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# Nano-sized $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$ as the cathode for solid oxide fuel cells with proton-conducting electrolytes of $BaCe_{0.8}Sm_{0.2}O_{2.9}$

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#### ABSTRACT

Nano-sized  $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$  (SSC) was fabricated onto the inner face of porous  $BaCe_{0.8}Sm_{0.2}O_{2.9}$  (BCS) backbone by ion impregnation technique to form a composite cathode for solid oxide fuel cells (SOFCs) with BCS, a proton conductor, as electrolyte. The electro-performance of the composite cathodes was investigated as function of fabricating conditions, and the lowest polarization resistance, about 0.21  $\Omega$  cm<sup>2</sup> at 600 °C, was achieved with BCS backbone sintered at 1100 °C, SSC layer fired at 800 °C, and SSC loading of 55 wt.%. Impedance spectra of the composite cathodes consisted of two depressed arcs with peak frequency of 1 kHz and 30 Hz, respectively, which might correspond to the migration of proton and the dissociative adsorption and diffusion of oxygen, respectively. There was an additional arc peaking at 1 Hz in the Nyquist plots of a single cell, which should correspond to the anode reactions. With electrolyte about 70  $\mu$ m in thickness, the simulated anode, cathode and bulk resistances of cells were 0.021, 0.055 and 0.68  $\Omega$  cm<sup>2</sup> at 700 °C.

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#### 1. Introduction

High temperature proton conductors (HTPCs) are now of special interest thanks to their potential applications as electrolytes for solid oxide fuel cells (SOFCs). With HTPC electrolyte, water is formed at the cathode. This makes proton-conducting fuel cells (H-SOFCs) gain some advantages over those cells with oxygen-ion conducting electrolytes (O-SOFC), such as greater fuel utilization efficiency and simpler fuel-recycling instruments [1–3]. Yet, the formation of water at the cathode induces large cathode polarization resistances. Ni et al. [4] have simulated the electrochemical performance of H-SOFC, and found that water generation at the cathode results in both pressure gradient and gas concentration gradient, and thus causes serious cathode concentration over-potentials, which limits the cell performance.

Efforts have been devoted to lowering the cathode polarization resistance for H-SOFCs by introducing novel cathode materials [5–9]. With Ba(Ce<sub>0.8</sub>Gd<sub>0.2</sub>)O<sub>2.9</sub> as electrolyte, Iwahara et al. [5] have used (La<sub>0.6</sub>Ba<sub>0.4</sub>)MnO<sub>3</sub> and platinum as cathodes, and cathode polarization resistances were about 1.42 and 0.35  $\Omega$  cm<sup>2</sup> at 800 °C, respectively. And with the same electrolyte, the cathode polarization resistance reduced to 0.47  $\Omega$  cm<sup>2</sup> at 800 °C, when BaPr<sub>0.8</sub>Gd<sub>0.2</sub>O<sub>2.9</sub> was used as the cathode [6]. The polarization resistance was significantly reduced by using Ba<sub>0.5</sub>Sr<sub>0.5</sub>Co<sub>0.8</sub>Fe<sub>0.2</sub>O<sub>3</sub> as

the cathode [3], about  $0.37 \Omega \text{ cm}^2$  (anode + cathode) at 700 °C for a single cell with Ni–BaCe<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>2.9</sub> as anode and BaCe<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>2.9</sub> as electrolyte, respectively. Our previous work shows that the polarization resistance could be further reduced to  $0.2 \Omega \text{ cm}^2$  at 700 °C by using Sm<sub>0.5</sub>Sr<sub>0.5</sub>CoO<sub>3- $\delta}$  as the cathode, which was about 25% of the total cell resistance with 50-µm-BaCe<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>2.9</sub> electrolyte [8]. With the lowering of temperature, the polarization resistance (anode + cathode) increased to 0.66  $\Omega \text{ cm}^2$  at 600 °C, which is about 40% of the total cell resistance. With the thickness of BaCe<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>2.9</sub> electrolyte reduced to 10 µm, the polarization resistance became the major source of the cell resistance, about 71% of the total cell at 650 °C [9], suggesting that the key work is to reduce the polarization resistance of the cathodes to substantially improve the cell performance.</sub>

Besides exploring novel cathode materials, ion impregnation technique has been demonstrated as an effective way to improve the cell performance, by which 25–300 nm granular particulates randomly locate on the inner surfaces of the frame, and thus greatly enlarge the triple phase boundaries (TPBs) and promote the electroreactions [10–14]. In this work, nano-sized Sm<sub>0.5</sub>Sr<sub>0.5</sub>CoO<sub>3– $\delta$ </sub> (SSC) was fabricated on BaCe<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>2.9</sub> (BCS) backbones by an ion impregnation technique as H-SOFC cathodes to increase the TPBs and thus improve the electrode performance. The influence of preparation conditions on the performance of composite cathode was studied. And the rate-limiting step was investigated by impedance spectra analysis. To intensively explore the cathode performance, screen-printing was also adopted to prepare the cathode.

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#### 2. Experimental

#### 2.1. Fabrication of symmetric and single cells

BaCe<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>2.9</sub> (BCS) powders were prepared by a glycinenitrate process (GNP), and the synthesis details have been reported previously [3]. The powders were cold-pressed into cylindrical disks with size of 13 mm in diameter and 0.3 mm in thickness. The disks were then sintered at 1500 °C for 5 h in air to form dense electrolyte supports for symmetric cells. Porous BCS layers, about 100  $\mu$ m thickness, were screen-printed on each side of the supports and fired at 1200–1400 °C for 4 h in air to form symmetric electrode backbones. The weight of the backbone was measured by an electronic balance (AL104, METTLER TOLEDO).

Sm<sub>0.5</sub>Sr<sub>0.5</sub>CoO<sub>3</sub> (SSC) was deposited onto the inner surface of the porous backbones by an ion impregnation technique to form symmetric cells with SSC–BCS electrodes consisting of nanosized SSC particles and porous BCS backbones. Sm(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O, Sr(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, and Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O were mixed in distilled water at the stoichiometric ratio, and then glycine was added at the glycine/metal molar ratio of 2:1 to prepare an SSC solution. Ion impregnation was carried out by dropping the solution onto the surface of porous BCS backbones. The solution infiltrated into the pores of the backbones under capillary action [12]. After drying, the samples were fired at 700–900 °C in air for 2 h to form the SSC–BCS electrodes. The impregnation-firing cycle was repeated several times for different SSC loading, which was determined by weighing. The loading was expressed as SSC weight percent in the SSC–BCS electrode:  $W_{SSC} \times 100\%/(W_{SSC} + W_{BCS})$ .

Symmetric cells with composite SSC–BCS electrodes were also fabricated by the screen-printing method to evaluate the electrode performance prepared by different techniques. For the printing process, BCS and SSC (55 wt.% SSC) were ball-milled with ethyl cellulose-terpineol to form a slurry, which was then screen-printed onto green BCS disks. The sandwich structured samples were dried and co-fired at 1100 °C for 5 h to form the symmetric cells. The thickness of the composite cathode was about 100  $\mu$ m.

Single cells were fabricated by preparing the SSC–BCS cathodes onto anode (NiO–BCS) supported BCS electrolytes. NiO and BCS powders were mixed at the weight ratio of 65:35 for the composite anodes. Di-layers of NiO–BCS substrates and BCS electrolytes were fabricated by a co-pressing method. The electrolytes were densified by co-firing the di-layers at 1400 °C for 5 h. Cathodes were fabri-





cated by both ion impregnated and screen-printing methods. The thickness of the anode, electrolyte, and cathode were about 500, 70 and 100  $\mu$ m, respectively.

#### 2.2. Characterization

Ag paste was applied onto the cathode surface as current collector. AC impedance method was used to investigate the polarization resistances of symmetric cells with an electrochemical workstation (IM6e, Zahner). The measurements were conducted in the typical frequency range from 0.01 Hz to 1 MHz at 500–700 °C in air. The microstructure of the impregnated electrode was observed by scanning electron micrograph (JSM-6700F, JEOL). Single cells were tested from 600 to 700 °C in a home-developed-testing system with humidified hydrogen ( $\sim$ 3% H<sub>2</sub>O) as the fuel and ambient air as the oxidant. And the electrochemical performance of single cells was investigated using the electrochemical workstation.

#### 3. Results and discussion

## 3.1. Polarization resistances of electrodes prepared by impregnation

Fig. 1 shows the dependence of polarization resistance on SSC firing temperature. The BCS backbone was sintered at 1100 °C and the SSC loading was 38 wt.%. The resistance shows the lowest value when SSC was fired at 800 °C. For example, the resistances at 700 °C are about 0.32, 0.24, and 0.52  $\Omega$  cm<sup>2</sup> when SSC firing temperatures are 700, 800, and 900 °C, respectively. The three curves are almost parallel, implying that the activation energies for cathode reactions are not affected by the firing temperature. The activation energies calculated from the curves are 1.05 eV, higher than that of SSC-Ce<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>3- $\delta$ </sub> (SDC) composite cathode (30 wt.% SDC) for O-SOFC [15].

The SSC-loading is greatly important to the electro-performance of the electrode fabricated by impregnation [12,13]. Fig. 2 shows the dependence of SSC-loading on the backbone sintering temperature and the times of impregnation-firing cycles, as shown in Fig. 2. The SSC loading increases almost linearly with the cycle times. But the rate of increase reduces with the sintering temperature due to the loss of backbone porosity. When BCS was sintered at 1400 °C, the loading for the first cycle was 0.012 mg cm<sup>-2</sup> (6 wt.%). After 10 cycles it was only 0.083 mg cm<sup>-2</sup> (29 wt.%). When BCS was sintered at 1100 °C, the loading for each cycle was about 0.026 mg cm<sup>-2</sup>, three times larger than that sintered at 1400 °C, and the final SSC loading reached 0.312 mg cm<sup>-2</sup>, about 60 wt.%.



**Fig. 2.** Dependence of SSC loading on BCS backbone sintering temperature and the cycle times.



**Fig. 3.** Effect of SSC loading on the polarization resistance (R), high frequency resistance ( $R_{\rm H}$ ), and low frequency resistance ( $R_{\rm L}$ ) measured at 600 °C. The ratio of high frequency resistance to polarization resistance,  $r_{\rm H} = R_{\rm H}/R$ , is also shown.

The polarization resistances (*R*) of the symmetric cells measured at 600 °C are shown in Fig. 3 as functions of SSC loading. The resistance reduces with the increase of SSC loading and reaches a minimum at 55 wt.% SSC. At 600 °C, *R* are about 1.98 and 0.21  $\Omega$  cm<sup>2</sup> when the loading are 25 and 55 wt.%, respectively. The electrode morphologies with different SSC loading are shown in Fig. 4. Fig. 4(a) shows the microstructure of BCS backbone which is composed of particles with the size of 0.5–1  $\mu$ m. When 25 wt.% SSC was loaded, SSC particles, about 100 nm in size, are observed to cover the inner surface of the backbone and appear to be discrete to each other. This distribution character results in low conductivity and thus high polarization resistance. At the loading of 55 wt.%,



**Fig. 5.** Impedance spectra measured in air at 600 °C for symmetric cells with SSC loading of 43 wt.%.

the particles grow to about 250 nm and form a well-connected network over the BCS backbone, as shown in Fig. 4(c). The continuous distribution will lead to a high conductivity, and thus reduce the polarization resistance.

Fig. 5 is a typical impedance spectrum investigated at 600 °C for symmetric cells with BCS backbone sintered at 1100 °C, SSC fired at 800 °C, and SSC loading of 43 wt.%. The spectrum consists roughly of two depressed arcs, a high frequency arc and a low frequency arc, suggesting that there are at least two limiting steps for the cathode reactions. The general cathode reactions for H-SOFCs can be expressed as Eq. (1), which might consist of several steps including: (1) surface dissociative adsorption and diffusion of oxygen along with charge transfer; (2) proton migration to triple phase boundaries (TPBs); and (3) the formation and desorption of H<sub>2</sub>O. To intensively investigate the limiting steps of cathode reactions, the dependence of each arc resistance on SSC loading and temperature was investigated.

$$40H_0^{\bullet} + O_2 + 4e' \rightarrow 2H_2O + 4O_0^{\times}$$
(1)



Fig. 4. SEM pictures of the impregnated electrode with the SSC loading of: (a) 0, (b) 25 wt.%, (c) 55 wt.%, and (d) the screen-printed electrode.



**Fig. 6.** Dependence of activation energy for polarization resistances (AE), high frequency resistances (AE<sub>H</sub>), and low frequency resistances (AE<sub>L</sub>) of the symmetric cells on SSC loading.

The fitted high frequency resistance ( $R_H$ ) and low frequency resistance ( $R_L$ ) measured at 600 °C are also shown in Fig. 3 as a function of SSC loading. It can be seen that the dependence of both  $R_H$  and  $R_L$  on SSC loading is similar to that of the polarization resistance.  $R_H$  and  $R_L$  drop from 0.40 and 1.64  $\Omega$  cm<sup>2</sup> to the minimum of 0.09 and 0.13  $\Omega$  cm<sup>2</sup>, respectively, with the loading increases from 25 to 55 wt.%. However, a slower reducing rate was observed for  $R_H$ . The ratio of  $R_H$  to R is only 20% at loading of 25 wt.%, and increases to 43% at 55 wt.% SSC. The ratio of  $R_H$  also increased with testing temperature. Measured at 700 °C, the ratio was 50% at 55 wt.%, suggesting that the reaction corresponding to the low frequency arc is more sensitive to the testing temperature and SSC loading than the high frequency arc.

To further explore the reactions that correspond to the impedance spectra, the activation energies (AE) for R,  $R_{H}$ , and  $R_{L}$ were calculated and denoted as AE, AE<sub>H</sub> and AE<sub>L</sub>, respectively. These activation energies have different dependence on SSC loading, as shown in Fig. 6. AE<sub>H</sub> increases linearly with SSC loading first, and then keeps almost stable of 0.75 eV with SSC loading over 46 wt.%. At SSC loading of 25 wt.%, AE<sub>H</sub> is 0.64 eV, very similar to that of proton conduction in doped BaCeO<sub>3</sub>, about 0.50–0.60 eV [16,17], suggesting that the high frequency arc might be related to proton migration or reaction at the TPBs. Considering that the generating energy of OH<sup>-</sup> is usually about an order lower than that of proton migration [18], proton migration is highly nominated to correspond to the high frequency arc. The dependence of AE<sub>H</sub> on SSC loading also suggests that the high frequency arc should correspond to the proton migrating to SSC/BCS interface, rather than the grain boundary conduction of BCS. The increase of  $AE_H$  with SSC loading might be explained as the result of lattice distortion of external BCS due to the great surface adsorbability of nano-sized SSC. And when BCS backbone is fully covered by SSC, AE<sub>H</sub> becomes to stable. While AE<sub>L</sub> reduces with SSC loading first, it reaches a minimum at 55 wt.% SSC, and then increases. With SSC loading of 25, 55 and 60 wt.%, the AE<sub>L</sub> are 1.23, 1.02 and 1.13 eV, respectively, close to the activation energy of low frequency resistance of SSC-LSGM [19], which is deemed corresponding to the surface dissociative adsorption and diffusion of oxygen. Therefore, the cathode fabricated by the impregnation technique, the limiting processes might be proton transferring to the SSC/BCS interface and oxygen dissociative adsorption and diffusion, especially the latter would be dominant while the temperature is lower than 700 °C. And intensive study is greatly needed to specify the detailed rate-limiting steps.



**Fig. 7.** The polarization resistances of symmetric cells measured at 700 °C in air with electrode fabricated by screen-printing ( $\Box$ ) and ion impregnation technique ( $\bigcirc$ ). The SSC ratio is 55 wt.% and the electrode thickness is about 100  $\mu$ m for both SSC-PR and SSC-IM.

#### 3.2. Electrode performance for electrodes by screen-printing

Screen-printing method was also taken to fabricate SSC-BCS composite cathodes to evaluate the effect of preparation technique on electrode performance. The symmetric cells with electrode fabricated by the ion impregnation and printing processes are denoted as SSC-IM and SSC-PR, respectively. Fig. 7 shows the impedance spectra of SSC-IM and SSC-PR measured at 700 °C. The electrode thickness is about 100 µm and the SSC content is 55 wt.% for the two cells. The impedance spectrum of SSC-PR consists also of two arcs, suggesting similar reaction mechanism to that of SSC-IM. And for both cells, the high frequency arc peaks at 1 kHz, while low frequency arc peaks at 30 Hz. However, the resistances of SSC-PR, especially  $R_{\rm I}$ , are much larger than those of SSC-IM, suggesting the electrode performance is sensitive to the fabricating details. For SSC-IM, the fitted high frequency resistance is  $0.029 \,\Omega \,\mathrm{cm^2}$ , quite close to the fitted low frequency resistance of  $0.030 \,\Omega \,\mathrm{cm^2}$ . But for SSC-PR, the low frequency resistance is  $0.14 \,\Omega \,\mathrm{cm^2}$ , twice larger than the high frequency resistance, about 0.064  $\Omega$  cm<sup>2</sup>. And the low frequency resistance of SSC-PR is about five times larger than that of SSC-IM, implying high catalytic activity towards oxygen dissociation of SSC prepared by the ion impregnation process. The total polarization resistance of SSC-PR is about  $0.19 \,\Omega \,\mathrm{cm}^2$ , about three times larger than that of SSC-IM, mainly due to the large low frequency resistance. It should be noted that activation energy of high frequency resistance (AE<sub>H</sub>) are 0.59 eV for SSC-PR, almost the same with that for proton conduction [16,17], confirming that the high frequency arc is proton migration related. The closeness of  $\ensuremath{\mathsf{AE}_{\mathsf{H}}}$  for SSC-PR to that of SSC-IM with SSC loading below 25 wt.% also suggests that the increase of AE<sub>H</sub> of SSC-IM with SSC loading is due to the great surface tension of SSC in covering morphology. The AEL and AE of SSC-PR are 1.13 and 0.93 eV, respectively.

The microstructures of the cathodes fabricated by the printing technique are shown in Fig. 4(d), in which SSC and BCS particles could not be discerned with both sizes about  $0.5 \,\mu$ m, quite different with those prepared by impregnation technique, as shown in Fig. 4(c). The covering morphology of SSC-IM enlarges the region of TPBs to nearly all around the surface of BCS backbone, thus greatly reduced the polarization resistance [20].

#### 3.3. Single cells performance

Fig. 8 shows the Nyquist plots of single cells with SSC–BCS cathodes fabricated by the ion impregnation and screen-printing techniques. Unlike the spectra of symmetric cells as shown in Fig. 7, there are three arcs in each spectrum, a high frequency arc, a low frequency arc, and an additional low frequency arc. For both single cells, the high and low frequency arcs peak at about 1 kHz and 30 Hz, respectively, similar to those of symmetric cells; while the additional arc peaks at 1 Hz, suggesting that the additional arc might be associated with the anode process. For the cell with the impregnated cathode, the fitted resistances are 0.026, 0.029, and



**Fig. 8.** Impedance spectra of single cells with cathode fabricated by screen-printing  $(\Box)$  and ion impregnation technique  $(\bigcirc)$ . The measurement is conducted at 700 °C under open circuit conditions with wet H<sub>2</sub> as the fuel and ambient air as the oxidant. The weight percent of SSC is 55 wt.% and the cathode thickness is about 100  $\mu$ m.



Fig. 9. I-V(solid) and I-P(hollow) curves of single cells with impregnation cathode and printed cathode. The SSC-loading is about 55 wt.% and the cathode thickness is about 100  $\mu$ m.

 $0.021 \ \Omega \ cm^2$  for the high, low, and the additional arcs, respectively. And for the cell with the printed cathode, the fitted resistances are 0.067, 0.196, and 0.021  $\Omega \ cm^2$ , respectively. It should be noted that the fitted  $R_{\rm H}$  and  $R_{\rm L}$  of the two single cells are very close to those of the corresponding symmetric cells. In addition, the fitted additional low frequency resistances are almost identical for the single cells. These demonstrate that the additional arc should correspond to the anodic reaction, while the high and low frequency arcs are induced by the cathode processes. Thus the anode, cathode, and bulk resistances could be separated. For the single cell with impregnated cathode, they are 0.021, 0.055 and 0.676  $\Omega \ cm^2$  at 700 °C, respectively, implying that anode reactions are relatively fast compared with cathode reactions, and that the cell resistance is dominated by the electrolyte. Therefore, reducing the electrolyte thickness could intensively improve the cell performance.

*I–V* and *I–P* curves of single cells are shown in Fig. 9 with humidified hydrogen as the fuel and stationary air as the oxidant. The electrolyte thickness is about 70  $\mu$ m. The open circuit voltages are about 1.06, 1.03 and 0.99 V, and the peak power densities are 222, 273 and 307 mW cm<sup>-2</sup> at 600, 650 and 700 °C, respectively. The peak power density of the cell with printed cathode is much lower, only 239 mW cm<sup>-2</sup> at 700 °C, suggesting that application of ion impregnation technique can reduce the cathode polarization resistance and thus improve cell performance.

#### 4. Conclusions

Nano-sized-Sm<sub>0.5</sub>Sr<sub>0.5</sub>CoO<sub>3- $\delta$ </sub> (SSC) was coated on porous BaCe<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>2.9</sub> (BCS) backbones by the ion impregnation technique to consist a composite cathode for solid oxide fuel cells with proton-conducting electrolyte. The electro-performance of such composite cathode was investigated as function of fabrication details. Lowest polarization resistance, about 0.21  $\Omega$  cm<sup>2</sup> at 600 °C, was achieved under the optimized conditions, only 1/3 of that prepared by screen-printing.

Impedance measurement indicates that there are two ratelimiting steps in the cathode reactions, which correspond to the arcs peaking at 1 kHz and 30 Hz, respectively. The activation energies for the high and low frequency resistances are 0.6 and 1.02 eV, respectively, implying that the migration of protons and the dissociative adsorption and diffusion of oxygen might be the limiting steps. The low frequency resistance dominates the polarization resistance at temperature lower than 700 °C. An additional arc peaking at 1 Hz appeared in the Nyquist plots of single cells with cathode prepared by both printing and impregnation technique, which might correspond to the anode reactions. The simulated anode, cathode and bulk resistances of cell with impregnation cathode are 0.021, 0.055 and 0.676  $\Omega$  cm<sup>2</sup> at 700 °C, respectively, implying that anodic polarization resistance is relatively small, and that reducing electrolyte thickness could intensively improve the cell performance.

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