

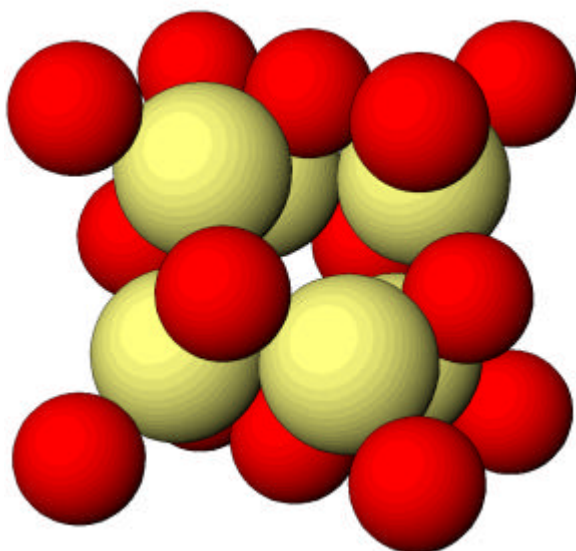


EUROPEAN SCIENCE FOUNDATION



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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PROGRAMME 2004

19-21 FEBRUARY
ROME, ITALY
WORKSHOP ON
ELECTRODE PROCESSES AND KINETICS IN SOFCs

11-12 JUNE
EINDHOVEN, THE NETHERLANDS
WORKSHOP ON
DEFECT IN OXIDES

17-19 NOVEMBER
LA LAGUNA, CANARY ISLANDS, SPAIN
FINAL WORKSHOP ON
**OPTIMISATION OF SOLID STATE FC PROCESSES FOR ALL ASPECTS OF
HYDROCARBON OXIDATION**

CERMETS WITH A PROTON-CONDUCTING CERAMIC AS POTENTIAL SOFC ANODES

G.C. Mather¹, F.M. Figueiredo^{2,3}, J.R. Jurado¹ and J.R. Frade²

¹ Instituto de Cerámica y Vidrio, CSIC, Camino de Valdelatas, s/n, Campus de Cantoblanco, 28049 Madrid, Spain

² Departamento de Eng^a. Cerâmica e do Vidro, Universidade de Aveiro, Aveiro 3810-193, Portugal

³ Departamento de Ciências Exactas e Tecnológicas, Universidade Aberta, R. Escola Politécnica, 147, 1269-001 Lisbon, Portugal

The cermet structure of a solid oxide fuel cell, most typically Ni-YSZ (yttria-stabilised zirconia), provides a number of important functions, including adhesion of the electrocatalytically active species (Ni) to the ion-conducting electrolyte, high porosity for gas transport and both thermal-expansion and chemical match of anode and electrolyte. In addition is the provision of active reaction sites at the triple-phase boundary (TPB) among fuel, electronic conductor (Ni) and oxide-ion conductor (anodic and electrolytic YSZ). The ionically conductive component of the cermet is generally believed to be requisite to the reaction mechanism, which primarily takes place in the interfacial region between cermet and electrolyte to a thickness of approximately 20 μm [1]. The electroactivity of the anode may be altered, however, on exchanging the pure oxide-ionic conductor of the cermet with a proton conducting ceramic since the exothermic enthalpy of hydration of the latter favours hydrogen absorption and higher proton mobility.

In this ESF project (duration 2 months), cermet anodes of Ni-CaZr_{0.95}Y_{0.05}O_{2.975} (Ni-CZY) and Ni-SrZr_{0.95}Y_{0.05}O_{2.975} (Ni-SZY) were synthesised by combustion from the component nitrates and urea as the fuel followed by sintering and reduction to Ni metal. The anodes were co-pressed and co-sintered on green YSZ electrolyte to produce anode/electrolyte/anode assemblies with good anode-electrolyte adherence and planar

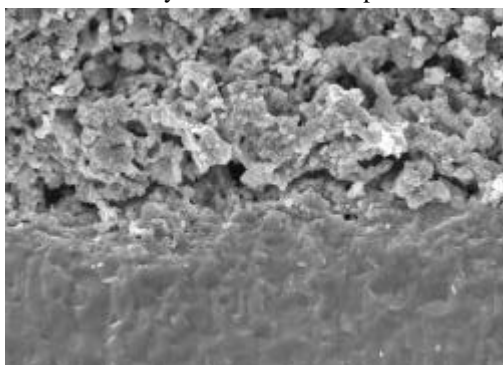


Fig. 1 Scanning electron micrograph of a fractured surface of the Ni-CSZ/YSZ interface.

electrodes of thickness, ca. 150-170 μm (Fig. 1). The anode microstructures are porous (ca. 45%) and are composed of a homogeneous and disperse distribution of submicronic Ni metal and ceramic particles. No significant microstructural differences between the anodes were apparent on extensive examination by scanning electron microscopy (SEM).

Impedance spectroscopy was conducted on the symmetrical cells in different regimes of hydrogen and water-vapour partial pressure. The polarisation behaviour of the Ni-CZY and Ni-SZY electrodes were examined with an Autolab impedance analyser (ECO Chimie) working in the frequency range, 10 mHz $\leq f \leq 50\text{kHz}$, with an amplitude of 30 mV ; Ni mesh was used as current collector. The polarisation resistance is composed of two or more pH_2O -sensitive contributions and appears to be significantly altered by the cermet composition. A lower polarisation resistance is associated with the ceramic with higher proton conductivity (SZY), Fig. 2, suggesting that protonic conductivity in the anodic ceramic is involved in the reaction of hydrogen oxidation. However, results also indicate that substantial oxygen-ion conductivity is required at the anode/electrolyte interface in order to achieve competitively low polarisation resistances. Further work will be devoted to developing anode configurations in which oxygen and proton conductivity are maximised.

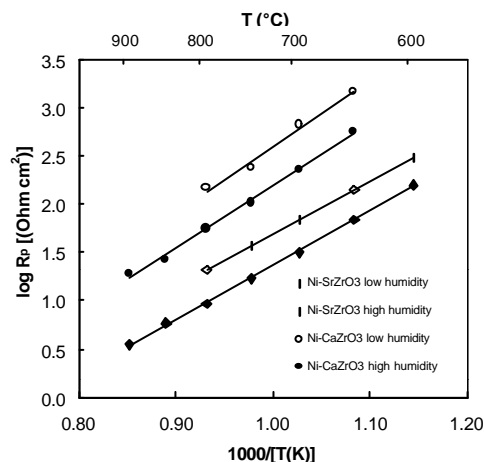


Fig 2. Total polarisation resistances of the Ni-CZY and Ni-SZY anodes in different humidities as a function of temperature. Open symbols correspond to 10% H_2 in N_2 bubbled through water at 0°C; closed symbols water at 29°C

OXYGEN DIFFUSION IN LAMOX MATERIAL

S. Georges¹, M.C. Steil², S.J. Skinner³, P. Lacorre¹.

¹ Laboratoire des Fluorures, Université du Maine, Le Mans, France.

² Laboratoire de Cristallographie et de Physicochimie du Solide, Université de Lille, France.

³ Imperial College of Science, Technology and Medicine, London, UK.

The recently discovered LAMOX family¹, from parent compound $\text{La}_2\text{Mo}_2\text{O}_9$, exhibits fast oxidation transport properties². This remarkable property is interesting in the current need for more efficient solid electrolytes in Solid Oxide Fuel Cells (SOFC).

Chemical composition was optimized looking at both ionic conductivity and reducibility^{3,4}. The densification process of the starting powders was then investigated⁵.

On the optimized compositions, turned into high relative density pellets (>96%), we performed oxygen diffusion by isotopic ($^{16}\text{O}/^{18}\text{O}$) exchange and Secondary Ion Mass Spectrometry (SIMS).

Preliminary exchange attempts under $^{18}\text{O}_2$ atmosphere were unsuccessful. In such conditions, we were unable to measure any diffusion profile. All the experiments were then performed under an H_2^{18}O atmosphere, which gave total satisfaction. The surface exchange coefficient is extremely low in the case of $^{18}\text{O}_2$, but was determined as always superior to D (diffusion coefficient) in the case of H_2^{18}O . The diffusion profiles were collected by line scanning on polished pellets cross sections.

A typical diffusion profile is given in figure 1.

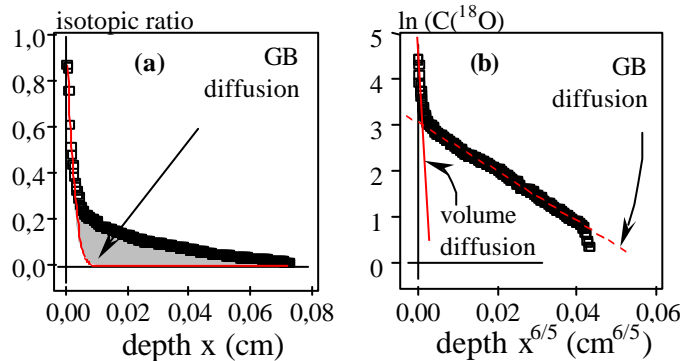


Figure 1: diffusion profile measured at 800°C on $\text{La}_{1.7}\text{Gd}_{0.3}\text{Mo}_{0.8}\text{W}_{1.2}\text{O}_9$. (a): fit with Crank equation, (b): fit with Le Claire equation⁶.

For near-surface regions, the Crank equation fits perfectly the experimental data (figure 1a). On the

contrary, as the depth increases, a second contribution appears. The latter behaves like grain boundary diffusion, in agreement with Le Claire equation⁶ (1):

$$Dd = 0.661 \left(\frac{\partial(\ln C)}{\partial x^{6/5}} \right)^{-5/3} \left(\frac{4D}{t} \right)^{1/2} \quad (1)$$

As mentioned in figure 2, the grain boundary diffusion contribution to the experimental diffusion profiles increases with temperature.

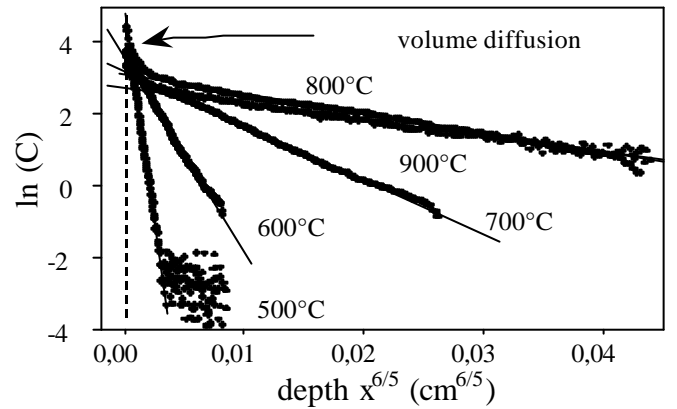


Figure 2: increase with temperature of the grain boundary diffusion contribution.

Finally, the volume diffusion coefficient was related to the ionic conductivity, determined by AC impedance spectroscopy, through the Nernst-Einstein equation ((2) for oxide-ion transport). We used a theoretical value of 0.653 for the correlation factor (value for a cubic primitive lattice). A least squares refinement allowed us to determine the charge carriers concentration c_i .

$$c_i = 1.348 \cdot 10^{14} \left(\frac{s_i T}{D_i} \right) \quad (2)$$

We found around 6 oxide-ions per unit formula (only slightly depending on both T and composition). This number has been related to the crystal structure of $\beta\text{La}_2\text{Mo}_2\text{O}_9$. Figure 3 shows, in

agreement with the LPS concept⁷, that the O3 oxygen site is mostly responsible for the ion transport in our materials. Indeed, a strong and anisotropic thermal factor affects this partially occupied site. However the O2 site with partial occupancy might also participate to the ionic mobility.

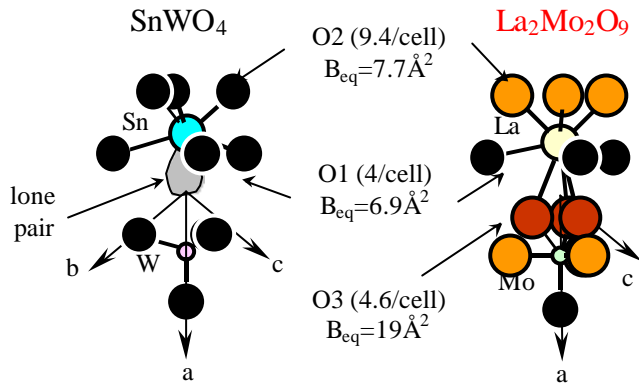


Figure: Illustration of the LPS concept⁴. Origin of the ion mobility in $\text{La}_2\text{Mo}_2\text{O}_9$.

A comparison with equivalent data from reference materials^{8,9} (table 1) such as stabilized zirconia or doped lanthanum gallate shows that members of the LAMOX family have higher tracer diffusion coefficients (ex. $D=1.41 \times 10^{-6} \text{ cm}^2 \cdot \text{s}^{-1}$ at $T=800^\circ\text{C}$ for $\text{La}_{1.7}\text{Gd}_{0.3}\text{Mo}_2\text{O}_9$).

Table 1: Tracer diffusion coefficients of reference materials in comparison with that of LAMOX materials

Composition ($T = 800^\circ\text{C}$)	D ($\text{cm}^2 \cdot \text{s}^{-1}$)	Réf.
$\text{Zr}_{0.81}\text{Y}_{0.19}\text{O}_2$	6.2×10^{-8}	(5,8)
$\text{Zr}_{0.858}\text{Ca}_{0.142}\text{O}_2$	7.54×10^{-9}	(8)
$\text{Zr}_{0.85}\text{Ca}_{0.15}\text{O}_2$	1.87×10^{-8}	(8)
$\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_2$	2.7×10^{-8}	(5,8)
$\text{La}_{0.9}\text{Sr}_{0.1}\text{Ga}_{0.8}\text{Mg}_{0.2}\text{O}_3$	3.24×10^{-7}	(8)
$\text{La}_{0.8}\text{Sr}_{0.2}\text{Ga}_{0.8}\text{Mg}_{0.2}\text{O}_3$	4.13×10^{-7}	(8)
$\text{La}_{1.7}\text{Gd}_{0.3}\text{Mo}_2\text{O}_9$	1.41×10^{-6}	
$\text{La}_{1.7}\text{Gd}_{0.3}\text{Mo}_{0.8}\text{W}_{1.2}\text{O}_9$	4.8×10^{-7}	
$\text{La}_{1.4}\text{Nd}_{0.6}\text{Mo}_2\text{O}_9$	7.44×10^{-7}	
$\text{La}_{1.9}\text{Y}_{0.1}\text{Mo}_2\text{O}_9$	9.62×10^{-7}	

It confirms conductivity measurements, and potential interest of these new oxide-ion conductors as electrolytes for Solid Oxide Fuel Cells.

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- ⁹ T. Ishihara et al. *Solid State Ionics*, 113-115 (1998) 593-600.

Jesús Canales-Vázquez¹, Mikhail Patrakeev², John Irvine¹ and Jorge Frade²

¹ *School of Chemistry, University of St Andrews, St Andrews, Fife KY16 9ST UK.*

² *Departamento de engenharia cerâmica e do vidro, Universidade de Aveiro, 3810 Aveiro, Portugal.*

The aim of the visit was to complete the electrochemical characterisation of a novel class of perovskite-related phases with interstitial oxygen, $\text{La}_2\text{Sr}_4\text{Ti}_6\text{O}_{19}$. For that purpose dense samples are desirable in order to carry out measurements such as electrochemical permeability, Faradaic efficiency and ionic transport numbers. Therefore the first efforts were directed to obtain dense samples (above 92%).

Several approaches were used to achieve dense samples, ranging from the use of sintering additives to different firing conditions and atmospheres and slight changes in stoichiometry. Synthesis of the mentioned phases was achieved by solid state reaction after previous intimate mixture of the precursors and pre-calcination. The formation of $\text{La}(\text{OH})_3$ was observed to play a very important role, preventing the best sintering conditions. Two calcination steps at 1200°C for 6 hours each with intermediate ball milling were used to avoid its formation. There were however some other aspects that helped the sintering, such as the use of additives (0.5% w. Fe_2O_3), isostatic pressing, small particle size in the precursors and slow ramping in the high temperature furnaces. Samples with density up

to 97% were obtained after firing for 48 hours at 1500°C , which means a drastic reduction of the sintering time previously used. Four terminal conductivity measurements under controlled oxygen partial pressure were performed revealing that the material behaves as a semiconductor until $P(\text{O}_2) = 10^{-18}$ atm. In more reducing conditions the material showed metallic behaviour. Thereby, there is an insulator to metal transition as a function of the oxygen partial pressure.

Seebeck coefficients were also determined confirming the n-type conductivity that ac impedance measurements suggested in previous studies. No evidences of ionic contribution to the total conductivity were found using the mentioned techniques.

Synthesis in ultra-reducing conditions was also attempted in order to improve the values of total conductivity. Such ultra-reducing conditions were achieved by using a high temperature graphite furnace up to 1700°C . Unfortunately, the conditions seemed to be too severe and the samples decomposed leading to TiO and $\text{La}(\text{OH})_3$. Lower temperatures were also attempted but they also provoked decomposition.

OSSEP
ELECTROSTATIC SPRAY DEPOSITION OF YSZ ELECTROLYTES FOR SOLID OXIDE FUEL CELLS (SOFC)

S. Uhlenbruck¹, T. Hoppe¹, H. P. Buchkremer¹, D. Stöver¹, J. Schoonman²

¹ *Forschungszentrum Jülich GmbH, Institute for Materials and Processes in Energy Systems, IWV,
D-52425 Jülich, Germany*

² *Delft University of Technology, Department of Inorganic Chemistry, Julianalaan 136, NL-2628 BL Delft, The Netherlands*

During a short-term attendance at the Department of Inorganic Chemistry at the Delft Technical University a set of experiments using the Electrostatic Spray Deposition (ESD) technique to coat anode substrates of solid oxide fuel cells with electrolyte precursors were carried out.

The electrolyte of an SOFC should be as thin as possible to reduce performance losses due to its internal ohmic resistance, but has to be gas-tight, as it has to separate the two gas chambers where the fuel gas is oxidized and the oxygen is reduced.

Currently, in an anode-supported concept the commonly used electrolyte material 8 mol % yttria-stabilized zirconia (8YSZ) is deposited by vacuum slip casting or screen-printing. With these techniques micrometer-sized powder particles of 8YSZ are deposited, and it is necessary to sinter these particles at high temperatures (about 1400 °C) to get a dense layer.

Another process used for deposition of 8YSZ is plasma spraying. However, sufficiently gas-tight layers for a proper application in SOFCs were not yet achieved without an additional heat treatment.

A new approach is to use metal-organic Y- and Zr-precursors instead of 8YSZ powders. The precursors are deposited on top of the substrate's surface. During a heat treatment the organic parts are decomposed, and very small particles of the desired metal oxides develop. These small particles have a high sintering activity and may lead to dense layers at lower temperatures than necessary for the above mentioned methods.

$Y(O_2C_5H_7)_3 \cdot xH_2O$ and $Zr(O_2C_5H_7)_4$ were dissolved in mixtures of ethanol and butyl carbitol ($C_8H_{18}O_3$).

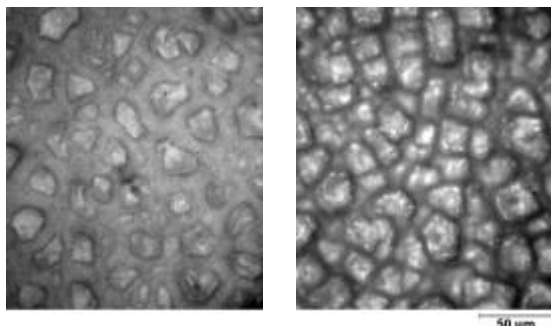


Figure: 8YSZ layers on anode substrates; substrate temperature during deposition: 300 °C (left) and 600 °C (right); layer thickness after heat treatment (700 °C): 2-3 μm [optical micrograph, Olympus PMG 3].

The concentration was between 0.01 and 0.05 mol Zr^{4+} per liter. The precursors were deposited on SOFC anode substrates made of NiO and 8YSZ [1]. The substrate temperatures varied between 200 and 500 °C. The acceleration voltage was between 7 and 11 kV, the spraying distance 10 to 40 mm and the precursor flow rate 6 ml/hour. Some of the experiments were carried out with an ESD device developed at TU Delft, The Netherlands [2], further experiments were performed with a commercially available ESD apparatus [3]. After deposition the samples were heated to 700 °C to remove all organic residues.

Two problems arose during the deposition. Due to the insulating nature of the substrate it was difficult to remove the charges that generate the spraying cone from the substrate's surface. Secondly, a significant portion of the precursor solution is sucked up by the porous substrate.

After removal of the organic residues a "floe structure" of flat, smooth and dense particles with an extension of about 30 μm, and broad cracks between them was found. The distance between the floes can be significantly reduced by an increase of the substrate temperature during the deposition (Figure).

Acknowledgements:

One of the authors (S.U.) would like to thank Prof. Schoonman and his group for valuable discussions and kind hospitality. The funding of this collaboration between Forschungszentrum Jülich and TU Delft by the European Science Foundation through the OSSEP scientist exchange program is gratefully acknowledged.

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- [3] AST Advanced Surface Technology, Beukenlaan 136, 2665 DR Bleiswijk, The Netherlands

ADVANCED ANODES FOR HIGH TEMPERATURE FUEL CELLS
SUMMARY AND RECOMMENDATIONS FOR RESEARCH ON MATERIALS PROPERTIES

A. Atkinson¹, S Barnett², R. Gorte³, J.T.S. Irvine^{4*}, A.J. McEvoy⁵, M.B. Mogensen⁶, S Singhal⁷, J. Vohs³,

¹*Dept of Materials, Imperial College, London, SW7 2BP, UK*

²*Northwestern University, Department of Materials Science, Evanston, IL 60208, USA*

³*University of Pennsylvania Department of Chemical Engineering, Philadelphia, PA 19104, USA*

⁴*School of Chemistry, University of St Andrews, St Andrews, Fife, KY16 9ST, UK*

⁵*DC-LPI Department, EPFL, 1015 Lausanne, Switzerland*

⁶*Risø National Laboratory, Materials Research Department, P.O. Box 49, Denmark*

⁷*Pacific Northwest National Laboratory, 902 Battelle Blvd, Richland, WA 99352, USA*

Major advances in SOFC anode development have been achieved in recent years and there is good encouragement that new fuel electrode formulations for second generation commercial fuel cells can be found. Such new anodes will offer improved redox tolerance and better resistance to coking in hydrocarbon fuels. Two key strategies have been identified, based on oxidation catalysts such as ceria or lanthanum chromium manganite or reforming catalysts such as nickel or ruthenium doped lanthanum chromite. Additionally the function of electronic conductivity in the anode current collector has also been addressed in oxides such as those based upon strontium titanate. Although no single material fulfils all the current collection, electrochemical and catalytic performance indicators required for a supported electrode design to the extent that nickel zirconia cermets do, a number of systems individually meeting these criteria have been attained. Clearly new composites are available to fulfil the roles of both the active electrode layer and the current collecting part.

Further research is required to optimise these materials (composition and microstructure); particularly to maximise electronic conductivity without sacrificing essential chemical stability under reducing conditions. Detailed studies are required on the most promising materials to establish and understand dimensional stability on redox cycling and long-term operation and how this can be improved without compromising electrical conductivity. Studies of oxygen diffusion, ionic conductivity and surface exchange in reducing environments are required on the most promising materials in order to improve understanding of their electro-catalytic properties. Particular care is necessary to allow reproducibility of results between laboratories and especially to ensure that meaningful experiments are

performed before their results are presented. Understanding of crystal structure, point defect populations and electronic structure need to be improved at a fundamental level and related to the key properties mentioned above in order to guide the search for new materials. More basic work to probe the mechanistic details of fuel utilisation reactions needs to be undertaken. These details are extremely important to rational design of more efficient anodes and thus high-performance SOFCs powered by practical fuels. Practical studies in real fuels need to be expanded, especially addressing issues such as sulphur tolerance and ability to utilise biofuels. The search for totally new oxide materials with even better properties should continue, because even the best ones only just meet the requirements. Most importantly this should include the highest possible electronic conductivity since such a material would also serve as a current collector.

Acknowledgements

Conclusions from the joint ESF/NSF OSSEP workshop organised in Strasbourg, France, December 2002, under the auspices of the ESF OSSEP programme with support from the US DOE and NSF. A manuscript based on this workshop has been published in Nature Materials - Atkinson A.; Barnett S.; Gorte R.J.; Irvine J.T.S.; McEvoy A.J.; Mogensen M.; Singhal C.; Vohs J.M. Nature Materials 2004, 3,17.

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ESF scientific programmes are principally financed by the Foundation's Member Organisations on an *à la carte* basis. OSSEP is supported by Statens Teknisk-Videnskabelige Forskningsråd, Denmark; Hermann von Helmholtz-Gemeinschaft Deutscher Forschungszentren (HGF), Germany; Consiglio Nazionale delle Ricerche (CNR), Italy; Nederlandse Organisatie voor Wetenschappelijk Onderzoek (NWO), Netherlands; Norges Forskningsråd, Norway; Instituto de Cooperaçao Científica e Tecnológica Internacional, Portugal; Consejo Superior de Investigaciones Científicas (CSIC), Spain; Oficina de Ciencia y Tecnologia (OCYT), Spain; Schweizerischer Nationalfonds zur Förderung der Wissenschaftlichen Forschung (SNF), Switzerland; Engineering and Physical Sciences Research Council (EPSRC), United Kingdom.

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Email: rrjurado@icv.csic.es

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Email: Mogens.Mogenseen@risoe.dk

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Department of Inorganic Chemistry,
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Email: j.schoonman@tnw.tudelft.nl

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Universita degli Studi di Roma "La Sapienza",
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Email: scrosati@uniroma1.it

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