



Original scientific paper

Preparation and optimization of $\text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{1-y}\text{Co}_y\text{O}_{3-\delta}$ cathodes for intermediate temperature solid oxide fuel cells

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Received: September 27, 2024; Accepted: October 30, 2024; Published: November 7, 2024

Abstract

It is shown in this work that a synthetic route based on the auto-combustion of an ethylene glycol-metal nitrate polymerized gel precursor can be efficiently used to easily produce a range of $\text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{1-y}\text{Co}_y\text{O}_{3-\delta}$ nanopowders at moderate temperatures. We have been able to determine on air-sintered samples the effect of sintering temperature on the microstructure. At sintering temperatures as low as 1100 to 1200 °C, grains are well defined with a uniform round spherical morphology and have a homogeneous sub-micrometer size distribution showing a highly densified microstructure. The electronic conductivity and thermal expansion coefficients (TEC) of sintered LSCF samples have been determined according to the variation of Fe/Co composition. Both measures clearly increase with the Co content. These materials also must exhibit chemical stability with electrolytes, most commonly used for intermediate temperature solid oxide fuel cells (IT-SOFCs). In this way, the material obtained is optimized in terms of chemical homogeneity and good stoichiometric control, microstructural characteristics of sintered samples, and finally, the adequate cobalt content to avoid high TEC mismatch with other components of the SOFC. This is a crucial issue as it causes an important thermomechanical stress, promotes extensive microcracking and causes significant performance degradation. Finally, these cathodes must exhibit acceptable electrochemical parameters for use in IT-SOFC.

Keywords

Ferro-cobaltite cathodes; electrochemical properties; thermal expansion coefficients; chemical compatibility

Introduction

Fuel cells represent a viable alternative for producing electricity with high efficiency, low pollution and cost-effectiveness. In recent years, among the different types of fuel cells, the interest in solid oxide fuel cells (SOFCs) has increased enormously as a commercially viable power source [1]. Additional advantages of this type of fuel cells are the high tolerance to impurities and the possibility of internal reforming, which leads to the possibility of using different fuels [2]. In addition, the

possibility of producing cogenerative energy from heat waste during the operation process results in being very attractive for this kind of fuel cell [3]. However, considering the high working temperature (around 900 to 1000 °C), some difficulties limit the use of SOFCs, among them the selection of interconnects, sealing materials, coefficient thermal expansion (CTE) mismatch and chemical compatibility of cell components [4]. Therefore, a lower operating temperature (600 to 800 °C) is necessary to reduce manufacturing costs and increase durability. For that purpose, new materials are required that can efficiently operate at a lower temperature, *i.e.*, electrolytes with higher conductivity such as doped-CeO₂ [5] or La(Sr)Ga(Mg)O₃ [6] and also, it is crucial the development of more effective cathode materials with increased electrocatalytic activity [7].

The new cathode materials should be characterized by increased oxide ion transport in addition to high electronic conductivity. La_{1-x}Sr_xFeO₃ (LSF) has demonstrated higher electrical and ionic conductivity than the conventional La_{1-x}Sr_xMnO₃ (LSM), which may extend the cathode reaction sites beyond the triple-phase boundaries [8,9]. In this same way, La_{1-x}Sr_xCoO₃ (LSC) can be considered as a promising cathode for IT-SOFCs with high values of conductivity. However, the good electrocatalytic performance of this Cobalt-based-cathode is somehow limited by a thermal expansion coefficient (TEC) mismatch with other components of the SOFC, causing significant thermomechanical stress between these thermally mismatched components and finally leading to significant performance degradation that may be rapid and catastrophic. TECs of both the electrolyte and electrode layers should be well matched to ensure long-term operational stability of the SOFCs, either by modifying existing cathodes or designing new ones [10]. Considering all the above aspects, alternative materials could be ferrite/cobaltite cathodes such as La_{0.6}Sr_{0.4}Fe_{1-y}Co_yO_{3-δ} (LSFC). The electronic conductivity of these samples in air is characterized by the higher values at increasing Co contents because the activation energy of LSF is higher than that for Co-doped ferrites [11,12]. It must be considered that the presence of Fe helps to minimize mechanical stresses as it can reduce the TEC mismatch. Also, it should be ensured that there are no reaction phases on the electrode/electrolyte interfaces since this could be detrimental to the efficiency of the cell. Furthermore, cation interdiffusion must be precluded because it may negatively modify the properties of the electrode and electrolyte. In recent years, many studies have proposed relevant mechanisms and solutions [13].

On the other hand, the solid-state reaction of the corresponding oxides is an easy synthesis process to obtain ceramic powders of different materials. However, it requires high processing temperatures and the homogeneity of the final synthesized powder is poor. Due to the number of cations involved, the compositional complexity of the materials mentioned above requires a synthesis method that ensures optimal chemical homogeneity and good stoichiometric control in which cations involved are distributed uniformly at an atomic scale. Thus, chemical methods of processing are highly recommended techniques to produce these materials [14-17]. Here, we use a low-cost and simple synthesis alternative method, which was previously studied and has excellent results [18]. It is based on the chelation of complex cations leading to the formation of an intermediate resin, which, on charring and calcining, leads to a sinterable powder that can be efficiently used to easily produce a range of La_{0.6}Sr_{0.4}Fe_{1-y}Co_yO_{3-δ} (LSFC) at moderate temperatures.

Therefore, the purpose of this work is to prepare LSFC cathodes using a low-cost and simple synthesis method with adequate electrochemical properties and thermomechanical compatibility with the electrolyte. First, to optimize these materials, microstructural control is necessary by determining the adequate sintering temperature. In addition, the chemical compatibility between electrodes and electrolytes is an important issue for successfully operating the fuel cells. It is also

crucial to determine the LSFC composition that avoids microstructural problems caused by the TEC's mismatch with the electrolyte and adequate electrochemical performance in terms of electrical conductivity and polarization resistance.

Experimental

$\text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{1-y}\text{Co}_y\text{O}_{3-\delta}$ nanopowders with compositions $y = 0$ to 1 were prepared by the ethylene glycol-metal nitrate polymerized complex process. Aqueous solutions (≈ 0.5 M) of corresponding nitrates were mixed by stirring nitric acid (65 %) and ethylene glycol to make a gel. It was treated thermally at different temperatures [18]. The as-obtained powder was calcined at 900 °C for 5 h and then attrition milled for 1 h in ethanol with zirconia ball media. For sintering, the powders were isopressed in pellets at 200 MPa and thermally treated at temperatures between 1000 and 1200 °C after drying. The powders were characterized by differential thermal (DTA) and thermogravimetric (TG) analysis (Perkin-Elmer 7). The calcined powders and sintered samples were characterized by X-ray diffraction (XRD) with a Bruker D8 Advance diffractometer ($\text{Cu K}\alpha_1$ radiation). After polishing and thermal etching, the microstructures of the sintered samples were examined by Scanning Electron Microscopy (SEM) in a Zeiss Microscope (model DSM 950, Germany). Shrinkage during sintering, at a heating rate of 5 °C min^{-1} without holding, was followed in a dilatometer Netzsch Gerätebau (model 402 EP, Selb-Bayern Germany) by the conventional rampant-holding methods. The TECs were measured using the above-mentioned dilatometer in air from room temperature to 800 °C on air-sintered rectangular shapes. For determining the total electrical conductivity, impedance was measured at 800 °C in argon using a potentiostat/galvanostat (Autolab PGSTAT302 N). The platinum paste is used as a current-collecting electrode on either side of the sintered pellet and the electrode is cured at 950 °C for 2 h before the electrical conductivity measurements. To study the effect on the microstructure of the electrode/electrolyte TEC mismatch, LSFC powders were fixed on the sintered electrolyte of $\text{La}_{0.9}\text{Sr}_{0.15}\text{Ga}_{0.8}\text{Mg}_{0.2}\text{O}_{3-\delta}$ (LSGM) by uniaxial pressing. The sandwich electrode/electrolyte was co-fired at 1200 °C for 5 h and continuously subjected to 10 cycles of rise and fall of temperature (25 to 800 °C). The microstructures of these samples were examined by SEM. Energy dispersive X-ray analysis (EDX) was performed to study the possibility of cations diffusion through the cathode-electrolyte interfaces. The measurements of area-specific resistance (ASR) dependence with temperature were carried out in a half cell with a 3-electrode configuration, using $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$ (CGO, Cerpotech) as electrolyte. In this study, pellets of electrolyte powder were uniaxial pressed and sintered at 1500 °C for 6 hours. A mix of a commercial organic solvent, Decoflux, and the studied electrodes were deposited using a 6 mm-diameter mask on both sides. The electrode-electrolyte pellets were then co-fired at 1200 °C for 6 h. A reference platinum electrode was deposited in the edge of the pellet to make 3-probe electrode polarization measurements. Impedance spectroscopy was carried out with a Metrohm Autolab PGSTAT302 N, employing a signal amplitude of 1 mA across a frequency range of 0.1 to 10^6 Hz and a temperature range of 600 to 950 °C. Energy dispersive X-ray analysis (EDX) was performed to study the possibility of cations diffusion.

Results and discussion

The simultaneous TG/DTA curves of a representative LSFC polymeric gel are shown in Figure 1. In the DTA curve, the more relevant feature corresponded to that present at about 230 °C, in which an abrupt exothermal peak is detected and associated with the decomposition-oxidation of the metal chelates and the evolved gases. This process continues around 300 to 350 °C. The TG curve shows a total weight loss

of about 60 % up to 900 °C. Also, Figure 1 shows the XRD analysis of LSFC powders calcined at 900 °C for 5 h and sintered at 1200 °C for 12 h. Only two representative samples have been included in the XRD diagrams for better visualization. The X-ray diffraction patterns are very similar in that all peaks correspond to the pure perovskite phase [19]. In those corresponding to calcined powder, an extra peak appears at the left of the main peak. It can be attributed to the SrFeLaO₄ phase [19]. This secondary phase disappears when heated at 1200 °C, leaving a pure perovskite.

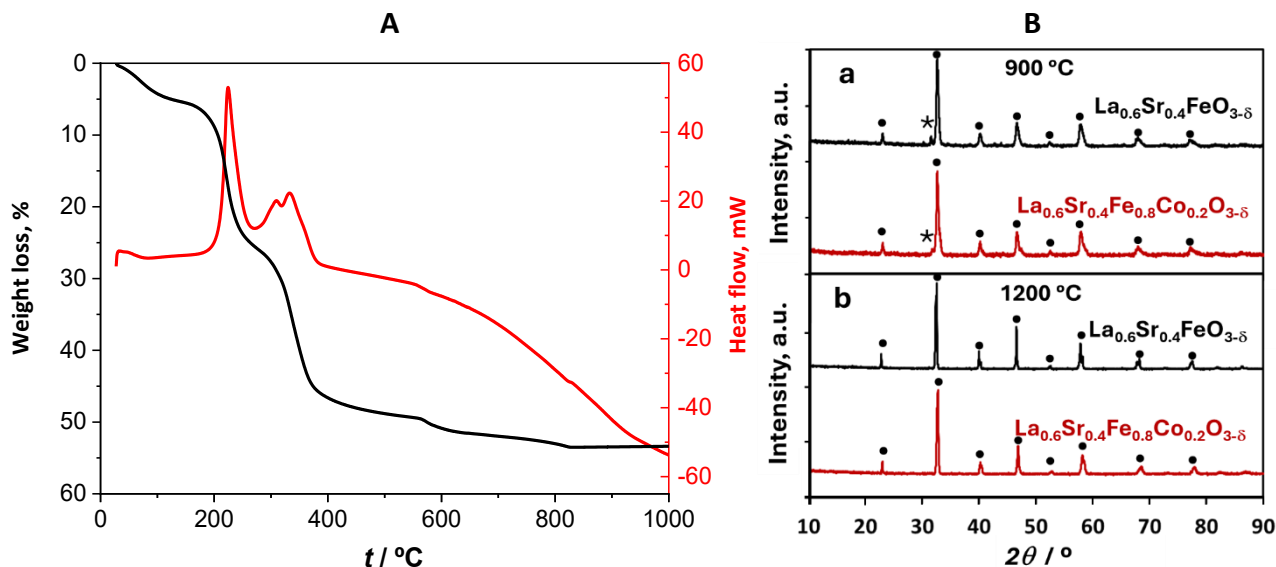


Figure 1. A - TG and DTA curve correspond to the LSFC polymeric gel precursor; B - XRD patterns of a - calcined powders at 900 °C - 5 h and b - sintered powders at 1200 °C.
 • Perovskite phase, * SrFeLaO₄ phase

Linear shrinkage behavior and shrinkage rate curves of typical green compacts with temperature are shown in Figure 2. Green compacts initiate detectable bulk shrinkage at a very low temperature (≈1000 °C).

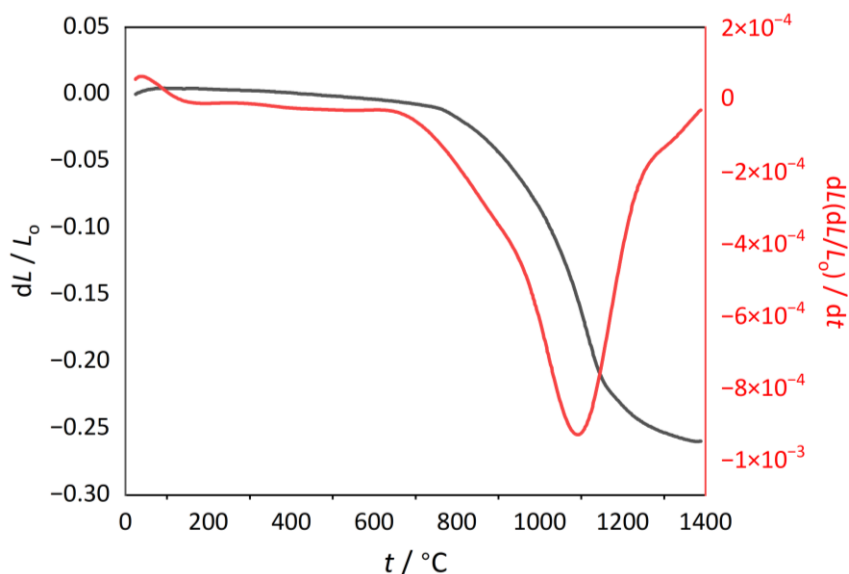


Figure 2. Typical linear shrinkage behavior and shrinkage rate curves of green LSFC compacts vs. sintering temperature.

Although several causes can contribute to this rapid sintering, such behaviour could be explained as the consequence of the nanometer size of powders and low agglomeration state and, consequently, a higher amount of intercrystallite porosity present in the green compacts, which is

rapidly eliminated [18]. The shrinkage process is clearly intensified at ≈ 1100 °C temperature for maximum densification rate. At 1200 °C, most of the shrinkage process has taken place; therefore, it is close to the endpoint density.

Figure 3 reflects the effect of a sintering temperature below and above the temperature for maximum densification rate, (≈ 1100 °C). At 1000 °C, the microstructure showed low densification and high porosity but with sufficient integrity. Above that temperature, 1100 °C, grains are well defined with a uniform round spherical morphology and have a homogeneous sub-micrometre size distribution. The importance of this kind of microstructure is mentioned by Acuña *et al.* in their work [20]. The electrochemical properties of these materials significantly improve with decreasing grain size, and thus, the high specific surface area of these nanostructured cathodes increases the number of active sites for the oxygen reduction reaction. Finally, at 1200 °C, most of the densification process takes place; therefore, it is close to the endpoint density, showing a highly densified microstructure.

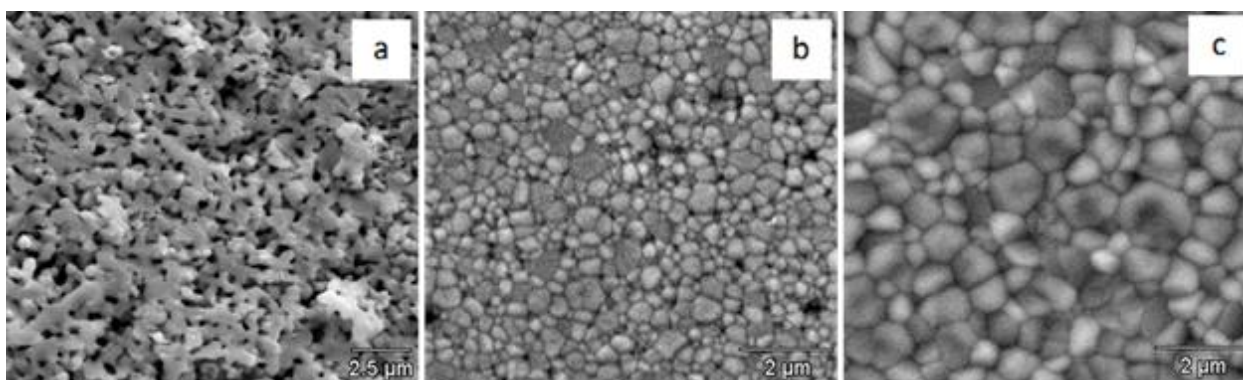


Figure 3. SEM micrographs of typical LSFC compacts sintered at a - 1000, b – 1100 and c -1200 °C for 12 h.

Table 1 shows the thermal expansion coefficients (TEC) and the electronic conductivity (EC) of sintered samples as a function of the Fe and Co content, that is $\text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{1-y}\text{Co}_y\text{O}_{3-\delta}$ ($y = 0$ to 1). As it is observed, the increase of the TEC and EC is influenced clearly by the increase of Co concentration. As Shah *et al.* [10] explain, cobalt undergoes a spin state change at high operating temperatures, resulting in a significant difference in TEC values between cobaltite perovskite cathodes and other components, such as the electrolyte material. The TEC values obtained are quite high ($>17 \times 10^{-6}$ / °C) for samples with higher cobalt proportion, which is incompatible with the values corresponding to the electrolytes mentioned in the table and calculated by the same method as the LSFC cathodes. This produces an important thermomechanical stress. On the contrary, samples with lower Co content reduce the TEC to values that are more comparable. To highlight these considerations, Figure 4 shows cross-sectional SEM views corresponding to electrode/electrolyte interface co-fired at 1200 °C for 5 h. In both cases, the LSFC layer is strongly attached to the LSGM phase and shows a well-defined interface. It can be observed, in the case of higher Co content ($\text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.5}\text{Co}_{0.5}\text{O}_{3-\delta}$), that an extensive microcracking is presented near the interphase. Clearly, this is a consequence of a TEC mismatch, above recommended tolerance values. On the contrary, by reducing the cobalt, ($\text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$), no delamination or other types of structural defects were formed within the laminate structure. The electronic conductivity values in Table 1 are similar to those previously reported in the literature [7]. Also, Banerjee *et al.* [21] report similar results in cathodes synthesized by autocombustion technique for producing powders with particulate sizes ranging 50 to 100 nm, like our procedure. An increase in conductivity is clearly observed with increasing cobalt content. Thus, very significant values of 2000 S/cm are reached for the pure cobaltite. However, a drawback of the

Co-based cathodes is that TEC mismatches with those that correspond to the electrolytes. Therefore, even considering the reduction of the EC, shown in this table, the substitution of Co by Fe is strongly recommended in order to reduce the TEC values and optimize the thermomechanical behavior of these materials. The substitution of Co by Fe changes the orbital configuration of Co 3d orbital and O 2p orbital, decreasing the electrical conductivity of the LSCF [11]. The objective is that TECs of both electrolyte and electrode layers should be well matched to ensure long-term operational stability of the SOFC and also maintain acceptable values of EC. Considering both parameters, of great importance for its satisfactory application in SOFCs, the recommended composition could be nearby to La_{0.6}Sr_{0.4}Fe_{0.8}Co_{0.2}O_{3-δ}, with good thermomechanical stability and exhibiting and adequate electronic conductivity, 244 S/cm at 800 °C.

Table 1. Thermal expansion coefficients (25 to 800 °C) and electronic conductivity (800 °C) as a function of the Fe and Co content in La_{0.6}Sr_{0.4}Fe_{1-y}Co_yO_{3-δ} (LSF y = 0 to LSC y = 1). TEC of the most typical electrolytes for IT-SOFC are also included

Cathode	TEC, 10 ⁻⁶ °C ⁻¹	σ / S cm ⁻¹
La _{0.6} Sr _{0.4} FeO _{3-δ}	12.27	148
La _{0.6} Sr _{0.4} Fe _{0.9} Co _{0.1} O _{3-δ}	14.28	221
La _{0.6} Sr _{0.4} Fe _{0.8} Co _{0.2} O _{3-δ}	14.52	244
La _{0.6} Sr _{0.4} Fe _{0.7} Co _{0.3} O _{3-δ}	14.84	303
La _{0.6} Sr _{0.4} Fe _{0.6} Co _{0.4} O _{3-δ}	15.24	597
La _{0.6} Sr _{0.4} Fe _{0.5} Co _{0.5} O _{3-δ}	15.47	642
La _{0.6} Sr _{0.4} Fe _{0.4} Co _{0.6} O _{3-δ}	15.70	963
La _{0.6} Sr _{0.4} Fe _{0.3} Co _{0.7} O _{3-δ}	16.63	1190
La _{0.6} Sr _{0.4} Fe _{0.2} Co _{0.8} O _{3-δ}	17.11	1380
La _{0.6} Sr _{0.4} Fe _{0.1} Co _{0.9} O _{3-δ}	17.91	1800
La _{0.6} Sr _{0.4} CoO _{3-δ}	18.62	2000
Electrolyte		
La _{0.9} Sr _{0.15} Ga _{0.8} Mg _{0.2} O _{3-δ} (LSGM)	12.83	
Ce _{0.9} Gd _{0.1} O _{2-δ} (CGO)	13.20	

Furthermore, the long-term stability of these materials also depends on the chemical compatibility of these cathodes with the electrolytes. It must be considered that LSC-based materials that react with ZrO₂-based electrolytes have presented serious challenges to their practical application [22]. In fact, reactivity between LSCF and yttria-stabilized zirconia (YSZ) electrolyte with the formation of insulating phases such as SrZrO₃ has been reported [9]. According to the EDX analysis, also included in Figure 4, there is no presence of any new secondary phases, that is, interfacial reactions did not take place between the cathode and the electrolyte. Fe or Co were not detected at the electrolyte surface, even close to the cathode side. Also, it is possible to observe that in the mark located in the cathode (near the interface), there are no indications of the presence of gallium and magnesium. Then, these LSCF cathodes also exhibit chemical stability with the LSGM electrolyte mentioned above. These results are in agreement with Zhang *et al.* [13], LSGM or CeO₂-based electrolytes had good chemical compatibility with the LSCF cathode materials.

LSCF emerges as the most promising cathode for IT-SOFC due to its low polarization resistance. This performance can be attributed to the increasing number of active sites for the oxygen reduction reaction. The Arrhenius representation of area-specific resistance (ASR) dependence on temperature is shown in Figure 5. For that, two electrodes without and with Co (La_{0.6}Sr_{0.4}Fe_{0.8}Co_{0.2}O_{3-δ}) were deposited, in each case, on electrolytes of Ce_{0.9}Gd_{0.1}O_{2-δ}.

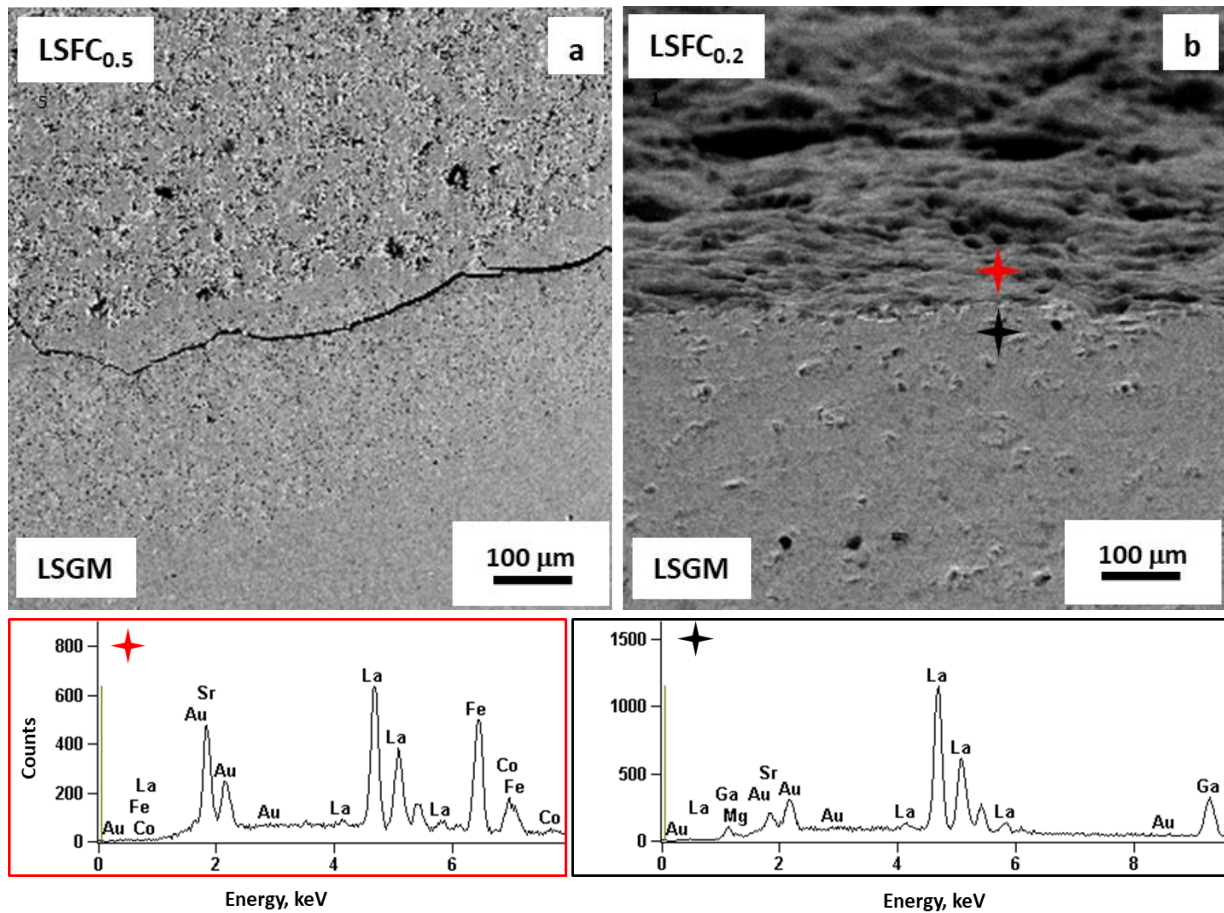


Figure 4. SEM micrographs of cathode/electrolyte pairs co-fired at 1200 °C for 5 h and treated during 10 cycles of raising and lowering of temperature (from room temperature to 800 °C). a - LSCF_{0.5} (La_{0.6}Sr_{0.4}Fe_{0.5}Co_{0.5}O_{3-δ}), b - LSCF_{0.2} (La_{0.6}Sr_{0.4}Fe_{0.8}Co_{0.2}O_{3-δ}). (LSGM: La_{0.9}Sr_{0.15}Ga_{0.8}Mg_{0.2}O_{3-δ}). Include EDX analysis on both sides of the interface corresponded to micrograph b.

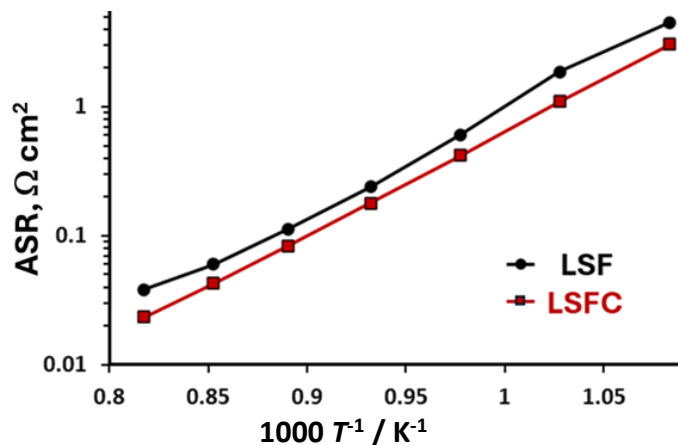


Figure 5. Area-specific resistance dependence with temperature. LSF (La_{0.6}Sr_{0.4}FeO_{3-δ}) and LSFC (La_{0.6}Sr_{0.4}Fe_{0.8}Co_{0.2}O_{3-δ}).

This study is carried out to verify the good electrochemical performance of these materials, specifically for that composition, which had been recommended according to thermomechanical stability and electronic conductivity, (La_{0.6}Sr_{0.4}Fe_{0.8}Co_{0.2}O_{3-δ}). The values of ASR at 700, 750 and 800 °C are 1.87, 0.61 and 0.24 Ω cm² for LSF, and 1.1, 0.42 and 0.18 Ω cm² for LSFC. First, the electrochemical performance is improved with cobalt, which has the same oxidation states as Fe³⁺ and Fe⁴⁺, but the Co-O bonds are weaker than Fe-O bonds, so oxygen ions can move more easily

through the structure. These materials exhibited adequate area-specific resistance around 0.20 Ω cm² for an operating temperature of 800 °C, showing the capability of these structures to move oxygen ions through them. Other authors as Banerjee *et al.* [21], report similar results of 0.142 Ω cm² at 800 °C for La_{0.54}Sr_{0.4}Co_{0.2}Fe_{0.80}O_{3-δ}. Also, it must be considered that the deposition process to improve the microstructure of the cathodes is important to increase their performance (decrease the ASR). La_{0.6}Sr_{0.4}Fe_{0.8}Co_{0.2}O_{3-δ}, showing excellent area-specific resistance values for an operating temperature of 800 °C, constitute a first step in understanding these promising compositions and optimizing them. Finally, the EDX analysis of these samples indicates that no cationic diffusion processes have occurred on either side of the interface. These LSFC materials also exhibit chemical stability with electrolytes based on CGO, in agreement with Zhang *et al.* [13].

Conclusions

La_{0.6}Sr_{0.4}Fe_{1-y}Co_yO_{3-δ} perovskite nanopowders were satisfactorily prepared by the polymeric organic complex solution method. The densification process of powder compacts is initiated at a very low temperature (≈1000 °C) and is completed at ≈1200 °C. We have been able to determine on these samples, the sintering temperature conditions for an effective microstructural control, that is, materials with highly densified microstructure, an effective grain size control, and then optimum microstructures for improving the electrochemical performance of these materials. It has been established how electronic conductivity and thermal expansion coefficients change with the Fe, Co composition. TEC values obtained for LSFC samples with lower Co content are compatible with the values corresponding to the electrolytes used based on LSGM and exhibit adequate electronic conductivity. These materials also exhibit chemical stability with the LSGM and CGO electrolytes. In addition, La_{0.6}Sr_{0.4}Fe_{0.8}Co_{0.2}O_{3-δ} shows excellent area-specific resistance values for an operating temperature of even 700 °C. As a consequence of these results, it can be concluded that those compositions with a small amount of cobalt, La_{0.6}Sr_{0.4}Fe_{0.8}Co_{0.2}O_{3-δ}, are highly recommended for use as cathodes for advanced IT-SOFCs.

The presented results are important not only for the synthetic route itself but also because they establish practical cathode operational parameters on sintered samples for the selection of the most suitable composition, considering the TECs of the electrolyte utilized and the operating temperature of the cell. These results regarding the development of these cathodes will contribute to the fact that solid oxide fuel cells (SOFCs) are expected to play a major role in many power generation applications.

Acknowledgements: The authors acknowledge the financial support by PID2021-123308OB-I00 project, funded by MCIN/AEI/10.13039/501100011033 in Spain and “ERDF A way of making Europe”, by the European Union.

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