

## **CHEMICAL COMPATIBILITY OF RARE EARTH COBALTITE PEROVSKITES WITH YSZ**

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### **ABSTRACT**

Rare earth cobaltite perovskites are interesting cathode materials for the reduced temperature SOFC because of their high catalytic activity for oxygen reduction. The Chemical compatibility of perovskites in  $\text{Ln}_{1-x}\text{Sr}_x\text{CoO}_{3-\delta}$  (Ln=Sm, Dy),  $\text{Ln}_{0.4}\text{Sr}_{0.6}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_{3-\delta}$  (Ln=La, Pr, Nd, Sm, Gd),  $\text{Gd}_{0.8}\text{Ca}_{0.2}\text{Co}_{1-x}\text{Mn}_x\text{O}_{3-\delta}$  with 8 mol% yttria stabilized zirconia (8YSZ) has been investigated. Powder mixtures of these perovskites and 8YSZ have been annealed at different temperatures for 96h in air. As the main reaction product,  $\text{SrZrO}_3$  has been found in 8YSZ/ $\text{Ln}_{1-x}\text{Sr}_x\text{CoO}_{3-\delta}$  (Ln=Sm, Dy) with high Sr content and 8YSZ/ $\text{Ln}_{0.4}\text{Sr}_{0.6}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_{3-\delta}$  (Ln=La, Pr, Nd, Sm, Gd) at 900°C. No reaction product has been detected in 8YSZ/ $\text{Gd}_{0.8}\text{Ca}_{0.2}\text{Co}_{1-x}\text{Mn}_x\text{O}_{3-\delta}$  by XRD. However, significant diffusion of Co into 8YSZ has been found by EDX at the interface of 8YSZ/ $\text{Gd}_{0.8}\text{Ca}_{0.2}\text{Co}_{0.6}\text{Mn}_{0.4}\text{O}_{3-\delta}$  after annealing at 1200°C for 24h. The bond-valence model has been used to discuss the chemical compatibility of the different perovskites with 8YSZ.

### **KEYWORDS**

SOFC, rare earth cobaltite, perovskite, bond-valence model.

## **THE FORMATION OF ZIRCONIUM HYDROXIDE NANOPARTICLES FROM AQUEOUS NITRATE SOLUTIONS**

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### **ABSTRACT**

Concentrated zirconia nano-sols have been prepared by dissolving zirconium oxycarbonate in an acidic solution of zirconyl nitrate. Hydrolysis and condensation of polynuclear 'zirconyl' cations promotes the formation of nano-sized, polymeric, oxy-hydroxide particles. The evolution of the nanoparticle structure during the formation of the sol has been investigated with a range of complementary characterisation techniques. Dynamic light scattering indicates that the vast majority of particles are of size 3-6 nm. The Raman spectra of the zirconyl nitrate, and the sol, have been assigned with the assistance of vibrational-modelling software. The characteristic Raman peaks of the polynuclear 'zirconyl' cations at 450 and 575  $\text{cm}^{-1}$  shift to 375 and 535  $\text{cm}^{-1}$ , respectively, as the particles are formed, indicating the condensation of terminal-hydroxy groups to form hydroxy bridges. <sup>14</sup>N NMR and Raman spectroscopy show that all of the unassociated nitrate groups present in the precursor solution become weakly associated with the surface of the particles, but are not directly coordinated to the zirconium atoms.

### **KEYWORDS**

Zirconium hydroxide, colloids, sol-gel, Raman spectroscopy, NMR.

## **ANALYSIS ON THE PARTICLE GROWTH IN SILANE PCVD REACTOR FOR SEMICONDUCTOR PROCESSING**

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### **ABSTRACT**

The particle growth by coagulation was investigated theoretically in bulk plasma region of plasma CVD reactor. The predator particles in bulk plasma region grow by the coagulation with small protoparticles and the model equations for the particle size and particle charge distribution were solved for various plasma conditions. Most of the protoparticles in plasma reactor were charged negatively, but a few percent of the particles were neutral or positively charged and those particles were found to be very active for particle growth by coagulation. As the time increases, the predator particle sizes and the average charges on predator particles increase and the coagulation coefficients between predator particles and protoparticles increase.

### **KEYWORDS**

Protoparticles, predator particles, Gaussian distribution, particle charge distribution, particle growth model, plasma conditions.

## **AESTHETICAL DURABILITY AND DESIGNING PRODUCTS MADE BY WASTE MATERIALS**

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### **ABSTRACT**

This study was preceded by a study about using waste materials like sewage sludge, fly ash and recycled glass as a partial raw material in bricks and tiles.

Aesthetic values have often been given the lowest priority in practical construction work, not necessarily in planning. However, I believe that the aesthetic values of our environment will be increasingly important in the future, as the material well being can not be endlessly increased, but we need to find new values for our living, housing and activities instead of the materialistic criteria.

This presentation will discuss the aesthetic durability of ceramic building materials using waste as a partial raw material. What is aesthetical durability and how can it be examined? Different materials age and wear differently; the aging process of brick, stone and wood is found acceptable, even beautiful, whereas e.g. worn concrete or corrugated iron do not generally correspond to our image of a beautiful building material.

In the future, many countries may have to reevaluate their waste management and seriously consider tasks, to reduce the production of waste and to make recycling of all produced waste materials possible. At this point it is important to realize that it is not unimportant what kind of materials and products we develop from waste materials. If we develop an ugly environment by using waste materials the waste is not really utilized, but it has only changed form.

## **THERMAL EVOLUTION OF MULLITE FROM GEL PRECURSORS STUDIED BY MASS SPECTROMETRY AND MAS NMR**

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### **INTRODUCTION**

Mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ ) has long been a compound of technical importance, being the principal phase formed when clay minerals are heated. More recently, the development of sol-gel hybrid (organic-inorganic) synthesis methods has opened the way for the production of high-purity mullite of controllable grain size, which has excellent potential in high-technology engineering applications. The finished properties of gel-derived mullite depend on the homogeneity of the gel, which is strongly influenced by the starting materials and reaction conditions. A less-understood aspect of the evolution of crystalline mullite from the gel precursor is the effect of the thermal pre-treatment which can lead to the formation of an Al species with a characteristic MAS NMR resonance at about 30 ppm. The role of this species, which is often ascribed to 5-coordinated Al, is also not well understood. This paper reports an investigation into the relationship between the thermal pre-treatment regime of mullite precursor gels and the appearance of the 30 ppm Al MAS NMR signal as mullite crystallization proceeds.

### **KEYWORDS**

Mullite, sol-gel synthesis, MAS NMR, evolved gas analysis.

## **A SOLID STATE MAS NMR STUDY OF THE THERMAL REACTIONS IN ALKALI-LEACHED ALUMINOSILICATES**

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### **INTRODUCTION**

The thermal transformations of aluminosilicate minerals such as kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ) are of importance for the production of both clay-based ceramics and high-technology ceramics such as sialons. Solid-state MAS NMR can provide information about the intermediate stages in the formation of mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ ). These intermediates, which are only poorly crystalline and less amenable to XRD study, may include poorly crystalline mullite, a cubic spinel similar to  $\gamma\text{-Al}_2\text{O}_3$  but which has been suggested to contain Si, and other amorphous aluminosilicate phases of variable composition. Since the  $^{29}\text{Si}$  and  $^{27}\text{Al}$  MAS NMR spectra of all these phases are expected to contain resonances broadly in the same spectral area, unambiguous differentiation of these phases has not so far proved possible by this technique. The work reported here was suggested by the possibility of selective alkali extraction of some of the more silica-rich phases using techniques developed by Chakravorty and Ghosh, which was hoped to reveal the MAS NMR features of the less-leachable phases. NMR study of the leached products after subsequent thermal treatment also provided useful information about the leaching reactions themselves.

### **KEYWORDS**

Alkali leaching, aluminosilicates, MAS NMR.

## **SOLID STATE MAS NMR STUDIES OF RARE EARTH MELILITES**

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### **INTRODUCTION**

Melilites are a group of minerals whose end members are akermanite ( $\text{Ca}_2\text{MgSi}_2\text{O}_7$ ) and gehlenite ( $\text{Ca}_2\text{Al}_2\text{SiO}_7$ ). The structure, which consists of sheets containing  $\text{Si}_2\text{O}_7$  double tetrahedra linked by polyhedra, is versatile, and is adopted by a number of other compounds including rare earth silicon oxynitrides of which the best known is N-melilite,  $\text{Y}_2\text{Si}_3\text{N}_4\text{O}_3$ . This is one of the intergranular phases formed during processing of nitride ceramics with  $\text{Y}_2\text{O}_3$  sintering additive. The structure of N-melilite, in which the Ca, Mg and four of the seven oxygens in akermanite are replaced by Y, Si and N respectively, has recently been confirmed by a Rietveld refinement of X-ray powder data.

Substitution of Al for Si can also occur in the melilite structure, with charge balance accommodated by changes in the N-for-O substitution resulting in the general formula  $\text{Y}_2\text{Si}_{3-x}\text{Al}_x\text{O}_{3+x}\text{N}_{4-x}$ . Al substitution has been suggested to occur preferentially in the corner and central  $\text{MO}_4$  tetrahedra which link the  $\text{Si}_2\text{O}_7$  units, but the possible substitution of additional Al into other sites cannot be excluded. MAS NMR spectroscopy has provided a certain amount of structural information about the melilites, including analogues containing other rare earth elements substituted for Y.

The present paper reports the  $^{27}\text{Al}$  and  $^{29}\text{Si}$  MAS NMR spectra of a more complete series of these compounds, both without Al substitution and with Al substituted up to the maximum amount, which varies from compound to compound.

### **KEYWORDS**

Rare earth melilites, MAS NMR.

## **CEMENT WORKSHOP AT AUSTCERAM 98**

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### **ABSTRACT**

The Cement Section of AUSTCERAM 98 was concluded with a workshop, which was run by the organising committee, Laurie Aldridge (Materials Division, ANSTO, Menai, NSW), Tom Mason (Department of Materials Science and Engineering, Center for Advanced Cement-Based Materials, Evanston, IL 60208 USA), Neil Milestone (New Zealand Institute for Industrial Research, Lower Hutt, New Zealand), Abhi Ray (University of Technology, Sydney NSW), Kathryn Turner (Blue Circle Southern Wentworthville, NSW). The committee wrote this summary of a stimulating and enlightening afternoon that all participants enjoyed and contributed to.

The aim of the workshop was to bring together workers from industry, university, and government laboratory to stimulate interaction and collaboration from industry and science.

The first part was an overview of new techniques that could be applied to determining the microstructure and performance of cementitious materials.

The second part of the workshop consisted of an animated discussion on the needs and opportunities in cement and concrete in present day Australia.

## **POSSIBILITIES OF MINERALISING CEMENT CLINKER USING ALUMINA INDUSTRY WASTE**

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### **ABSTRACT**

Modern cement companies are required to investigate solutions to lower energy consumption and limit greenhouse gas emissions. Alternate strategies involve the increased use of blended cements or the use of materials that lower the temperature in the cement clinker kiln.

This paper reports the preliminary findings of an investigation into the use of two waste products from the alumina industry. Both these materials were selected because of their fluorine composition and their fuel value. The mineralising ability was compared by measuring the amount of free lime left in the solid after heating the mineraliser at predetermined temperatures with a raw mix. This mineralising ability was compared with pure calcium fluoride (a well known mineraliser).

The results showed that, when used in sufficient quantities, these materials can lower the free lime content of a given raw mix at elevated temperatures. However, this level of addition is considered to have an adverse effect on the performance of cement ground using this clinker. It is therefore recommended that these materials be considered for use in combination with other materials as a supplementary fuel.

## **ALKALIS IN CONCRETE: WHERE DO THEY COME FROM AND WHAT EFFECT DO THEY HAVE?**

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### **ABSTRACT**

One of the features of using volcanic aggregates in concrete for countries such as New Zealand and Japan is that if the alkali content of the concrete pore solution is high, these aggregates can react with those alkalis. The reaction product is an alkali silicate gel, which can cause expansive cracking - the phenomenon better known as AAR.

Where do these alkalis come from? Cement is one obvious source but alkalis are also contributed by admixtures, mixing water, and salt and, as we have recently found, large amounts can be released by certain aggregates.

What can we do about alkalis in concrete? We can use non-reactive aggregates, although which aggregates are not reactive? We can use a low alkali cement but what constitutes a low alkali cement? We can use a pozzolan but is that sufficient to control all the alkalis available in the pore solution?

We will attempt to answer some of these questions with examples from field evidence in New Zealand structures.

## **EVALUATION OF PHASES IN HYDROTHERMALLY CURED CaO-SiO<sub>2</sub>-H<sub>2</sub>O AND CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-H<sub>2</sub>O SYSTEMS**

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### **ABSTRACT**

Thermal analysis, DTA-TGA, in combination with other techniques such as XRD offers a powerful means for evaluation of phases in the hydrothermally treated CaO-SiO<sub>2</sub>-H<sub>2</sub>O and CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-H<sub>2</sub>O systems. Interpretation of thermal data, however, can be a highly complex exercise. This paper emphasizes the usefulness of DTA-TGA in the evaluation of critical phases formed under hydrothermal conditions.

## **DETERMINATION OF CHLORIDE INGRESS RATE FOR REINFORCED CONCRETE DESIGN LIFE PREDICTION**

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### **INTRODUCTION**

The New Zealand Building Code has a durability performance requirement based on a design life of 50 years for structural building components. The Concrete Structures Standard gives guidelines for designers of reinforced concrete structures based on providing adequate protection to the reinforcing steel for various exposure zones. For the three zones nearest the coastline, the tidal/splash zone, the coastal frontage zone, and the coastal perimeter zone, ingress of marine chlorides is the controlling criteria, whilst for the inland zone and interior exposure carbonation of the concrete is the controlling criteria. The normal benign environment around the reinforcing bar resulting from the concrete alkalinity can be compromised either by the presence of chlorides or by carbonation.

Protection of the reinforcement to ensure that corrosion does not occur within the 50 year design life is based on specifying an adequate quality and thickness of cover concrete. The standard's guidelines suppose the use of General Purpose (GP) concrete with various combinations of specified compressive strength and cover depth. For the most severe marine exposure conditions for example a strength above 50 MPa achieved with a w/c ratio < 0.40, combined with a minimum cover of 70 mm is required.

Within the last decade, the increasing adoption of blended cements containing supplementary cementitious materials (SCMs) such as flyash, blast furnace slag and silica fume has underlined the limitations in this approach. Both laboratory testing and field performance have clearly demonstrated that the judicious use of these materials significantly enhances resistance to chloride ingress. Whilst the current standard recognises this, it offers no guidelines as to how this potential benefit can be most effectively realised. Thus questions which often arise include "What concrete should I specify for a 100-year design life?" or "If I use a pozzolanic material can the necessary cover or possibly strength be reduced?"

## **DIFFUSION OF IONS THROUGH CEMENT PASTE**

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### **INTRODUCTION**

Most methods used to calculate diffusion and leaching of radionuclides through cement assume that sorption of the ions from the pore water into the cement matrix takes place instantaneously. This assumption may not be appropriate for certain ion species, which are known to react slowly with the cement matrix. In this paper we present a modified mathematical model for the diffusion of ions through cement where sorption processes do not necessarily take place instantaneously but are governed by a rate parameter. It is shown that if the sorption rate is of the order of, or less than, the intrinsic diffusion rate, the diffusion and leaching rates can differ significantly from those calculated using the standard theory. Experimental results are presented which provide what appears to be direct evidence of a slow sorption process.

## **THOUGHTS ON A DURABILITY PREDICTION METHODOLOGY FOR CONCRETE IN THE MARINE ENVIRONMENT**

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### **INTRODUCTION**

Steel reinforced concrete is widely used for structures in, or in close proximity to, maritime environments in New Zealand. Under normal conditions the natural alkalinity of the concrete provides a protective oxide layer around the steel which guards against corrosion. However, if chloride ions (e.g. from salt-spray aerosols) penetrate the cover concrete to the depth of the reinforcement depassivation occurs, allowing corrosion to proceed. Thus an important facet of predicting the durability of reinforced concrete structures is being able to model the rate of ingress of chloride ions for various concrete qualities and exposure conditions.

Chloride ions can penetrate the steel by a number of mechanisms including diffusion (movement under a concentration gradient), permeation (movement by hydrostatic pressure) and absorption (capillary uptake). An ideal model of chloride penetration should include all these factors to define the chloride concentration in the pore solutions of the concrete. Whilst a number of models have been published which include at least some of these features, the detailed information necessary to accurately model moisture movement precludes their general usefulness as a tool for service life prediction.

## **CONCRETE DURABILITY HOW TO MAKE A REACTIVE MATERIAL LAST A LIFETIME**

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### **ABSTRACT**

Well formulated mix designs and on site practices can allow the placement of concrete which develops high strength. However, high compressive strength does not always imply good concrete durability. Concrete is a reactive chemical composite, which under the effects of certain conditions, can have a less than expected lifetime. Environmental effects can cause chemical attack on the outside of the concrete opening the structure to degradation. Examples are acid attack and sulphate attack. Chemical reactions can also occur within the hardened structure of concrete. One of the most common of these is alkali aggregate reaction, AAR, but unsoundness and other hydration effects can also cause reactions which lead to cracking and ultimate corrosion of reinforcement.

These reactions must be taken into account as engineers design concrete for use in demanding conditions where the demands on the concrete are pushed closer to the ultimate limits of the cement binder properties. Some of these chemical reactions and their effects on durability will be discussed using examples from New Zealand situations.

## **CAN CONCRETE BE MADE BETTER?**

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### **INTRODUCTION**

Today's constituents of concrete binders are remarkably similar to that of concrete produced in early Roman times. The improvement of concrete performance has resulted from increased control of material properties and production techniques, but the question is still asked, 'Can concrete be made better?'

There are many participants involved in the production of concrete and each has requirements with respect to the final concrete product. The engineer and architect must consider the application and performance viability of the concrete. The concrete owner is concerned with the concrete cost in addition to performance properties. The concrete manufacture must produce a cost effective product which achieves performance objectives and can be suitably placed by the builder.

An understanding of key aspects has enabled concrete producers to control concrete performance including strength, placability and durability. In this paper, aspects of concrete production such as raw materials, economic constraints and production techniques are evaluated with the view to improving concrete performance.