

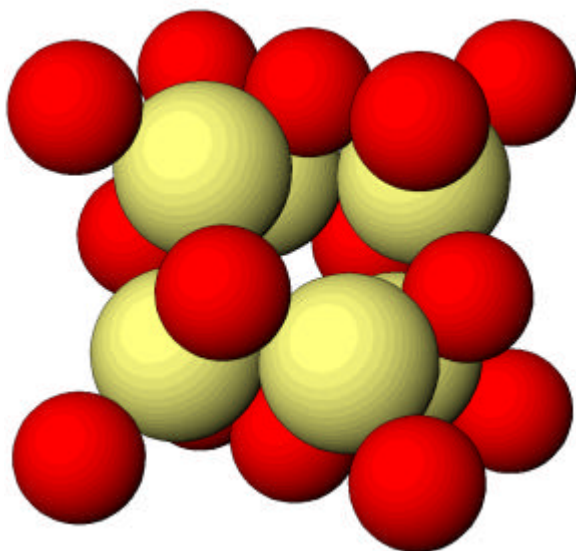


EUROPEAN SCIENCE FOUNDATION



OPTIMISATION OF SOLID STATE ELECTROCHEMICAL PROCESSES FOR HYDROCARBON OXIDATION (OSSEP)

An ESF Scientific Programme



In the search for new, improved energy systems for the 21st century, the emphasis is on environmentally friendly, efficient means of energy conversion and storage. High temperature fuel cells offer energy conversion efficiencies as high as 70% although there are still problems to overcome before the optimum fuel - natural gas, can be utilised without difficulty. Future applications of high temperature fuel cells in addition to power generation, is an area of considerable excitement. There is growing interest in transport applications and the use of alternative fuels. High temperature fuel cells offer considerable promise for working with more complex fuels, such as biogas or reformed higher alkanes, as their high temperature of operation facilitates reforming engineering. Closely allied to these applications in fuel cell technology is the utilisation of electrochemical reactors for the industrially important electrocatalytic oxidation of hydrocarbons. In the long term solid oxide fuel cell (SOFC) technology is foreseen to be a candidate for much more efficient electrolysis of steam and carbon dioxide. The Programme focuses on both fundamental and applied aspects

The high temperature electrochemical oxidation of hydrocarbons is an area of considerable strategic, environmental and economical importance. The purpose of this Programme is to promote investigations of the processes involved in the solid state electrochemical oxidation of hydrocarbons and hence to develop new fuel cell materials and systems to meet the challenges of the new millennium. The Programme facilitates the exchange of ideas and development of common programmes of activity. This is achieved by organising workshop meetings, by regular exchange of personnel on short-term visits and Collaborative Project Grants involving periods of research up to three months at one or more host laboratories.

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TESTING OF OXIDE ELECTRODES IN HYDROCARBON FUELS

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Many researchers have investigated electrode reaction to improve the cell performance. Since hydrogen, methane, methanol, etc... are used as fuel for SOFC, it is important to understand in detail electrode reaction on the anode side. Zirconia-mixed conductors are good candidates to replace the state of the art in SOFC cermet anodes. Ceria- and titania-doped YSZ mixed conductors could be good candidates as fuel electrode matrix.¹

In this work we have combined the good properties of the titania-doped yttria stabilised zirconia system with the good catalytic behaviour of Ceria, $Y_{0.15}Zr_{0.57}Ti_{0.13}Ce_{0.15}O_{1.925}$, (YZTC6).

The electrochemical properties of YZTC6 pure and the cermets with NiO or CuO have been studied, using the electrochemical impedance spectroscopy (EIS). And we have compared the properties of the traditional anode material (NiO cermets) with the properties of CuO cermets, given that copper is an excellent electronic conductor and inert to hydrocarbon activation.

The materials were prepared by the ceramic method, and characterized by PXRD analysis. Four cermets have been prepared by mixing YZTC6 with NiO and CuO in different weight ratios (sample 2a, 60%CuO; 2b, 20%CuO; 3a, 60%NiO; 3b, 20%NiO).

The electrochemical performance was investigated using two-electrode, symmetrical cell format. Experiments were performed in air, 5% H₂(wet) and 5%CH₄(wet) in the temperature range 500°C - 900°C in 50°C steps, cooling back to 500°C before changing atmosphere. For each temperature sufficient time was allowed to obtain the thermal equilibrium and the reproducibility of the measurements, a new tool to assess the reproducibility have been proposed.²

The impedance measurements were performed with a Solartron 1260 Frequency Response Analyser, at OCP, with a 15mV of a.c. perturbation. Measurements were carried out in the 1MHz to 0.1Hz frequency domain. Typical AC impedance measurements for YZTC6 are shown in Fig.1, at three different temperatures and in three different gas compositions.

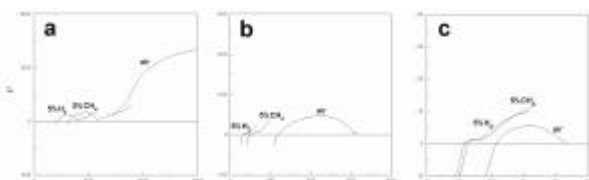


Fig. 1. Symmetrical-cell measurements for sample 1 at 500°C (a), 700°C (b), 900°C (c); in air, 5% H₂(wet) and 5% CH₄(wet).

The impedance data has been analysed in terms of equivalent circuit theory in order to obtain the series, polarisation and diffusion resistances of the materials, Fig. 2. The best fitting can be obtained with the following model: a combination of a series resistance

inductance element, at high frequencies; one or two R-CPE elements at intermediate frequencies related with the charge transfer processes; and at lower frequencies a Warburg impedance from the gas diffusion. We have used a new alternative representation of the impedance data³ to check the number of elements in the fitting model.

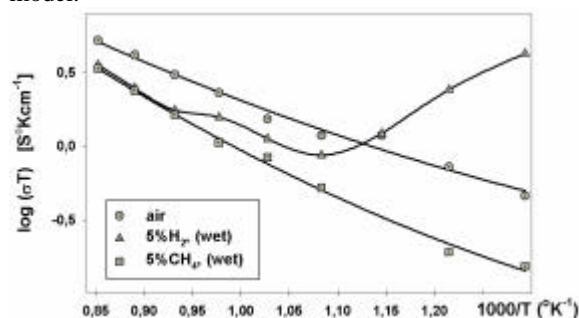


Fig. 2. Temperature dependence of R_{series} , symmetrical cell, for YZTC6+60%CuO.

Surface and cross-section SEM micrographs have been obtained to characterize the samples after the measurements, Fig. 3.

The sample with 20% in CuO has a very good distribution of the porosity, but the sample with higher content in CuO tends to form agglomerates. The cermets with NiO has a very bad porosity distribution, for example, the sample with 60% in NiO has large cavities.

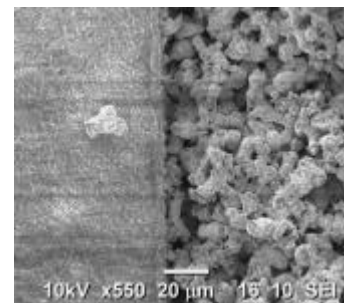


Fig. 3. SEM micrographs, cross-section, YZTC6+60%CuO.

In conclusion, we have found that the (YZTC6+60%CuO) cermet at 500°C in reducing conditions presents the best behaviour and it could be interesting to study this material at lower temperature. At this temperature, the conductivity for 60%CuO cermets is about 0.7 units higher than the conductivity of the corresponding NiO with the same weight composition. Copper cermets produced from reduction of the oxides suffer serious microstructural degradation above 600°C and this provides a major concern for utilisation of Cu based anodes in SOFCs. Although the series resistance was slightly lower for the cermets than the simple oxides at high temperatures, the polarisation resistances were slightly higher, indicating that the change in microstructure due to Cu migration was detrimental to electrode performance.

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TETRAGONAL ZIRCONIA AS NANOSTRUCTURED BULK AND THIN FILMS

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This report is focused on the initiation of different collaborations between Prof. Dr Joop Schoonman (The Netherlands) and Dr Elisabeth Djurado (France) since 2000 through a third Russian partner Dr Alla Pivkina relevant to OSSEP programme since 2001. The work was divided into two parts in the domain of materials for IT-SOFCs:

1 - Deposition and characterization of nanocrystalline tetragonal zirconia films using electrostatic spray deposition¹⁻³

Pure tetragonal and nanocrystalline 2 mol% Y₂O₃ doped ZrO₂ thin films were deposited on stainless steel substrates by Electrostatic Spray Deposition (ESD) technique. Tetragonal phase was evidenced by Raman spectrometry and X-ray diffraction in an as-deposited film at 400°C. Good homogeneity in composition is confirmed using wavelength dispersion scanning. Surface microstructure of dense to porous thin films was investigated varying both deposition parameters: substrate temperature and deposition time.

Key Words: Nanocrystalline tetragonal yttria doped zirconia, Electrostatic Spray Deposition, IT-SOFC

2 - Cold isostatic and explosive isodynamic compaction of Y-TZP nano-particles^{4,5}.

2.5 mol.% yttria-doped zirconia compacts of spray-pyrolysed powders were consolidated by three routes of dynamic compaction, i.e., explosive isodynamic compaction (EXIC), magnetic pulse compaction (MPC), and gas-hydraulic compaction (GHC). Starting powders of 69 nm mean primary crystallite size were tetragonal single-phased with good homogeneity in chemical composition and narrow crystallite size distribution. A comparative study of the microstructure of these pellets was

investigated in terms of grain size and density. Ceramics with maintained 6.9 nm average grain size were obtained by EXIC with a green compact density of 66% of the theoretical one. MPC led to nanocrystalline ceramics consisting in an average primary grain size of 10 nm, but not fully consolidated. Green compacts with a relative density of 72% and primary crystallite sizes of 2.3 nm were obtained by applying the GHC technique. All green compacts were still tetragonal single-phased 2.5 mol.% yttria-doped zirconia. Good sintering behavior is expected for green compacts of higher relative density, because the sintering temperature and the sintering time could be decreased to prevent grain growth and to retain the tetragonal crystal structure.

Keywords: explosive isodynamic compaction, magnetic pulse compaction, gas-hydraulic compaction, nanostructured tetragonal zirconia, IT-SOFC.

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EXPERIMENTAL SETUP FOR ELECTROCHEMICAL CHARACTERIZATION OF CERIA BASED MATERIALS

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The Department of Inorganic Chemistry of La Laguna University includes a solid state electrochemistry group with interest in the field of materials for Solid Oxide Fuels Cells. The visit was dedicated to the installation of some experimental techniques for electrical measurements of interest in this scientific area, and their automatic data acquisition methods; this comprised impedance spectroscopy and other ac and dc electrical measurements (e.g. the van der Pauw and other 4 point methods), in air and in controlled atmospheres. An additional ion blocking method was also tested for the determination of electronic contributions in mainly ionic conductors.

Two alumina sample holders were prepared for measurements in air, with 4 wires for connection to the sample and a type-S thermocouple for temperature control. A panel with fixed connections was used to improve the contacts and to reduce the risk to breaking the platinum wires in use. An additional system with the corresponding sample holder was also prepared for measurements under variable atmosphere; this comprises a tubular furnace with supply of adjustable gas-mixtures, and an oxygen sensor. It was necessary to use metallic covers to guarantee gas tightness, while allowing for the gas inlet and outlet, and for the exit of 4 measurement wires, and 2 thermocouple wires. These wires, were all fastened to panels of standard terminals, to avoid breaking the fragile platinum threads. This sample-holder was installed in a horizontal furnace and it can be used for impedance spectroscopy and dc conductivity measurements with 2 or 4 electrodes.

This methods are been used for the characterization of ceria based materials prepared from commercial powders (Rhodia) and alternative $Ce_{0.8}Gd_{0.2}O_{1.9}$ (GCO) powders prepared by a freeze-dried method. This preparative method has allowed reducing dramatically both the reaction time (e.g. 2 hours) and the reaction temperature (e.g. 600°C) in this

material, when compared with the classic ceramic method. Grain sizes lower than about 100nm were found in most of samples prepared by this method. Small amounts of cobalt were added to samples of GCO to enhance the densification. The sintering characteristics have been evaluated by measuring density (Archimedes method) and grain size (SEM). The electrochemical properties of gadolinia-doped ceria have been studied using AC impedance spectroscopy at the temperature range of 200-1000°C. AC measurements of cobalt-doped GCO revealed that the conductivity and activation energy remain nearly unchanged compared to pure GCO. Typical values of activation energy and total conductivity at 700°C were $\sigma=3.33$ S/m and $E_a=0.72$ eV for pure GCO, $\sigma=2.86$ S/m and $E_a=0.75$ eV for GCO+2cat%Co.

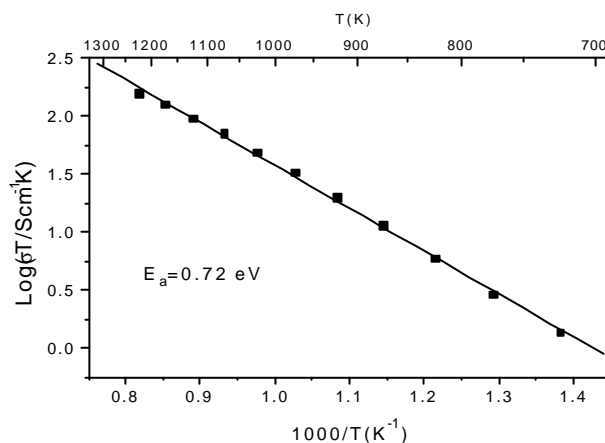


Figure 1- Total conductivity for GCO.

Reference: D.Pérez-Coll, P. Núñez, J.R. Frade and J.C.C. Abrantes, "Nanosize $Ce_{0.8}Gd_{0.2}O_{2\pm\delta}$ Prepared by Freeze Drying

LSCF-CGO COMPOSITE CATHODES FOR INTERMEDIATE TEMPERATURE SOLID OXIDE FUEL CELLS (IT-SOFCs)

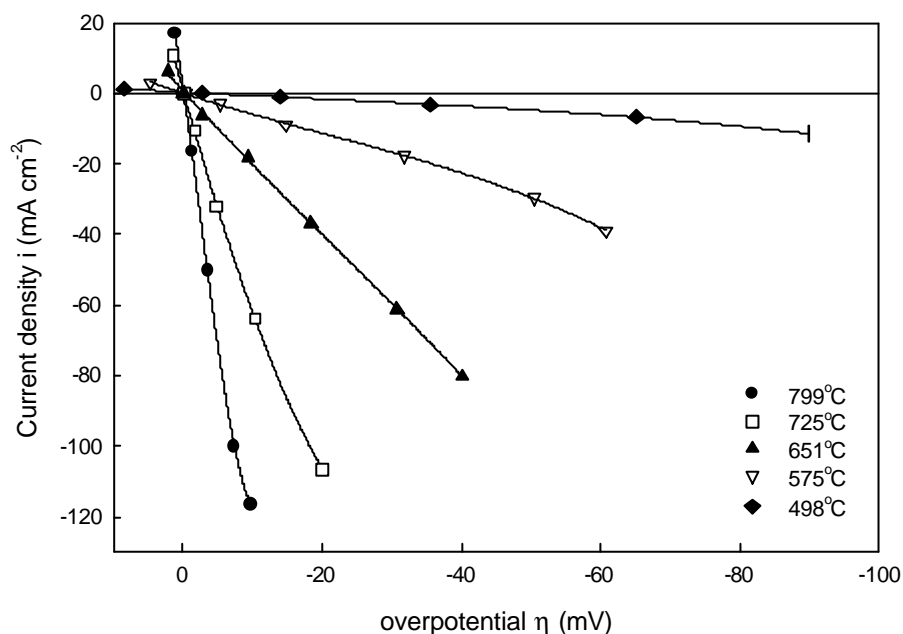
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Composite materials based on mixtures of $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_3$ (LSCF)- $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$ (20CGO) were examined for use as cathode materials in intermediate temperature solid oxide fuel cells operating at 500 C. The electrochemical kinetics of oxygen reduction have been investigated at different temperatures (500-800°C) using impedance spectroscopy and three-electrode

voltammetry. The composite cathode exhibited an area specific resistance of $0.08 \Omega \cdot \text{cm}^2$ at 800°C and $9.92 \Omega \cdot \text{cm}^2$ at 498°C. Exchange current densities at different temperatures were estimated from the low overpotential measurement (<10 mV) and were in good agreement with predictions based on the surface exchange coefficient k .



Current density versus overpotential of 70%wt $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-d}$ /30%wt $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.9-x}$ composite cathodes measured in air

PREPARATION AND CHARACTERISATION OF COPPER/YTRIA TITANIA ZIRCONIA CERMETS FOR USE AS POSSIBLE SOLID OXIDE FUEL CELL ANODES

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The potential of a new anode cermet based on Cu and titania doped yttria stabilised zirconia (YZT) for high temperature fuel cells was examined. Cermets were prepared by standard solid state reaction using $Y_{0.2}Ti_{0.18}Zr_{0.62}O_{1.9}$ and CuO as starting materials. Green pellets were sintered at 1000 °C for 10 hours resulting in sound pellets. These were successfully reduced in 5% H_2 in Argon again yielding sound pellets with a porosity of 50%. These were characterised by SEM XRD and TGA methods and their conductivity measured by ac impedance and the 4 point DC method as a function of both temperature and oxygen partial pressure.

On sintering there was evidence for a small amount of reaction between CuO and YZT. This resulted in a slight tetragonal distortion of YZT; however, most of the copper oxide was not incorporated into the zirconia. The cermet was successfully redox cycled and percolation was achieved when the copper composition exceeded 33% of the volume. Conductivity remains high under a wide range of oxygen partial pressures from the most reducing conditions up to 10^{-4} atm O_2 .

Electrochemical testing performed using three electrode geometry showed good performance for hydrogen oxidation for temperatures up to 800°C. At higher temperatures up to 1000°C copper was observed to be very mobile with considerable agglomeration of metallic copper particles. Indeed in some instances there was a total segregation of copper from YZT resulting in a copper layer forming at the electrolyte interface with the outer layer of the electrode being essentially YZT. This agglomeration and migration of Cu led to a significant degradation in electrochemical performance with large increases in the series resistance and polarisation resistance, especially under anodic bias. Due to these segregation problems copper based cermets produced in this manner are not thought to be good candidates for fuel cell electrodes operating at 1000°C.

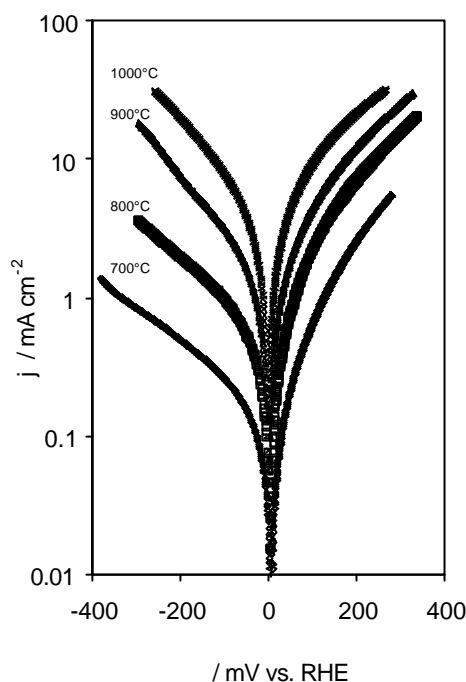


Figure 1 Current overpotential characteristics as a function of temperature in 97% H_2 /3% H_2O . Data are corrected for the series resistance. RHE refers to the reversible hydrogen electrode potential of this gas atmosphere at the various temperatures

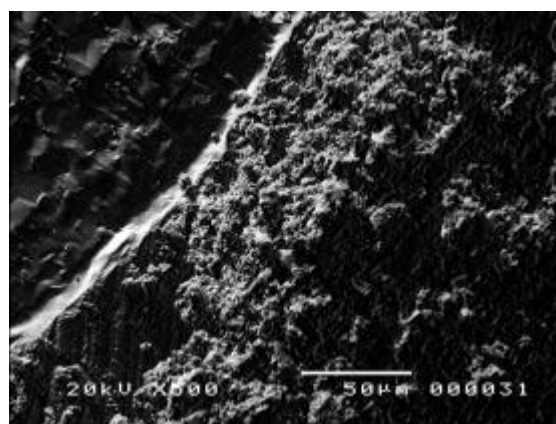


Figure 2 Scanning electron micrograph of the YZT/Cu(O) electrode after measurement, bright area shows segregation of Cu at the electrode/electrolyte interface.

THE EXTENT OF THE CUBIC FLUORITE PHASE IN SYSTEM ZrO_2 - TiO_2

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Aim

To further work done on the system ZrO_2 - TiO_2 - Y_2O_3 by member institutes¹⁻⁸. The phase limits in air of compositions containing minimum concentrations of stabilising aliovalent dopant were reassessed as it is these compositions which offer the highest levels of mixed conductivity. The influence of pO_2 on phase stability was evaluated due to the hypothesis that at lower pO_2 a larger concentration of metal ions will be accommodated into the cubic fluorite lattice.

Results and Discussion.

The composition dependence of lattice parameter is shown in figure 1 for compositions containing 20% Y as a function of Ti content. Literature data is included from Feighery et al.³ and Worrell et al.⁸ Lattice parameter is also calculated using the relation suggested by Kim.⁹ The relation for compositions based on ZrO_2 is given by

$$a = 0.512 + \sum(0.0212\Delta r_k + 0.00023\Delta z_k)m_k$$

where a (in nm) is the lattice constant of the ZrO_2 solid solution at room temperature, Δr_k (in nm) is the difference in ionic radius ($r_k - r_h$) of the k th dopant (r_k) and the host cation (r_h) in eightfold coordination according to Shannon¹⁰, Δz_k is the valency difference ($z_k - z_h$) and m_k is the mole percent of k th dopant in the form MO_x . This relation was empirically derived from literature data for binary ZrO_2 - MO_x systems at low concentrations of dopant (<25mol%). For higher dopant concentrations the fit of the Kim relation to experimental data is observed to be poor showing increased deviation from true values as dopant concentration increases. Despite this clear accuracy restriction, the empirical relation of Kim has been utilised in full phase diagram calculations for the ternary system ZrO_2 - $YO_{1.5}$ - TiO_2 by Yokokawa et al.¹¹ Assessment of the accuracy of the Kim relationship for the ternary system is therefore imperative.

Figure 1 exhibits a reasonable correlation between the experimental results of the different authors. Previously the data of Feighery et al. was analysed by fitting to a curve³. With more data now available in the low Ti range, it is clear that a linear fit better describes the data in this region. The lattice parameters calculated from the relation of Kim are in good agreement with the measured data in this low Ti concentration range. Such a good agreement between the Vegards slope of the calculated and experimental data is perhaps somewhat surprising when we consider

the previously mentioned accuracy restriction of the Kim relationship at high dopant concentrations. In this case several factors assist the fit of the Kim equation. Firstly, the Vegards slope is dependent only on the concentration of the Ti dopant as the Y dopant concentration is fixed at 20mol% $YO_{1.5}$ in these compositions. In the Kim calculation the fixed Y content serves only to vary the value of lattice parameter which corresponds to the intercept at a Ti content of zero. Secondly both Ti and Zr have the valency of 4+. The valency difference ($z_{Zr} - z_{Ti}$) of this dopant therefore reduces to zero in the Kim equation to leave the gradient dependent solely on the term $0.0212(r_{Zr} - r_{Ti})$. Finally the size of Ti^{4+} and Zr^{4+} in eightfold coordination are comparable (0.074, 0.084nm respectively). Subsequently any slight error in the constant 0.0212 stated by Kim therefore would not lead to serious variations in Vegards slope for the levels of Ti content analysed here.

The limit of the cubic fluorite phase is given by the location of the break in vergard slope as Ti composition increases. In figure 1, the work by Worrell et al.⁸ and Feighery et al.³ show a similar limit of the cubic fluorite solid solution at 15mol% Ti. The new data show the limit to occur later at 18mol% Ti. This variation of solid solution limit could be explained by variations in cooling profile or by slight furnace temperature variations between the authors. In the current work temperature was accurately monitored by a thermocouple placed near the samples and samples were quenched by dragging them into the furnace cold zone. To test the former hypothesis samples were reground and refired with a slower cooling rate of 4°C/min. Lattice parameters of the cubic phase of these samples are also plotted on figure 1. No significant change in lattice parameters could be induced by a change in cooling profile. Experiments to assess the importance of slight temperature variations on the solid solubility limit, by firing at 1450°C, are now in progress.

Figure 2 plots composition dependence of lattice parameter for compositions containing 25mol% $YO_{1.5}$ as a function of Ti content. Data from Kobayashi et al.¹² and Vegard behaviour predicted from the empirical relation of Kim⁹ are also presented. A close correlation between all data sets is observed. The Kim relation still appears to hold at this dopant level. The limit of the cubic solid solution is observed to be in the region of 25mol% Ti for both experimental sets of data. This is a much higher level of Ti for this $YO_{1.5}$ concentration than that of 15mol% Ti predicted from the phase diagram of Feighery et al.³

Samples $Zr_{(1-x)}Y_xTi_{0.3}O_{2-\delta}$, $x = 0.1, 0.125, 0.15, 0.175, 0.20$ and 0.25 fired in a gas mixture of $7\%H_2/93\%N_2$ or in pure H_2 at $1350^\circ C$ for 12 hours, $\log pO_2 = -17.58$ or -19.97 atms. respectively (determined by an oxygen sensor), are shown by XRD to be bi-phasic mixtures of monoclinic and cubic zirconia. The reduction of Ti under these conditions therefore cannot lead to full stabilisation of the cubic phase of zirconia for the compositions analysed. Assessment of Ti oxidation state by TGA and the variation of the monoclinic/cubic ratio for oxidised and reduced samples from XRD results is now in progress.

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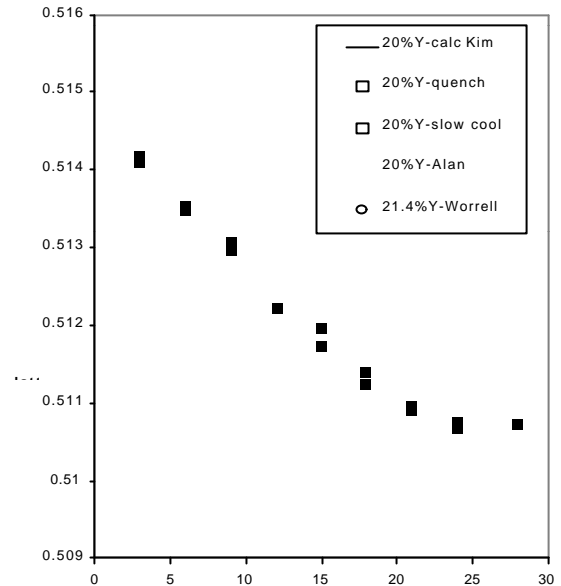


Figure 1. The composition dependence of lattice parameter for compositions containing 20% Y as a function of Ti content quenched from $1500^\circ C$. Literature data is included from Feighery et al.³ and Worrell et al.⁸ (21.4 mol% Y) Lattice parameter is also calculated using the relation suggested by Kim⁹. Data for samples slow cooled at $4^\circ C/min$ are presented

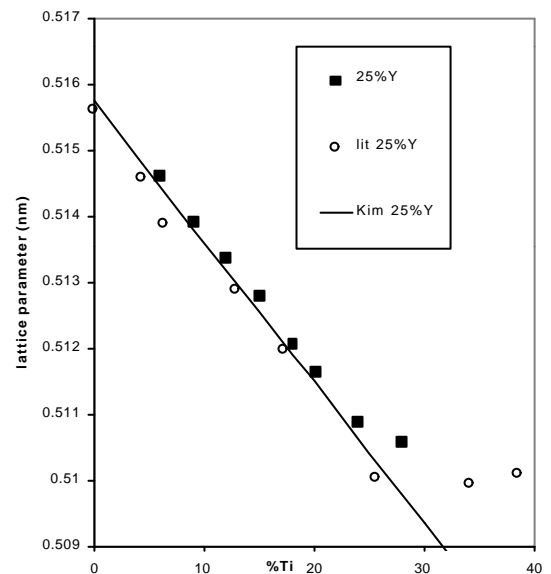


Figure 2. Composition dependence of lattice parameter for compositions containing 25mol% $YO_{1.5}$ quenched from $1500^\circ C$ as a function of Ti content. Lattice parameter is also calculated using the relation suggested by Kim⁹

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