



Short communication

## Development of electrolyte-supported intermediate-temperature single-chamber solid oxide fuel cells using $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$ (Ln = Pr, La, Gd) cathodes

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## ABSTRACT

Iron–cobalt-based perovskite oxides with general formula  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  (where Ln = La, Pr and Gd) have been investigated for their application as intermediate-temperature cathodes in solid oxide fuel cells (SOFCs). Powdered samples of these materials were synthesized by a novel gel combustion process and then characterized by X-ray powder diffraction (XPD) and scanning electron microscopy (SEM). XPD patterns were satisfactorily indexed with an orthorhombic  $\text{GdFeO}_3$ -type structure and, for all samples, a mean particle size of less than  $1\ \mu\text{m}$  was estimated from the SEM data. Experimental single-chamber SOFCs using with these materials as cathodes and NiO–SDC (samaria-doped ceria) and SDC as anode and electrolyte, respectively, were evaluated at  $600\ ^\circ\text{C}$  in a methane/oxygen mixtures. Peak power densities of 65.4, 48.7 and  $46.2\ \text{mW cm}^{-2}$  were obtained for  $\text{Ag}|\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}|\text{SDC}|\text{NiO–SDC}|\text{Pt}$  cells with Ln = Pr, La and Gd, respectively. The relatively high power density obtained for the Pr compound shows that it could be an interesting material for cathode of single-chamber SOFCs.

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## 1. Introduction

Depending on the specific composition, perovskite oxides of general formula  $\text{Ln}_{1-x}\text{A}_x\text{BO}_{3-\delta}$  (Ln = rare earth; A = Sr, Ca, Ba; B = Cr, Mn, Fe, Co or Ni) have been found to exhibit a wide variety of magnetic and electronic properties. Members of the family  $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$  (LSM) are archetypal electronic conductors and have been extensively used as cathodes in  $\text{ZrO}_2$ -based solid oxide fuel cells (SOFCs) [1]. However, the lack of oxide-ion vacancies and its conductivity at the SOFC working temperature, makes it necessary to use thick and porous electrodes containing an array of triple-phase boundaries where gas, electrolyte and electrode meet, which limits their efficiency. Their use is further hindered by the formation, at the typical annealing temperatures for preparation of SOFCs ( $>1200\ ^\circ\text{C}$ ), of the insulating pyrochlore oxide  $\text{La}_2\text{Zr}_2\text{O}_7$  at the boundary with the yttria-stabilized zirconia (YSZ) electrolyte. This poses a major problem in the fabrication processes. Since the reactivity depends on the Ln and Sr content, the use of  $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  cathodes, which exhibit better compatibility with YSZ, is an interesting option [2].

The use of lanthanum cobaltites as cathodes has been intensively investigated in the last years [3]. Initially, research focused on the

$\text{La}_{1-x}\text{Sr}_x\text{CoO}_{3-\delta}$  (LSC) family due to its considerable ionic conductivity and sizeable electronic conductivity, which makes LSC materials interesting mixed ionic–electronic conductors (MIECs) [4]. Their disadvantage, however, is their high reactivity with the commonly used YSZ electrolyte, which restricts its use and makes it necessary the search for alternative electrolytes: LSC cathodes combined with ceria-based ceramic electrolytes – particularly samaria-doped ceria (SDC) – have been shown to outperform LSM cathodes.

More recently, further research has been aimed at attempting to overcome the shortfalls associated with LSC cathodes by replacing La by other lanthanide such as Gd, Sm or Dy. Thus, for example, the conductivity of  $\text{Gd}_{1-x}\text{Sr}_x\text{CoO}_3$  has been investigated by Takeda et al., who found that its performance as cathode material, although somewhat worse than that of LSC, is still acceptable for a working SOFC [5]. On the other hand, this material reacts with YSZ forming the phase  $\text{SrZrO}_3$ , which is a great disadvantage for the cathode material. Nevertheless, the  $\text{GdCoO}_3$  compound (non-Sr-doped) presents an acceptable behaviour even at temperatures as high as  $1000\ ^\circ\text{C}$ , without any reaction products in the electrolyte/electrode interface after long operating times, turning it into a good cathode for intermediate-temperature SOFC.

Finally, a large deal of research has been devoted to the development of new materials that overcome the technological problems associated with LSC materials at relatively high-temperatures. In general, this effort has been focused on B-doped perovskites, among which the  $\text{La}_{1-x}\text{Sr}_x\text{Co}_{1-y}\text{Fe}_y\text{O}_3$  (LSCF) series features as one of the

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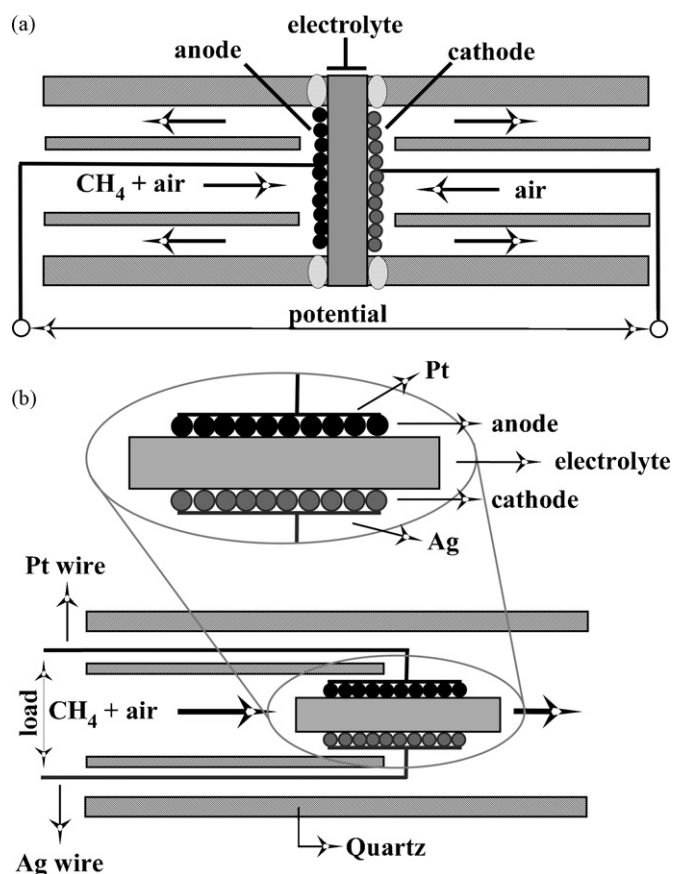


Fig. 1. Schematic illustrations of SOFCs designs: (a) dual-chamber and (b) single-chamber.

most promising systems (appreciating a decrease of the conductivity with the iron content) [6,7].

This paper describes the initial stages of the development of a new series of high-performance  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  ( $\text{Ln} = \text{La}, \text{Gd}, \text{Pr}$ ) cathodes (hereafter LnSFC) for SOFC use. The samples under study were synthesized by a novel gel combustion process and characterized by X-ray powder diffraction (XPD) and scanning electron microscopy (SEM). Their performance as cathodes was evaluated in an experimental single-chamber SOFCs (SC-SOFCs). While conventional dual-chamber SOFCs rely on the strict separation of fuel and oxidant (Fig. 1b), SC-SOFCs consist of only one gas chamber and, therefore, both electrodes are exposed to a uniform mixture of fuel and oxidant gases (Fig. 1b). The operation of these cells relies on the use of highly selective cathode and anode catalysts. In the case of the anode, it is possible to take advantage of the presence of  $\text{O}_2$  to produce the partial oxidation of the fuel (a hydrocarbon, usually methane), reaction that can be catalyzed at intermediate temperatures. The concept of SC-SOFC, put forward by Hibino [8–12], overcomes many difficult problems of conventional SOFCs that are unresolved so far.

## 2. Experimental

### 2.1. Synthesis and structural characterization

Powders of LnSFC compounds were prepared following a novel combustion route using arginine as fuel. Appropriate amounts of the nitrates [ $\text{Pr}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ ;  $\text{Sr}(\text{NO}_3)_2$ ;  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  and  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ] were dissolved in distilled water and arginine was added to the solution in the proportion required for a stoichiomet-

ric combustion reaction. A concentrated gel formed as the solvent evaporated, which finally burned due to the exothermic redox reaction between nitrate ions and the fuel. This exothermic reaction was relatively mild except for the case of Gd, for which a vigorous flame was observed. The resulting powders were calcined in air at  $800^\circ\text{C}$  for 2 h.

The crystal structure of the as-synthesized powders was determined by X-ray powder diffraction (XPD). Data were collected with a Philips X'Pert-MPD diffractometer operated with  $\text{Cu K}\alpha$  radiation and fitted by the Rietveld least-square method using the FULLPROF program [13]. The microstructure of the obtained particles was observed by scanning electron microscopy (SEM) using a JEOL JSM-6400 microscope at 20 kV accelerating voltage.

### 2.2. Manufacture of the single-chamber SOFCs

Based on the previous results on the system  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  ( $\text{Ln} = \text{La}, \text{Pr}, \text{Gd}$ ), SDC was chosen as electrolyte material [14]. SDC electrolytes were prepared from commercial  $\text{CeO}_2$ -10 mol%  $\text{Sm}_2\text{O}_3$  powders (Nextech Materials) by uniaxial pressing at 200 MPa followed by sintering in air at  $1500^\circ\text{C}$  during 2 h. The relative density of the obtained pellets was of 95%. The surface of these electrolyte pellets was polished with 100 grit SiC paper in order to control their thickness and get a good adhesion to the cathode. The electrolytes used in this study were polished down to a thickness of about 0.3 mm in order to increase the power density.

NiO-SDC (Nextech Materials—NiO:SDC=60:40 in weight) mixed with the vehicle ink (Nextech Materials) composite was used as anode. The resulting paste, of suitable fluidity, was painted onto one of the faces of the electrolyte pellets and then sintered at  $1250^\circ\text{C}$  for 2 h.

The as-synthesized LnSCF powders were dispersed in a commercial organic vehicle (Nextech Materials) forming a paste. For the praseodymium sample, a powder/vehicle ratio of 1:0.50 in weight was used to achieve a homogeneous paste. In case of the compounds of gadolinium and lanthanum, it was necessary to dilute the powder in larger quantity of vehicle, obtaining a powder/vehicle ratio of 1:0.75. This paste was painted on the surface of SDC pellets as thinly as possible with a brush and, finally, it was sintered at  $1050^\circ\text{C}$  during 90 min to ensure relatively high porosity and good adhesion. The choice of the above-mentioned conditions for the preparation of LnSCF cathodes materials was based on their performance for electrochemical oxygen reduction evaluated in a previous study by electrochemical impedance spectroscopy (EIS) [14].

Finally, the current collectors were painted with Pt and Ag on the anode and cathode, respectively. Taking into account that it is very important to keep the selectivity of the electrodes, this choice was based on the fact that Ag exhibits lower catalytic activity for methane oxidation than Pt. Ag coating was sintered at  $750^\circ\text{C}$ , whereas Pt one was treated at  $950^\circ\text{C}$ . Five cells were tested to confirm the reproducibility of the system.

Methane ( $\text{CH}_4$ ) is a viable fuel for SC-SOFCs, as has been demonstrated for several authors, and the development of devices using this fuel is very important for several applications. For this reason, a mixture of methane/air was chosen to study the operation of the cells that are described along this paper. It was used a mixture of  $\text{CH}_4$  and air in a  $\text{CH}_4:\text{O}_2$  ratio of 2:1 since this is the optimal condition to promote the partial oxidation of  $\text{CH}_4$ . After several tests, we chose a total flow rate of  $300 \text{ ml min}^{-1}$ .

Details of our experimental setup are described in Fig. 1b. The outer and inner quartz tubes have inner diameters equal to 40 and 15 mm, respectively. In order to let the gas mixture flow on both sides, the current collector wires were welded to the corresponding side of the cell.

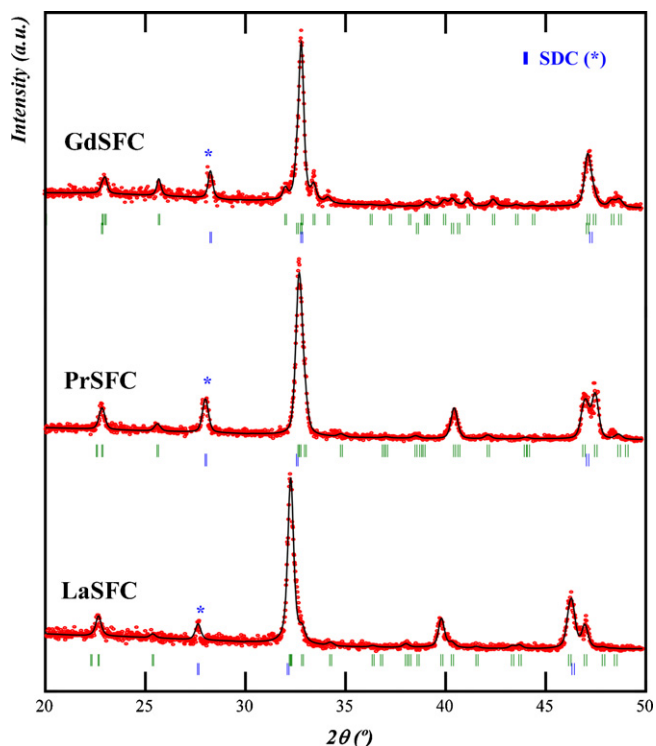


Fig. 2. XPD patterns of LnSFC samples showing observed (points) and calculated (line) profiles.

### 3. Results and discussion

#### 3.1. X-ray powder diffraction

As explained above, XPD data refinements were performed by the Rietveld method. The patterns of the different  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  ( $\text{Ln}=\text{La}, \text{Pr}, \text{Gd}$ ) samples are shown in Fig. 2. Lattice parameters are summarized in Table 1. In general, XPD results showed high crystallinity for powders fired at 1050 °C.

Table 1

Crystal structure and lattice parameters for  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  ( $\text{Ln}=\text{La}, \text{Pr}, \text{Gd}$ ).

Ln	Structure	a (Å)	b (Å)	c (Å)
La	Orthorhombic	5.3376 (29)	5.4353 (25)	7.7193 (26)
Pr	Orthorhombic	5.3732 (15)	5.4144 (19)	7.7069 (19)
Gd	Orthorhombic	5.3155 (30)	5.5384 (31)	7.6314 (48)
	Rhombohedral	5.4393 (22)		13.2641 (11)

XPD patterns were indexed with a single orthorhombic orthoferrite structure ( $\text{SG}=\text{Pbnm}$ ) in the case of the La and Pr samples, whereas for  $\text{Ln}=\text{Gd}$  segregation into two phases ( $\text{SG}=\text{Pbnm}$  and  $\text{R}\bar{3}\text{c}$ ) was observed. A similar result has been previously reported in the  $\text{Gd}_{1-x}\text{Sr}_x\text{FeO}_{3-\delta}$  system where the Gd/Sr size mismatch induces a structural strain that leads to phase segregation [15]. In addition, it has been reported that the  $(\text{Gd}_{0.6}\text{Sr}_{0.4})_{0.99}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  compound consists of two phases with different Sr content [16].

#### 3.2. Scanning electron microscopy

The effect of grain size on the conductive properties of electrode materials is a well-established fact: their performance improves with decreasing particle size. Porosity is also a major factor affecting it, as the presence of pores facilitates the gas transfer thus increasing the number of active reaction sites.

The results of the morphological study of the LnSFC phases are shown in Fig. 3. The La and Pr phases exhibit a very similar appearance with relatively homogeneous particle size. The Gd compound, on the other hand, shows a more heterogeneous aspect with regions of larger sintered grains. At the same time, La and Pr phases present a higher volume of pores than that of Gd, which, in principle, will favour transport within the electrode. These SEM observations seem to agree with the presence of two phases detected for the Gd compound in our XPD experiments and also reported by other authors [15,16], as discussed in the above section: two phases were observed by SEM, one with small average particle size (less than 1  $\mu\text{m}$ ) and the other one form by relatively large particles (3–4  $\mu\text{m}$ ). These results agree with those described in the literature [16].

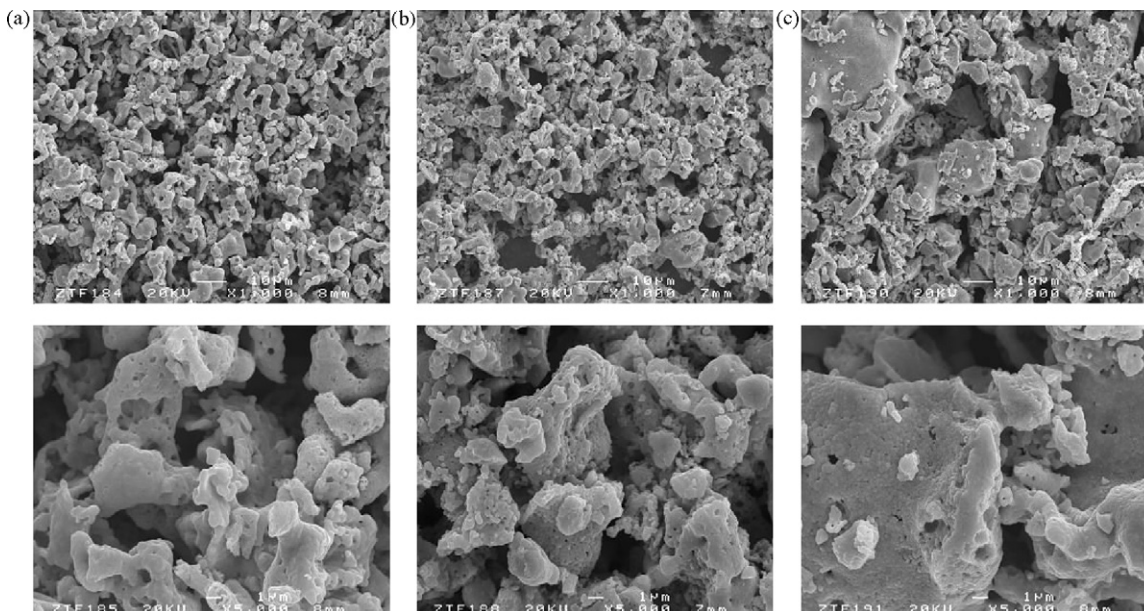


Fig. 3. SEM top-view micrographs of LnSFC thick films on SDC electrolytes: (a) LaSFC, (b) PrSFC and (c) GdSFC.



### 3.3. Electrochemical performance of single-chamber SOFCs

The  $\text{Ag|Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}\text{|SDC|NiO-SDC|Pt}$  SC-SOFCs were placed in the support described in Fig. 1b. The oxygen partial pressures at the anode and cathode sides in these conditions cannot be assumed as fixed, since they depend on the catalytic activity of the electrodes, the temperature and fuel and oxidant ratio and its flow rate. For the working conditions used in this study ( $\text{CH}_4:\text{O}_2$  ratio of 2:1), the most favourable reaction is the partial oxidation of  $\text{CH}_4$ :



$\text{H}_2$  and  $\text{CO}$  are then oxidized to  $\text{H}_2\text{O}$  and  $\text{CO}_2$ , respectively, by  $\text{O}^{2-}$  anions coming from the electrolyte.

However, the real situation can be more complex, particularly if the working temperature is relatively high, since many other chemical reactions can happen between the products of the above reaction (Eq. (1)) and the reagents. Besides, the selectivity of the electrodes can be affected if the working temperature increases, particularly because the cathode can start to promote the oxidation of the fuel. In addition, several dynamic phenomena taking place on the Ni catalysts in the case of partial oxidation of  $\text{CH}_4$  have been reported [17,18]. For these reasons, it is important to pay attention to the selection of an appropriate temperature in order to control the cyclic oxidation and reduction processes occurring on the surface of the anode.

The working temperature also affects the transport properties of the electrolyte. In the case of SDC electrolyte, for  $T > 700^\circ\text{C}$ , a sizeable electronic conductivity takes place. This causes a decrease of the open-circuit voltage of the cell, thus reducing its power density.

Taking into account all the above factors, we fixed the working temperature at a moderate value of  $600^\circ\text{C}$ . A recent study of the catalytic properties of NiO-SDC composites for partial oxidation of methane has shown that these materials have good performance at this temperature [19].

The performance of the  $\text{Ag|Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}\text{|SDC|NiO-SDC|Pt}$  SC-SOFCs at  $600^\circ\text{C}$  under a  $\text{CH}_4/\text{air}$  mixture with a  $\text{CH}_4:\text{O}_2$  ratio of 2:1 is shown in Fig. 4. Open-circuit voltages (OCVs), short-circuit currents and peak power densities obtained for each compound are listed in Table 2. After the initial reduction of the anode, SC-SOFCs with La, Pr and Gd cathodes exhibited OCV values of 756, 730 and 675 mV, respectively. The cell prepared with La compound showed the highest OCV value, but it decayed fast as the applied current density was increased, presenting a short-circuit

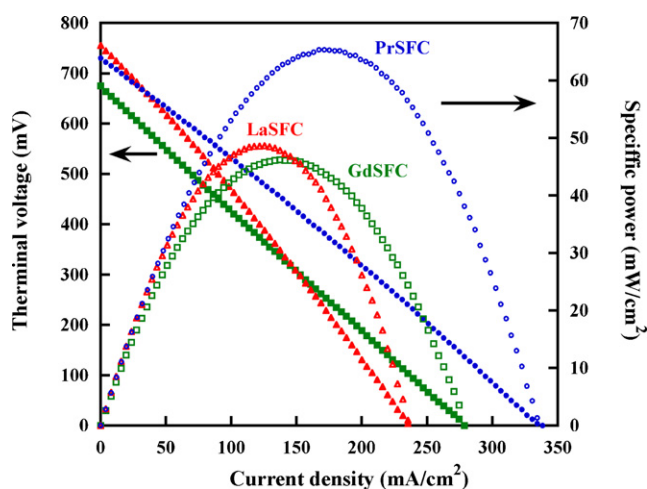


Fig. 4. Discharge curves at  $600^\circ\text{C}$  under a  $\text{CH}_4/\text{air}$  mixture with a  $\text{CH}_4:\text{O}_2$  ratio of 2:1 (flow rate:  $300\text{ ml min}^{-1}$ ) of electrolyte-supported single-chamber SOFCs for  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  ( $\text{Ln} = \text{La, Pr, Gd}$ ) cathodes, SDC electrolyte and NiO-SDC composite anode.

Table 2

Performance of SC-SOFCs at  $600^\circ\text{C}$  with  $R_{\text{mix}} = 2$  under  $300\text{ ml min}^{-1}$  of a SDC electrolyte-supported cell using Ni-SDC cermet and  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  ( $\text{Ln} = \text{La, Pr, Gd}$ ) materials as anode and cathode, respectively.

Cathode material	OCV (mV)	Short-circuit current ( $\text{mA cm}^{-2}$ )	Peak power density ( $\text{mW cm}^{-2}$ )
$\text{Pr}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$	730	171	65.4
$\text{La}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$	756	125	48.7
$\text{Gd}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$	675	137	46.2

current of  $125\text{ mA cm}^{-2}$ . For the  $\text{Gd}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  material, a lower OCV value was observed (675 mV), but the voltage drop in this cell was lower than that observed for the La compound. On the other hand, the cell using the  $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  cathode shows an OCV of 730 mV, with a voltage drop similar to the gadolinium cell. Peak power densities of 48.7, 65.4 and  $46.2\text{ mW cm}^{-2}$  were obtained for the La, Pr and Gd cells, respectively. The above results agree with the behaviour observed in our previous EIS study of LnSFC materials, since the Pr compound exhibited the lowest area-specific resistance ( $\text{ASR} = 0.30\ \Omega\text{ cm}^2$  at  $700^\circ\text{C}$ ), followed by that of La ( $\text{ASR} = 0.42\ \Omega\text{ cm}^2$  at  $700^\circ\text{C}$ ) and, finally, that of Gd ( $\text{ASR} = 1.05\ \Omega\text{ cm}^2$  at  $700^\circ\text{C}$ ) [14].

To check the reproducibility of the  $\text{Ag|Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}\text{|SDC|NiO-SDC|Pt}$  SC-SOFCs, five identical cells were prepared and tested for several hours. A highly reproducible cell performance was observed in all the studied systems. Under the described conditions, it has been observed that the OCV decreases slightly after 10 h operation time and the cell performance decayed relatively rapid with increasing current density. The complete study of the stability of these systems will be carried out in further works under way.

The obtained OCV values are similar to those reported in cells based on doped ceria electrolytes, which are relatively low due to the reduction of the  $\text{Ce}^{4+}$  to  $\text{Ce}^{3+}$  in the SDC electrolyte under the operation conditions. Nonetheless, in this type of systems using ceria the discharge is slower and the ohmic resistance is lower than those observed using YSZ or LSGM ( $\text{La}_{0.9}\text{Sr}_{0.1}\text{Ga}_{0.8}\text{Mg}_{0.2}\text{O}_3$ ) as electrolyte [11].

The obtained results show the clear influence of the cathode composition on the OCV and the performance of the SC-SOFCs. Highly active standard materials used in conventional SOFCs, such as  $\text{La}_{0.8}\text{Sr}_{0.2}\text{MnO}_{3-x}$  (LSM) and  $\text{La}_{0.8}\text{Sr}_{0.2}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-x}$  (LSCF), are not applicable in SC-SOFC because they do not exhibit enough selectivity [20]. Besides, we have shown that the  $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  compound shows higher performance than that reported for conventional cathode materials [20,21]. It, therefore, appears as an interesting potential cathode material for SC-SOFCs.

The main drawback of SC-SOFCs is their very low fuel utilization. Typical values are usually lower than 1% and even cells with high performance only reach about 3.6% [22]. In the present work, the fuel utilization was about 0.4%, similar to the values that can be calculated from the results reported by other authors (see, for example, the summary of results reported in Ref. [22]). However, a recent numerical study [23] demonstrated that high efficiency and fuel utilization can be obtained by optimizing all the relevant parameters, such as  $\text{CH}_4$  flow rate,  $\text{CH}_4:\text{O}_2$  ratio, the use of a balance gas, the flow geometry, etc. In addition, the same study showed that much better performance can be obtained in stacks (for example, a simple two-cell one). Experimental works confirming these results would be very important in order to enable the use of SC-SOFCs in practical applications.

### 4. Conclusions

The perovskite-type  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  ( $\text{Ln} = \text{La, Gd, Pr}$ ) compounds have been evaluated for their use as cathodes for

intermediate-temperature SC-SOFCs. This series of compounds was synthesized using a novel gel combustion process. For Ln = La and Pr, single orthorhombic phases were found, while for Gd compound an orthorhombic–rhombohedral phase segregation was detected. These LnSCF powders were used to prepare pastes with a commercial vehicle, which were then deposited over SDC electrolytes and sintered at high-temperature. Scanning electron microscopy showed that the surface of the painted pellets are highly porous, facilitating gas transfer and maximizing the number of active sites for the oxygen reduction reaction.

Electrolyte-supported SOFCs with  $\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  (Ln = La, Gd, Pr) materials as cathode have been shown to provide good performance when operated in a single-chamber design. The peak power density of  $\text{Ag}|\text{Ln}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}|\text{SDC}|\text{NiO}-\text{SDC}|\text{Pt}$  cells, evaluated at 600 °C, was of 65.4, 48.7 and 46.2  $\text{mW cm}^{-2}$  for Pr, La and Gd cathodes, respectively. However, our preliminary study clearly suggests new prospects for the  $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$  material as cathode in intermediate-temperature single-chamber SOFCs.

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