

# Lanthanum chromite materials as potential symmetrical electrodes for Solid Oxide Fuel Cells

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A commonly used interconnector material has been tested as electrode for a new concept of Solid Oxide Fuel Cell, where the same material could be used, simultaneously, as interconnector, anode and cathode. We have found that a typical substituted chromite, such as  $\text{La}_{0.7}\text{Ca}_{0.3}\text{CrO}_{3-\delta}$  (LCC) can be considered a good candidate for such configuration, due to its high electronic conductivity in both reducing and oxidising conditions, and moderate catalytic properties for oxygen reduction and hydrogen oxidation. The symmetrical design renders performances of  $100 \text{ mWcm}^{-2}$  at  $950^\circ\text{C}$ , using  $\text{O}_2$  and  $\text{H}_2$  as oxidant and fuel respectively. Performances exceeding  $300 \text{ mWcm}^{-2}$  can be predicted for a  $100\mu\text{m}$ -thick YSZ electrolyte.

*Keywords:* Solid Oxide Fuel Cell, symmetrical electrodes, interconnector, YSZ, Chromites

## Cromitas de Lantano como potencial electrodos simétricos para Pilas de Combustible de Óxido Sólido

Un material comúnmente utilizado como interconector ha sido probado como electrodo para un nuevo concepto de Pila de Combustible de Óxidos Sólido, en el cual el mismo material se utiliza, simultáneamente, como interconector, ánodo y cátodo. Hemos encontrado que una cromita típica como  $\text{La}_{0.7}\text{Ca}_{0.3}\text{CrO}_{3-\delta}$  (LCC) puede ser considerada una buena candidata para dicha configuración, debido a sus altas conductividades eléctricas tanto en condiciones reductoras como oxidantes y una aceptable actividad catalítica para la reducción del oxígeno y la oxidación del hidrógeno. El diseño simétrico permite obtener rendimientos del orden de  $100 \text{ mWcm}^{-2}$  a  $950^\circ\text{C}$ , utilizando  $\text{O}_2$  e  $\text{H}_2$  como oxidante y combustible, respectivamente. Rendimientos que superan los  $300 \text{ mWcm}^{-2}$  pueden predecirse para pilas con electrolitos de YSZ de  $100 \mu\text{m}$  de grosor.

*Palabras clave:* Pilas de Combustible de Óxido Sólido, electrodos simétricos, interconector, YSZ, cromitas.

## 1. INTRODUCTION

In recent years Fuel Cells have become a potential alternative to the traditional combustion of fossil fuels to produce cleaner energy, directly from the electrochemical reaction of a fuel and an oxidant (1-4). Generally, hydrogen containing species are used as fuel, e.g. hydrogen, hydrocarbons, alcohols, ammonia, etc. and as oxidant the simplest option is air.

A Solid Oxide Fuel Cell (SOFC) comprises three solid components: a porous anode and a cathode which are separated by a dense solid electrolyte. At the cathode, the oxygen is reduced to oxide ions that diffuse through the electrolyte to the anode, where they can recombine with protons coming from the fuel oxidation. This process produces usable electricity, heat and water when hydrogen is used as fuel. Typical working temperatures are in the range of  $800\text{--}1000^\circ\text{C}$  that allows an adequate ionic conductivity from the electrolyte.

Due to the high operation temperatures all the elements must fulfil some requirements. The electrodes must show high electronic conductivity and high catalytic activity in the corresponding gas-environment. Both electrodes should exhibit an adequate degree of porosity; the higher limit is controlled mainly by mechanical properties and the lower

value is controlled by the good transient of gaseous species. All the elements must show chemical and dimensional stability, during cell operations and the fabrication process and compatibility with the other cell elements. More specifically, the electrolyte should be a gas-tight material to avoid contact between the reactants gases and it should be a pure ionic conductor with negligible electronic conductivity to avoid short-circuits across the cell, which in turn would result in poorer performances.

Precious metals could be used as SOFC cathodes, although they are rather expensive and therefore not adequate for mass-scale applications. Alternatively,  $\text{LaMnO}_3$ -based materials typically substituted with lower valence cations, e.g. Sr, Ba, in the A-site are used due to its good electronic conductivity (1,2) and compatibility with YSZ electrolyte.

The state-of-the-art anode material is the Ni-YSZ cermet, due to its good catalytic activity and electronic conductivity. Moreover, nickel is really cost-effective, which facilitates the implementation of these anodes for mass-scale production. However, Ni-YSZ presents some disadvantages related to the low tolerance to sulphur, carbon build-up when using hydrocarbon fuels and volume instability upon redox cycling

(5-6). Another important and frequently forgotten issue concerns NiO toxicity (7-8). Most of these problems have been overcome with the introduction of new materials based mainly upon perovskite structures such as chromo-manganites (9-11), titanates (12-14) or double perovskites as  $\text{Sr}_2\text{MgMoO}_{6-8}$  (15).

As for the electrolyte, the most common choice are materials exhibiting the fluorite structure such is the case of yttria-stabilised zirconia (YSZ) or rare earth substituted ceria for IT-SOFCs (1,2).

An additional component is necessary to connect several cells in series to produce a stack, i.e. the interconnector (1,2). It requires the highest electronic conductivity in both reducing and oxidising conditions, it should be chemically inert with the electrodes and it should have low permeability for oxygen and hydrogen to avoid direct combination of oxygen and fuel during cell operation. A commonly used material that meets these rather restrictive requirements is the lanthanum chromite ( $\text{LaCrO}_3$ ). The replacement of lanthanum by other cations such as Sr or Ca results in enhanced electronic conductivity.

A new concept in Solid Oxide Fuel Cell, recently proposed, is the Symmetrical Solid Oxide Fuel Cell (SFC) (16-18). In this approach, the same electrode material is used, simultaneously, as cathode and anode in a Fuel Cell, thus replacing the traditional SOFC configuration, Figure 1. The development of SFCs would simplify notably the production of fuel cells, minimising compatibility problems between components, reduction of costs derived from the simpler fabrication process and minimisation of problems related with reversible sulphur poisoning and carbon deposition by simply reversing the gas flows. The first fuel cell tested in this new configuration (17) rendered promising performances, e.g. ~550 and ~350  $\text{mWcm}^{-2}$  in  $\text{H}_2$  and  $\text{CH}_4$  respectively at  $950^\circ\text{C}$ .

In theory, the chromites generally used as interconnectors fulfil almost all the conditions required for a potential symmetrical electrode material. The main problem of the chromites relates to their rather modest catalytic activity towards hydrocarbon oxidation (19-20). However a very simple fuel cell could be produced if an interconnector material could be used as a symmetrical electrode, given that the four elements of a typical fuel cell, e.g., interconnector, anode, electrolyte and cathode, Figure 1, could be reduced to just 2 elements: interconnector and electrolyte.

In the communication presented herein, we study this new SOFC configuration, using a Ca-doped lanthanum chromite,  $\text{La}_{0.7}\text{Ca}_{0.3}\text{CrO}_{3-8}$  (LCC), as possible electrode material for Symmetrical Solid Oxide Fuel Cells (SFC).

## 2. EXPERIMENTAL

### 2.1. Materials preparation

LCC powders were prepared by conventional solid state reaction. Stoichiometric amounts of pre-dried high purity reactants:  $\text{La}_2\text{O}_3$  (99,99%, Aldrich),  $\text{CaCO}_3$  (99+%, Merck) and  $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (99% purity, Aldrich) were mixed and ground in acetone using zirconia ball mills for 30 min. and then fired at  $1100^\circ\text{C}$  for 10 hours, this last step was repeated twice. The powders were pressed into pellets and finally fired at  $1400^\circ\text{C}$  for 5 hours. Phase purity was evaluated by X-ray diffraction (XRD) on a Philips X'Pert Pro automated diffractometer, equipped with a Ge(111) primary monochromator and a X'celerator detector. The compatibility between YSZ and LCC was also monitored by XRD. A 1:1 YSZ-LCC composite was prepared and fired at different temperatures, e.g. 1000, 1200 and  $1400^\circ\text{C}$ , for five hours prior to running the XRD experiments.

Some of the LCC pellets were used for the electrochemical characterisation by impedance spectroscopy. In that case, a platinum-based ink was used to paint a current collector in both sides of the pellet. Then, the sample was fired at  $950^\circ\text{C}$  for 30 min. to remove the organic material.

YSZ used as electrolyte and in the composite mixtures was from Tosoh. Dense YSZ pellets, for the symmetrical cell measurements and fuel cell tests, 2 mm-thick and 20 mm diameter were obtained after uniaxially pressing YSZ powders at 1 ton for 1.5 min. The resulting pellets were sintered at  $1500^\circ\text{C}$  for 10h.

YSZ-based composites of LCC were prepared in a ratio 1:1 (w/w). The powders were mixed with acetone in a agatha mortar and left to dry at room temperature.

### 2.2. Overall conductivity

AC Impedance measurements were carried out in a Pt-wire electrochemical cell in both air and flowing 5%  $\text{H}_2$  atmospheres in the  $400\text{-}950^\circ\text{C}$  temperature range. First, the sample was heated-up to  $950^\circ\text{C}$  and the impedance was measured on cooling. After that, the sample was heated-up again to  $950^\circ\text{C}$  to perform an in situ sample reduction. The measurements were performed after reaching equilibrium conditions. The measurements were carried out in a Solartron FRA 1260 in the 0.1 Hz to 1 MHz frequency range. A 15mV a.c perturbation was used rendering reproducible spectra. The automatization of the measurements was carried out using ZPlot (21) and the

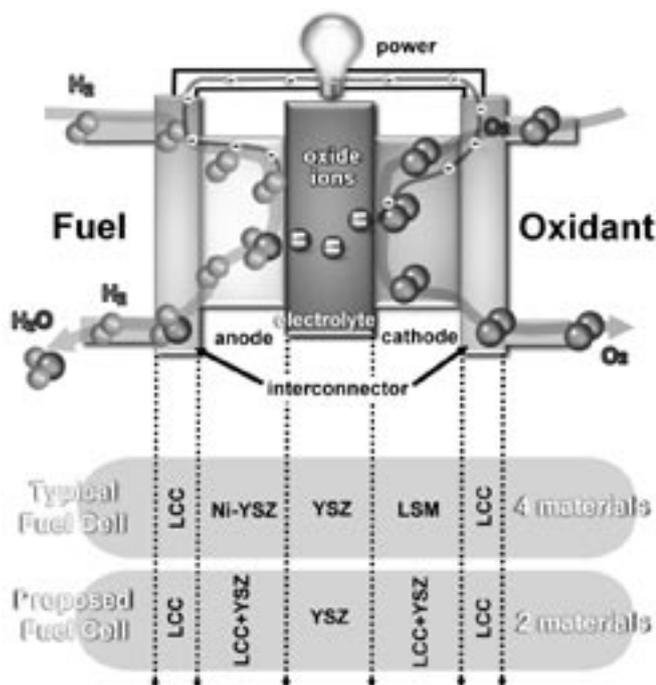


Fig. 1- Scheme of a Solid Oxide Fuel Cell, with the components for traditional and new proposed configuration.

impedance data were analysed using ZView software (22).

### 2.3. Assembly of cells of Fuel cell Test or polarisation measurements

The as-prepared powders, LCC, LCC or YSZ, were mixed with a Decoflux(WB41, Zschimmer and Schwartz) binder to obtain a slurry, which was used to paint electrodes onto a YSZ dense pellet, in symmetrical configuration. Samples were fired at 1200°C, for 2 hours. After that, a Pt-based ink was used to paint a current collector in both sides and fired at 950 °C for 2 hours.

### 2.4. Symmetrical Measurements, polarisation resistances

The polarisation measurements and the fuel cell tests were performed on a two-electrode arrangement (17). The ASRs were obtained under symmetric atmospheres. a.c. impedance spectroscopy of the electrochemical cell was carried out using a Solartron 1260 FRA, at open circuit voltage (OCV), in the  $10^0$ - $10^4$  Hz frequency range using a 50 mV amplitude a.c. signal.

### 2.5. Fuel cell tests

Fuel cell tests have been performed on a two-electrode arrangement (17). Tests were carried out using humidified 5% $H_2$ , pure  $H_2$  and  $CH_4$  as fuels and  $O_2$  as oxidant, at temperatures ranging between 850 and 950°C. The sample was sealed using a ceramic-based material (Ceramabond 668, Aremco). I-V plots were recorded by cyclic-voltammetry at a scan rate of 10 mVs<sup>-1</sup>, using a Zahner IM6ex unit. The water content was fixed by bubbling the gas through a humidifier thermostated at 20 °C. The concentration of water at that temperature was estimated as 2.3%. Fuel cell tests were performed after reduction of the anode materials in humidified 5% $H_2$  for several hours.

### 2.6. Surface Electron Microscopy (SEM)

Electron Microscopy images were obtained after fuel cell testing to check microstructure and connectivity between electrodes and electrolyte, or any evidence of degradation. The samples were cut and the cross sections were covered with a fine gold layer by sputtering. A Scanning Microscope, Jeol LTD., Model JSM-6300 was used.

## 3. RESULTS AND DISCUSSION

Figure 2 shows the Rietveld refinement for XRD pattern powder XRD patterns of  $La_{0.7}Ca_{0.3}CrO_3$  prepared at 1400°C. Refinement was carried out in an orthorhombic unit cell, Pnma space group,  $a=5.4391(9)\text{Å}$ ,  $b=7.691(1)\text{Å}$ ,  $c=5.4580(8)\text{Å}$ . In agreement with the data reported in the literature (23).

Figure 3 shows the XRD patterns corresponding to 1:1 LCC-YSZ composites. The main diffraction peaks do not shift significantly and moreover there are no extra peaks, which may indicate that no reactions occur between the electrolyte and the electrode at least up to 1400°C. Although no degradation of LCC was observed in our short-term experiments under reducing further investigations after long-term operation are highly demanded to clarify this issue as reported by Primdahl et al. (24)

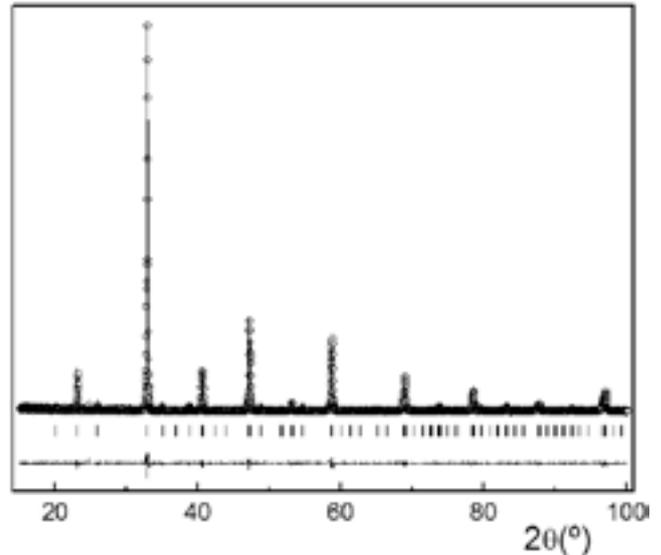


Fig. 2- XRD pattern Rietveld refinement for  $La_{0.7}Ca_{0.3}CrO_3$  prepared at 1400°C.

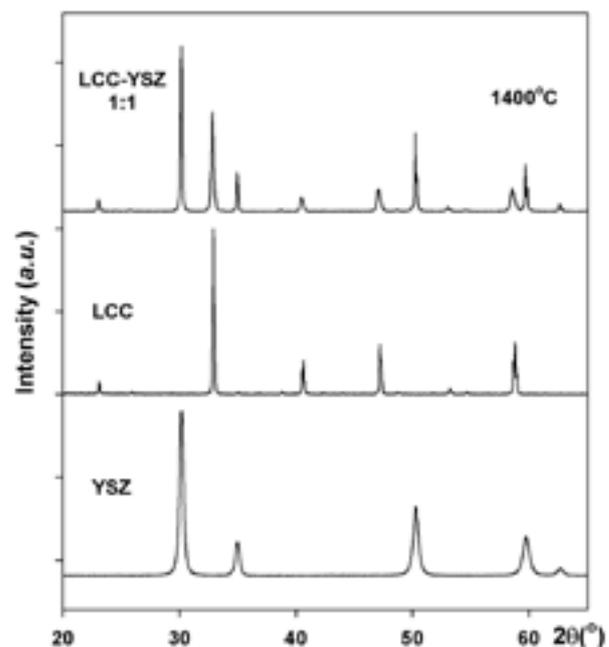


Fig. 3- XRD patterns for (a) YSZ, (b) LCC and composite of (c)  $La_{0.7}Ca_{0.3}CrO_3$ -YSZ, ratio 1:1 (w/w), fired at 1400°C.

The Nyquist plots of a dense  $La_{0.7}Ca_{0.3}CrO_3$  pellet only show the presence of inductive effects indicating predominant electronic contribution in this material. Thus the overall resistance, under both air and 5% $H_2$  conditions, was obtained from the high frequency x-intercept. This agrees with the data reported in the literature (1,2). Thus just an approximate evaluation of the conductivity can be obtained from the impedance data, Figure 4. The lower value of the conductivity, under reducing conditions is due to the oxygen loss that causes a reduction of the charge carrier concentration, thus decreasing the conductivity. On the other hand, the  $\log(\sigma T)$  exhibits a linear dependence with  $1/T$ , which indicates a thermally-activated hopping polarons mechanism, with an activation energy of 0.15eV.

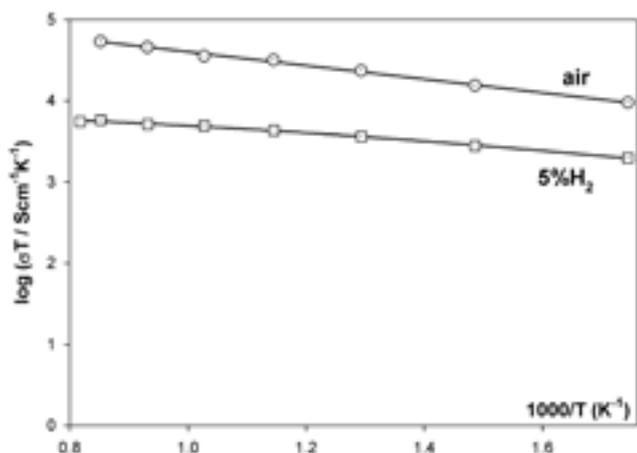


Fig. 4- Overall conductivity of LCC vs. Temperature obtained from impedance measurements.

A symmetrical fuel cell was assembled using the same material as both anode and cathode. The performance was evaluated considering a range of atmospheric conditions, Figure 5. As expected, the cell does not show an impressive performance under methane, in agreement with the reported low catalytic activity of chromites towards hydrocarbon oxidation (19-20). However, the performance under humidified pure hydrogen is rather promising, approximately 110mWcm<sup>-2</sup>, in a thick electrolyte cell.

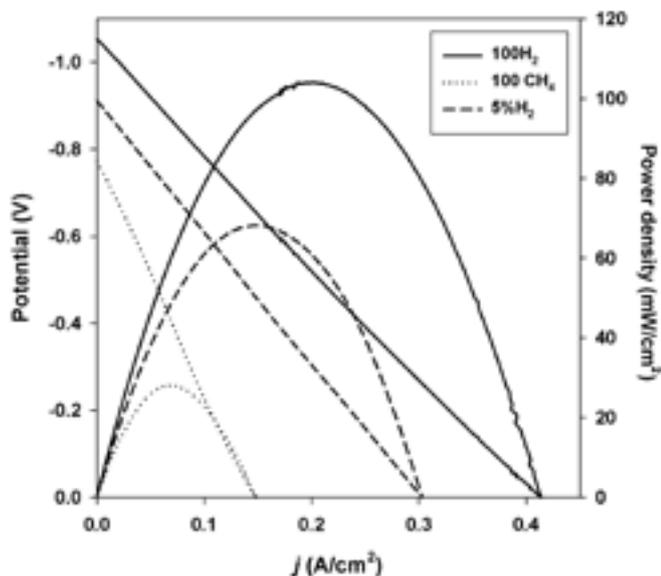


Fig. 5- Fuel Cell test performances using symmetrical materials, at 950°C, with different gases.

An estimation of the performance, for practical applications using a thin YSZ electrolyte, i.e. 100µm, can be calculated taking into account all the losses that may affect the overall performance, Eq.1, and the maximum power given by the product of the current (i) and the corresponding potential E.

$$E = OCV - i R_{\Omega} - \eta_{anode} - \eta_{cathode} = OCV - i (R_{\Omega}^{electrolyte} + R_{\Omega}^{anode} + R_{\Omega}^{cathode}) - i (R_p^{anode} + R_p^{cathode})$$

Eq.1

where: E is the potential of the cell; OCV is the Open Circuit Voltage; R<sub>Ω</sub> includes all the ohmic losses coming from electrolyte, anode and cathode and is usually known as Series Resistances (R<sub>s</sub>). If the electrode materials have a high electronic conductivity, this value will be given by the electrolyte resistance; η represents the corresponding anodic or cathodic overpotentials and it is given by the product of the electrical current and the corresponding polarisation resistance (R<sub>p</sub>), which strongly depends on the catalytic activity and the microstructure of the electrodes. Considering these parameters, an approximation for the maximum performance (1) can be obtained using Eq.2.

$$P_{max} = \frac{OCV^2}{4 R_{total}} = \frac{OCV^2}{4 (R_{\Omega}^{electrolyte} + R_{\Omega}^{anode} + R_{\Omega}^{cathode} + R_p^{anode} + R_p^{cathode})}$$

Eq.2

Given that all the parameters in Eq.1 are known, and considering a 100µm-thickness YSZ, the expected performance can reach a value of 300mWcm<sup>-2</sup>. Similarly, the expected maximum power density for methane is rather modest, i.e. 50mWcm<sup>-2</sup>.

On the other hand, the (P<sub>max</sub>)<sub>e</sub> / (P<sub>max</sub>)<sub>h</sub> ratio may be considered as an estimation of the efficiency of the materials under study. Where (P<sub>max</sub>)<sub>e</sub> is the maximum experimental value of the power. And (P<sub>max</sub>)<sub>h</sub> is the maximum power that can be obtained considering that we are using electrodes with the highest possible efficiency. In that case, the corresponding overpotentials and polarisation resistances can be considered as negligible, and the corresponding Series Resistance is controlled by the electrolyte resistance. Thus, the highest values of E and P<sub>max</sub> will be given by:

$$E_h = OCV - i R_{\Omega}^{electrolyte}$$

Eq.3

$$(P_{max})_h = \frac{OCV^2}{4 R_{\Omega}^{electrolyte}}$$

Eq.4

As can be observed in Table I, only the values obtained under pure hydrogen show an acceptable performance. At 950°C the polarisation contribution from the anode and cathode is approximately 0.8Ωcm<sup>2</sup> and probably it might be easily decreased to the half by adding ceria-based material to the original composite (18) as these materials are known to exhibit ionic conductivity and certain catalytic activity towards fuel oxidation. In 5%H<sub>2</sub>, the values are larger as a consequence of the lower concentration of the electroactive gas. Therefore, changing from 100%H<sub>2</sub> to 5%H<sub>2</sub> causes a reduction in the (P<sub>max</sub>)<sub>exp</sub> / (P<sub>max</sub>)<sub>hig</sub> ratio to just a 10%. This implies that it is very likely that the value in pure hydrogen could be largely improved compared to 5%H<sub>2</sub>. The use of pure H<sub>2</sub> should usually allow a reduction of the polarisation values by 3 compared to the value obtained under 5%H<sub>2</sub>. Nevertheless, Table I shows the very bad results obtained under methane fuelling. This could be explained mainly by two factors: the material does not have almost any catalytic

activity towards hydrocarbon oxidation (19-20) and/or the microstructure is very poor, i.e. it does not show an adequate porosity and/or a bad pore distribution.

Indeed, the SEM images corresponding to the electrode-

TABLE I. ELECTROCHEMICAL VALUES OBTAINED FOR A SYMMETRICAL FUEL CELL: LCC-YSZ/YSZ/YSZ-LCC, AT 950°C AND 850°C, UNDER DIFFERENT GAS COMPOSITIONS. THE BEST PERFORMANCE SO FAR IS OBTAINED AT 950°C UNDER PURE HYDROGEN. (YSZ ELECTROLYTE THICKNESS 2mm)

T (°C)	gas	OCV (V)	$(P_{max,e})/(P_{max,h})$	$(R_{p,anode+cathode})$ ( $\Omega\text{cm}^2$ )
950	100% H <sub>2</sub> <sup>a</sup>	1.090 <sup>a</sup>	69.5 %	0.809
	5% H <sub>2</sub> <sup>b</sup>	0.932 <sup>b</sup>	60.9 %	1.187
	100% CH <sub>4</sub> <sup>***</sup>	0.78 <sup>c</sup>	36.0 %	3.281
850	100% H <sub>2</sub> <sup>a</sup>	1.106 <sup>a</sup>	46.2 %	3.142
	5% H <sub>2</sub> <sup>b</sup>	0.961 <sup>b</sup>	41.2 %	3.866
	100% CH <sub>4</sub> <sup>***</sup>	0.75 <sup>c</sup>	19.5 %	11.20

<sup>a,b</sup> using air as reference

<sup>c</sup> experimental value

The concentration of water is always estimated as 2.3%, and once the equilibrium is reached, the concentration of the other gases in gas inlet was: <sup>a</sup> 97.7% H<sub>2</sub>, <sup>\*\*</sup> 4.9% H<sub>2</sub> and 92.8% (N<sub>2</sub>), <sup>\*\*\*</sup> 97.7% CH<sub>4</sub>

electrolyte interface, Figure 6, reveal a very dense electrode material. This could certainly explain the very modest performances obtained under hydrocarbons. H<sub>2</sub> is a very small molecule and it can easily diffuse through the material. However, methane and other larger gas molecules produced during the oxidation, e.g. CO, CO<sub>2</sub>, H<sub>2</sub>O, cannot diffuse through the dense electrode, hence the corresponding polarisations are considerably worse. Nevertheless, this also indicates that the material is open to further improvement by optimising

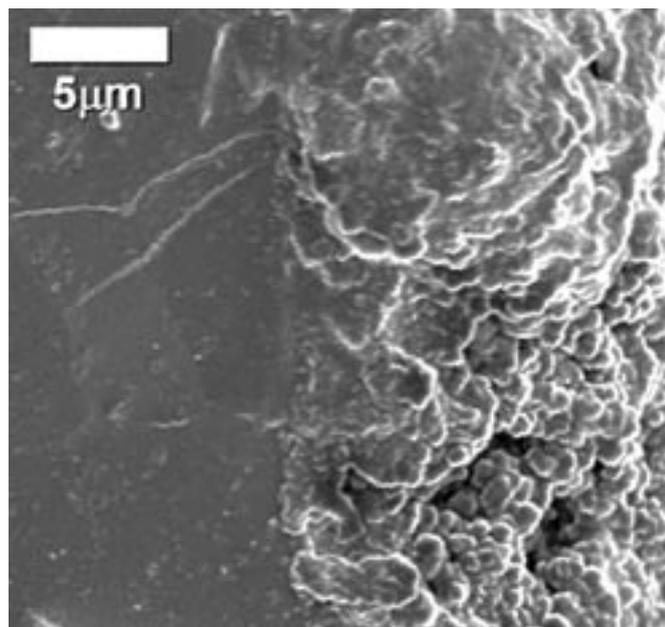


Fig. 6- SEM image of the cross-section, showing the interface electrolyte-electrode. In this case a poor microstructure, with very low porosity, could be observed for a LCC-based anode material. That could easily explain the low performances values obtained.

the microstructure. The dense electrode material obtained suggested that is necessary to use a pore former additionally to the binder used, such as graphite, and probably in high ratios, 30-50%. On the other hand, the catalytic activity could be improved, producing high specific surface area chromites, through precursors synthetic methods (25-28) as alternative to the solid state route reported in this work.

#### 4. CONCLUSIONS

It has been shown that a classical interconnector material has the potential to become a possible symmetrical electrode for Solid Oxide Fuel Cells. The approach renders promising performance in excess of 100mW/cm<sup>2</sup> using thick YSZ-electrolyte, at 950°C under pure hydrogen. An expected value of 300mWcm<sup>2</sup> was predicted for the same configuration, although using a thin YSZ layer of 100μm.

Despite the very low catalytic activity under hydrocarbon, the performance of the material could be enhanced by optimising the microstructure, which was rather poor in the present case.

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## SOCIEDAD ESPAÑOLA DE CERÁMICA Y VIDRIO

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