney, NSW 200 7, Australia*

ABSTRACT

The hardening of concrete as well as other materials based on calcium silicates is accelerated by the use of saturated steam under pressure in an autoclave. The principal strength-producing phase in autoclaved calcium silicate based materials is 1.1nm tobermorite (tobermorite). Due to tobermorite's significance, there is great interest in optimising the conversion of tobermorite from C-S-H. This article provides an updated and integrated overview of the current understanding of factors influencing the phase developments pertaining to hydrothermally cured cement-based systems.

KEYWORDS

Hydrothermal curing, 1.1nm tobermorite, hydrogarnet, C-S-H, Al-substitution, gibbsite, metakaolin, quartz fineness.

CONCRETE RESEARCH USING BLENDED CEMENTS

W. B. BUTLER

*Ash Development Association of Australia, 117 Marmong Street, Marmong Point,
NSW, 2284, Australia*

ABSTRACT

Concrete research increasingly involves the use of mixes containing one or more of the supplementary cementitious materials (SCMs), often in conjunction with chemical admixtures. The influence of materials is commonly evaluated on the basis of water/ cement or water/ binder ratio and SCM content as a percentage of total binder, with dosage level of chemical admixture varied to maintain workability. As a result, more than one variable is introduced at a time and the objectives of the research may not be achieved. The significance of water/ cement ratio and addition rates of admixtures are examined from a practical standpoint with suggestions for more appropriate means of evaluation of the influence of individual materials.

KEYWORDS

Supplementary cementitious materials, water-cement ratio, total free water content.

MICROSTRUCTURE AND PROPERTIES OF ALKALI-ACTIVATED SLAG CONCRETE

T. BAKHAREV¹, J. G. SANJAYAN¹ and Y. B. CHENG²

¹Department of Civil Engineering

²Department of Materials Engineering

Monash University, Victoria 3168, Australia

ABSTRACT

The microstructure of alkali activated slag (AAS) concretes was investigated using scanning electron microscopy. AAS concretes prepared using different activators and cured at room and elevated temperatures were examined by SEM, using backscattered electron imaging. In particular, the microstructures of basic paste and paste-aggregate bond were studied. The effect of type of activator and curing temperature on the concrete microstructure was investigated and related to concrete properties. The microstructure of AAS concrete was also compared with that of OPC concrete.

KEYWORDS

Alkali activated slag, microstructure, SEM, interfacial transition zone.

CAESIUM DIFFUSIVITY THROUGH CEMENTED CLINOPTILOLITE

V. K. PETERSON^{1*}, A. RAY¹ and L. P. ALDRIDGE²

¹*Department of Chemistry, Materials and Forensic Science, University of Technology Sydney,
PO Box 123, Broadway, NSW, 2007, Australia*

²*Australian Nuclear Science and Technology Organisation, PMB 1, Menai, NSW, 2234, Australia
email: vkp@ansto.gov.au

ABSTRACT

Cs⁺ diffusion through ordinary Portland cement (OPC) pastes was estimated from the rate at which Cs⁺ diffused through a slice of cement paste. The intrinsic diffusivity was calculated using modified diffusion equations. The diffusion of Cs⁺ through OPC pastes was compared with that through pastes containing the zeolite clinoptilolite. The zeolite binds the Cs⁺ ions and thus would be expected to decrease the diffusivity of Cs⁺. The diffusion of Cs⁺ in pastes cured at temperatures of 25, 40, and 60°C was compared. Cs⁺ diffusivity was found to increase with increasing cure temperature. The addition of clinoptilolite to OPC was found to reduce Cs⁺ diffusivity through the sample.

KEYWORDS

Cement, caesium, zeolite, clinoptilolite, wasteform, diffusion, nuclear technology.

J. Aust. Ceram. Soc., [37], 1, 2001, pages 127-129

**CEMENT WORKSHOP AUSTCERAM 2000
HELD ON TUESDAY, JUNE 27, 2000
AT THE UNIVERSITY OF TECHNOLOGY, SYDNEY**

L. ALDRIDGE, D. BAWERA, H. BERSTEN, B. BUTLER,
R. KHATRI, B. POTTER, A. RAY and W. SOUTH

ABSTRACT

This report summarises the workshop held on Tuesday afternoon at the end of the cement section. The workshop organisers prepared the report.

KEYWORDS

Cement, durability, industry-research interaction, greenhouse gas.