

# Study of LaBa<sub>0.52</sub>Sr<sub>0.48</sub>Co<sub>2</sub>O<sub>5+δ</sub> as A Potential Cathode Material for Intermediate Temperature Solid Oxide Fuel Cells

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# Study of $\text{LaBa}_{0.52}\text{Sr}_{0.48}\text{Co}_2\text{O}_{5+\delta}$ as A Potential Cathode Material for Intermediate Temperature Solid Oxide Fuel Cells

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## Abstract

$\text{LaBa}_{0.52}\text{Sr}_{0.48}\text{Co}_2\text{O}_{5+\delta}$  (LBSC) was studied as cathode material for intermediate-temperature solid oxide fuel cells. The crystal structure, thermal expansion behavior and electrochemical properties of LBSC cathode were investigated. A tetragonal layered perovskite structure was observed in LBSC using a GSAS program for refinement. The average thermal expansion coefficient (TEC) was  $24.9 \times 10^{-6}\text{K}^{-1}$  in the temperature range of  $100^\circ\text{C}$ - $800^\circ\text{C}$ , and an area specific resistances (ASR) were  $3.6 \Omega \text{ cm}^2$ ,  $0.70 \Omega \text{ cm}^2$ , and  $0.23 \Omega \text{ cm}^2$  at the operating temperature of  $600^\circ\text{C}$ ,  $700^\circ\text{C}$  and  $800^\circ\text{C}$ , respectively. In another part of this study is to enhance a SOFC performance by cathode surface modification via infiltration technique. The ARS values of symmetrical cell exhibited significant decrease to  $1.31 \Omega \text{ cm}^2$ ,  $0.30 \Omega \text{ cm}^2$ , and  $0.06 \Omega \text{ cm}^2$  at the temperature of  $600^\circ\text{C}$ ,  $700^\circ\text{C}$  and  $800^\circ\text{C}$ , respectively compared with those without infiltrated specimens. Based on the electrochemical properties, LBSC with mixed-ionic-and-electronic-conductor (MIEC) behavior is a potential cathode for intermediate temperature solid oxide fuel cells (IT-SOFCs) based on a SDC electrolyte.

**Keywords:** cathode; electrochemical properties; solid oxide fuel cells (SOFCs)

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## INTRODUCTION

Solid oxide fuel cells (SOFCs) have the potential to be one of the cleanest, most efficient energy technologies and excellent fuel flexibility for direct conversion of chemical fuels to electricity [1-5]. For commercialization of SOFC technologies is hindered by the high cost and the problems associated with the high operating temperature of SOFCs ( $>800^\circ\text{C}$ ), including degradation of cell components because of corrosion, interdiffusion and structural failure [6]. Therefore, to develop a new generation cathode material with low overpotential at intermediate temperature is urgently necessary [7-9]. Generally, electrochemical resistances of key cell components will increase with reduced operating temperatures, especially of the cathode owing to the relatively high activation energy required for the oxygen reduction reaction (ORR) [10-13]. Hence, many researchers made great efforts to reduce the cathode polarization resistance in hopes of improving cathode performance [14-16]. One of an effective and economical approach for cathode surface modification is a wet chemical infiltration process which could enhance the catalytic activity improvement [5].

Based on literatures [17-24], a remarkable increase in cathode polarization resistance after using the infiltration process. For example, the area-specific resistance (ASR) of the  $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$  (LSCF) cathode was  $0.4 \Omega \text{ cm}^2$ ,  $0.15 \Omega \text{ cm}^2$ , and  $0.064 \Omega \text{ cm}^2$  at  $700^\circ\text{C}$ ,  $750^\circ\text{C}$ , and  $800^\circ\text{C}$ , respectively; whereas, after infiltration of  $0.25 \text{ mol L}^{-1} \text{ Ce}_{0.8}\text{Sm}_{0.2}\text{O}_{1.9}$  (SDC) into the

porous LSCF reduced the polarization resistance to  $0.17 \Omega \text{ cm}^2$ ,  $0.074 \Omega \text{ cm}^2$ , and  $0.041 \Omega \text{ cm}^2$  at 700, 750, and 800°C, accordingly. After SDC infiltration, there was a significant improvement for  $\text{O}_2$  reduction on LSCF electrodes, indicating the enhancement of the electrochemical activity [25]. In this study, the crystal structure, thermal expansion behavior and polarization resistance ( $R_p$ ) for symmetrical cell are investigated, and also successfully introduced SDC nanoparticles into the porous LBSC cathode via an infiltration method to improve cathode electrochemical properties.

## EXPERIMENTAL

### Cathode and electrolyte preparation

$\text{LaBa}_{0.52}\text{Sr}_{0.48}\text{Co}_2\text{O}_{5+\delta}$  (LBSC) oxide was synthesized using the solid state reaction process. Stoichiometric amounts of  $\text{La}_2\text{O}_3$ ,  $\text{BaCO}_3$ ,  $\text{SrCO}_3$  and  $\text{CoO}$  powders were used as starting materials. These powders were mixed under ethanol and milled using zirconia balls for 12 h. The ball-milled mixture was dried and ground into a powder with mortar and pestle, and then calcined in air at 1100°C for 4 h.

The  $\text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_{1.9}$  (SDC) powder was synthesized by co-precipitation using  $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  and  $\text{Sm}(\text{NO}_3)_3$  as the starting materials. These starting materials were dissolved in distilled water in stoichiometric ratio amounts and then added to an ammonia solution. The detailed preparation procedure of SDC was described in reference 26.

### Cathode characterization

The structure of the calcined LBSC cathode powder was characterized by X-ray powder diffractometer (XRD; Rigaku D/MAX-2500V) using a  $\text{Cu K}\alpha$  ( $1.5418 \text{ \AA}$ ) radiation source with a scanning rate of  $4^\circ/\text{min}$  and in the range of  $20\text{-}80^\circ$ . The LBSC powder pattern and lattice parameter was analyzed by Rietveld refinement using the GSAS program. The LBSC powder was pressed with cylindrical form and sintered at  $1200^\circ\text{C}$  in air for 2 h, and the sintered pellet was used to determine the thermal expansion coefficient. The thermal expansion coefficient was measured by a thermomechanical analyzer (TMA; TMA 7300, Hitachi) from 100 to  $800^\circ\text{C}$  with heating rate of  $10^\circ\text{C}/\text{min}$ .

### Fabrication of symmetrical cell

Symmetrical cell can be described in detail as follows. The cathode paste consists of cathode powder, solvent, binder and plasticizer. The cathode paste was applied by screen-printing on both sides of SDC electrolyte discs. On one side, the cathode paste was painted as the working electrode (WE) with surface area of  $0.385 \text{ cm}^2$ . The reference electrode (RE) was placed away from the WE by about 0.3-0.4 cm. Such a distance was chosen to avoid measurement errors due to the misalignment of the working and counter electrodes [27-29]. The cathode counter electrode (CE) was placed on the other side of the SDC disk. After the cathode material was painted on the electrolyte, it was heated at  $800^\circ\text{C}$  for 2 h in air.

The infiltration technique based on the procedure was described in reference 30. The appropriate amounts of  $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  and  $\text{Sm}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  were dissolved in water with 0.5 M aqueous nitrate solution for SDC precursors. Ethyl glycol was added to the nitrate solution as a complexing agent. Ethanol was added into the aqueous solution at a ratio of 1:1 to improve the wetting ability on the LBSC backbone;  $0.03 \mu\text{L}$  of this solution was infiltrated into each side of the porous LBSC cathode using a micro-liter syringe to control the amount of loading. After the aqueous solution dried in air, the infiltrated symmetrical cell was fired at  $700^\circ\text{C}$  for 2 h to obtain the desired SDC nano-sized particles within the LBSC skeleton.

### Symmetrical cell measurement

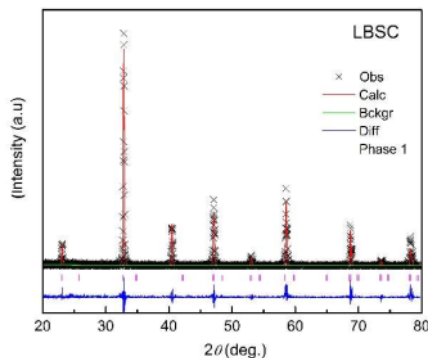
The symmetrical cell measurement was carried out under air ( $p(\text{O}_2) = 0.21 \text{ atm}$ ) in temperatures ranging from 600 to  $800^\circ\text{C}$  at intervals of  $50^\circ\text{C}$ . The AC impedance measurement was performed using the VoltaLab PGZ301 potentiostat with frequency applied range from 100 kHz to 0.1 Hz

with 10 mV AC signal amplitude. The electrochemistry impedance spectrum (EIS) analysis was performed with the Zview software.

## RESULT AND DISCUSSION

### LBSC cathode characterization

Figure 1 shows the refinement of LBSC patterns including the measured XRD data, the calculated profile and the difference between them. There are no peaks due to impurities in the structure of LBSC, suggesting that the cathode is successful synthesis. Lattice parameters regarding LBSC obtained from the Rietveld refinement is listed in Table 1. The experimental data highly agree with the calculated profiles, suggesting that cations are well ordered in the perovskite lattice [31, 32].



**Fig.1:** Observed (crosses) and calculated (solid line) XRD profiles and the difference (bottom line) for LBSC calcined at 1100°C.

**Table 1:** Crystallographic information for LBSC at room temperature, cell parameters obtained from the Rietveld refinement\*

Atom	Wyckoff position	x	y	z	Uiso	Occup.
LA1	1b (0 0 ½)	0	0	0.5	0.0162	0.9162
CO2	2h (½ ½ z)	0.5	0.5	0.2532	0.0128	0.9644
BA3	1a (0 0 0)	0	0	0	0.0101	0.5167
O4	1c (½ ½ 0)	0.5	0.5	0	0.0573	0.9325
O5	4i (½ 0 z)	0.5	0	0.2487	0.0572	1.4271
O6	id (½ ½ ½)	0.5	0.5	0.5	0.0417	0.7247
SR7	1a (0 0 0)	0	0	0	0.0003	0.4750

\*  $\chi^2 = 1.44$ ,  $R_p = 0.23$ ,  $R_{wp} = 0.32$ ,  $R_{exp} = 0.27$ , tetragonal (Space group: P4/mmm),  $a = 3.868 \text{ \AA}$ ,  $b = 3.868 \text{ \AA}$ ,  $c = 7.731 \text{ \AA}$ ,  $v = 115.69 \text{ \AA}^3$

The thermal expansion coefficients (TECs) compatibility between cathode and other components is important. Since a large difference in TECs between these components will introduce interfacial stress during thermal cycling and result in cracks and cell degradation [33]. The thermal expansion behavior in the temperature range of 100-800°C is shown in Fig. 2. The average TEC of LBSC is  $24.9 \times 10^{-6} \text{ K}^{-1}$ , and TECs at various temperature range is listed in Table 2. The LBSC shows a linear expansion in the low temperature region (100-300°C) and a slight increase in slope at higher temperatures region (300-800°C). At higher temperature, a part of the smaller  $\text{Co}^{4+}$  were reduced to larger  $\text{Co}^{3+}$  or  $\text{Co}^{2+}$  with a loss of oxygen,  $\text{Co}^{3+}$  ions are easy to transit from low-spin to high-spin state [34-36].

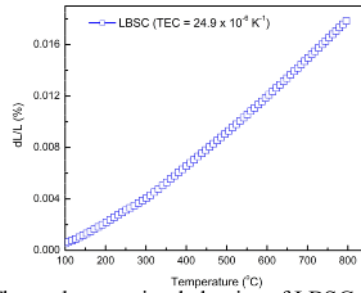


Fig.2: Thermal expansion behavior of LBSC from 100°C to 800°C.

Table 2: TECs for LBSC at various temperature range

Sample	TEC x 10 <sup>-6</sup> (K <sup>-1</sup> )		
	100-800°C	100-300°C	300-800°C
LBSC	24.9	17.6	27.8

### Interfacial polarization resistances

Figs. 3(a)-(e) are typical impedance spectra for LBSC and LBSC infiltrated with 0.5M SDC measured in symmetrical cells under open circuit conditions. The electrocatalytic activity of cathode was characterized by the interfacial polarization resistance ( $R_p$ ), and it was determined from the size of the impedance loop. [37]. The  $R_p$  of LBSC is 3.60  $\Omega \text{ cm}^2$ , 0.70  $\Omega \text{ cm}^2$ , and 0.23  $\Omega \text{ cm}^2$  at 600°C, 700°C and 800°C, respectively. After infiltration of 0.5 M SDC into the porous LBSC, the polarization resistance reduced to 1.31  $\Omega \text{ cm}^2$ , 0.41  $\Omega \text{ cm}^2$  and 0.06  $\Omega \text{ cm}^2$  at 600°C, 700°C, and 800°C, respectively. There was a significant reduction in the impedance for  $\text{O}_2$  reduction on LBSC, indicating the enhancement of the electrochemical activity as listed in Table 3.  $R_p$  of the symmetrical cells are shown in Fig. 3(f). The significant decrease in  $R_p$  was mainly attributed to the creation of SDC|LBSC phase boundaries for LBSC infiltrated with 0.5M SDC. The newly formed SDC deposited on the LBSC porous skeleton would allow gas-phase molecules to easily diffuse to the SDC|LBSC boundaries, which considerably increased the electrochemical sites for oxygen reduction reaction (ORR) [38].

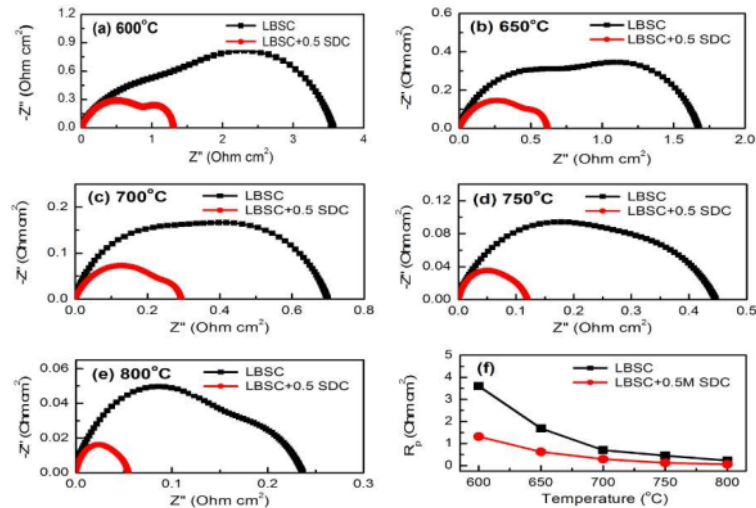


Fig. 3(a)-(e): Typical impedance of LBSC and LBSC infiltrated with 0.5M SDC and (f) polarization resistance over the temperature range of 600-800°C



**Table 3:** Interfacial polarization resistance of cathodes as a function of temperature

Specimens	$R_p(\Omega \text{ cm}^2)$				
	600°C	650°C	700°C	750°C	800°C
LBSC	3.60	1.68	0.70	0.45	0.23
LBSC+0.5M SDC	1.31	0.62	0.30	0.12	0.06

## CONCLUSIONS

This study mainly investigated the cathode performance improvement via infiltration technique. The average TEC of LBSC is  $24.9 \times 10^{-6} \text{ K}^{-1}$ , in which a part of the smaller  $\text{Co}^{4+}$  were reduced to larger  $\text{Co}^{3+}$  or  $\text{Co}^{2+}$  with a loss of oxygen,  $\text{Co}^{3+}$  ions are easy to transit from low-spin to high-spin state at higher temperature. After infiltration of 0.5M SDC on LBSC backbone, polarization resistance ( $R_p$ ) exhibited a significant decrease from  $3.60 \Omega \text{ cm}^2$ ,  $0.70 \Omega \text{ cm}^2$ , and  $0.23 \Omega \text{ cm}^2$  to  $1.31 \Omega \text{ cm}^2$ ,  $0.30 \Omega \text{ cm}^2$ , and  $0.06 \Omega \text{ cm}^2$  at  $600^\circ\text{C}$ ,  $700^\circ\text{C}$ , and  $800^\circ\text{C}$ , respectively. This behavior suggested that infiltration of the electrolyte nanoparticles into the porous cathode backbones really improved the electrochemical performance.

## ACKNOWLEDGEMENTS

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