

# Supporting effect of silver on BSCF cathodes for SOFC

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#### Abstract

Cathode materials for solid oxide fuel cells should show good catalytic activity in the oxygen reduction reaction (ORR), high electronic and ionic conductivities at relatively low temperature, and the thermal expansion coefficients (TECs) compatible with the electrolyte. Ba<sub>0.5</sub>Sr<sub>0.5</sub>Co<sub>0.8</sub>Fe<sub>0.2</sub>O<sub>3-5</sub> (BSCF) shows one of the greatest catalytic activity in ORR which occurs on the overall surface of the electrode. Oxygen vacancies in BSCF permit to transport a significant amount of oxygen ions. Nevertheless, BSCF revealed its high TEC and low electrical conductivity. Some properties of the electrode can be improved by adding a second phase to the BSCF electrode. Silver shows the greatest electronic conductivity among metals; it is cheap and has excellent catalytic activity in the ORR. Moreover, the presence of Ag in the composite cathode increases the electronic conductivity of the cathode and reduces the area specific resistance (ASR). The goal of this work is to make comparison among properties of the BSCF cathode, the Ag cathode, the cathode with silver interlayer between BSCF and electrolyte (Ag|BSCF), and the Ag|BSCF cathode with the additional silver layer on the top of the cathode (Ag|BSCF|Ag). The cathodes were prepared on the Ce<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>1.9</sub> electrolyte disks and tested using scanning electron microscopy (SEM) and electrochemical impedance spectroscopy. SEM examinations of the obtained electrodes revealed consistent with the goal, well connected, porous 3D structures. The contact was significantly better in case of the composite electrodes. Equivalent electrical circuits were used for fitting impedance spectra. The circuits consisted of a resistor Ro and three (or two) resistors and capacitors parallel connected in pairs (R, C) and combined in series. The fitting errors were smaller than 0.7% in all cases. The m and a coefficients were received from Arrhenius plots of ASR and the fitting, and indicated that the charge transfer was the rate-determining step for the Ag|BSCF|Ag electrode, whereas for the BSCF and Ag|BSCF electrodes it was the oxygen ions transport through the BSCF. Activation energies of ASR for the BSCF, Ag, Ag|BSCF and Ag|BSCF|Ag cathodes were similar, yielding the values of 55.1 kJ mol<sup>-1</sup>, 47.7 kJ mol<sup>-1</sup>, 47.7 kJ mol<sup>-1</sup> and 37.9 kJ mol<sup>-1</sup>, respectively.

keywords: Solid oxide fuel cell, Composite cathode, Barium strontium cobalt ferrite, Electrochemical impedance spectroscopy

#### WSPOMAGAJĄCY EFEKT SREBRA W KATODACH BSCF STOSOWANYCH W SOFC

Materiały katodowe stosowane w ogniwach paliwowych z elektrolitem ze stałego tlenku powinny charakteryzować się dobrą aktywnością katalityczną w reakcji redukcji tlenu (ang. oxygen reduction reaction, ORR), wysokim przewodnictwem elektronowym i jonowym w relatywnie niskich temperaturach, a także współczynnikiem rozszerzalności termicznej kompatybilnym z elektrolitem. Ba<sub>0.5</sub>Sr<sub>0.5</sub>Co<sub>0.8</sub>Fe<sub>0.2</sub>O<sub>3-6</sub> (BSCF) jest jednym z najlepszych katalizatorów ORR. Wysokie przewodnictwo jonowe anionów tlenkowych BSCF zawdzięcza obecności dużej ilości wakancji tlenowych. Niemniej jednak BSCF charakteryzuje się wysokim współczynnikiem rozszerzalności termicznej oraz niskim przewodnictwem elektronowym. Tworzenie katod kompozytowych z domieszkowaniem odpowiednich materiałów może doprowadzić do polepszenia właściwości elektrody wykonanej z BSCF. Doskonałym materiałem do tworzenia kompozytów z BSCF jest srebro. Srebro jest nie tylko tanim materiałem o wysokiej aktywności katalitycznej w ORR, ale przede wszystkim charakteryzuje się najwyższym wśród metali przewodnictwem elektronowym. Zjawisko "płynięcia" srebra pod wpływem wysokich temperatur przyczynia się do polepszenia kontaktu między elektrodą a elektrolitem, co jest niezbędne do zwiększenia przewodnictwa elektronowego katody oraz obniżenia wartości oporu polaryzacyjnego. Celem niniejszej pracy jest porównanie jednoskładnikowych elektrod wykonanych z BSCF oraz srebra z elektroda kompozytową Ag/BSCF, w której na warstwę srebra nałożono warstwę BSCF oraz z elektrodą Ag/BSCF/Ag z dodatkową warstwą srebra na powierzchni. Elektrody wykonano na elektrolicie w postaci dysku wykonanego z Ce<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>1.9</sub>. Otrzymane kompozyty scharakteryzowano metodami skaningowej mikroskopii elektronowej i elektrochemicznej spektroskopii impedancyjnej. Zdjęcia z mikroskopu elektronowego potwierdziły otrzymanie porowatych, trójwymiarowych struktur. W celu dopasowania widm impedancyjnych zastosowano zastępcze obwody elektryczne, składające się z połączonych szeregowo rezystora  $R_0$  i trzech (lub dwóch) połączonych równolegle par rezystorów i kondensatorów ( $R_i$ ,  $C_i$ ). Błędy dopasowań w każdym przypadku nie przekraczały 0,7%. Wartości współczynników *m* oraz  $\alpha$ , otrzymane z równania Arrheniusa oraz dopasowań, wskazują, że dla katody Ag|BSCF|Ag etap przenoszenia ładunku limituje szybkość reakcji, podczas gdy dla elektrod BSCF i Ag|BSCF etapem limitującym jest transport jonów tlenkowych poprzez BSCF. Energia aktywacji dla oporu polaryzacyjnego elektrod BSCF, Ag, Ag|BSCF i Ag|BSCF|Ag jest zbliżona i wynosi odpowiednio 55,1 kJ mol<sup>-1</sup>, 47,7 kJ mol<sup>-1</sup> 47,7 kJ·mol<sup>-1</sup> i 37,9 kJ·mol<sup>-1</sup>

Słowa kluczowe: stałotlenkowe ogniwa paliwowe, katoda kompozytowa, tlenek mieszany baru strontu kobaltu i żelaza, elektrochemiczna spektroskopia impedancyjna

# 1. Introduction

Solid oxide fuel cells (SOFCs) show the highest efficiency among all known types of fuel cells [1]. The big disadvantage of SOFCs is the high temperature of work (800-1000 °C) necessary to achieve a sufficiently high rate of the oxygen reduction reaction (ORR) at a cathode of the cell. Ba<sub>0.5</sub>Sr<sub>0.5</sub>Co<sub>0.8</sub>Fe<sub>0.2</sub>O<sub>3-5</sub> (BSCF) reveals one of the greatest catalytic activity in ORR at relatively low temperatures [2]. Zhou *et al.* stated that the oxygen reduction occurred on the overall surface of BSCF [3], and therefore the optimization of the surface is expected for the further reduction of polarization resistance of the BSCF cathode. Wang *et al.* found the electrical conductivity of BSCF at the same level of ~43 S·cm<sup>-1</sup> in the temperature range from 500 °C to 900 °C [4]. Such a low value influences the cathode performance.

The average TEC of BSCF in the temperature range of 30-850 °C is equal to  $18.53 \cdot 10^{-6}$  K<sup>-1</sup> [4]. Some properties of the cathode can be improved by addition of a second phase to the BSCF. Silver seems to be an ideal candidate. It reveals the greatest electronic conductivity among metals, is cheap, is ductile, and has excellent catalytic activity in the ORR [5].

Several methods of incorporation of silver to BSCF electrodes were described. Zhang et al. used paste containing 14% silver for the preparation of a composite Ag-BSCF cathode [6]. These authors reported that the incorporation of Ag into the BSCF cathode led to an increase in the electronic conductivity by at least 80%, although it lowered the power densities of the cells. The obtained cathodes reveal the reduced ohmic resistance and the enhanced catalytic O<sub>2</sub> reduction performance in comparison to the pure BSCF cathode. Tatko et al. proved that silver had a greater impact on the cathode performance when the oxygen concentration was low, so the presence of silver between the electrode and the electrolyte should lead to the noticeable increase in the cathode performance [7]. The silver layer applied between the cathode and the electrolyte increased the Ni-GDC|SDC|LSCF cell performance [8].

The goal of this work is to draw a comparison among the BSCF cathode, the Ag cathode, the cathode with silver interlayer between BSCF and electrolyte (Ag|BSCF) and the Ag|BSCF cathode with the additional silver layer on the top of the cathode (Ag|BSCF|Ag). The silver layers should improve the conductivity in the direction parallel to the electrolyte surface, and take part in ORR due to a large number of effective surface sites at the silver surface available for absorption and dissociation of oxygen species [9], improving the overall cathode performance.

## 2. Experimental

#### 2.1. Materials

Commercial lanthanum strontium cobalt ferrite (LSCF) paste (Fuel Cell Materials, USA), Ag powder of 0.3-1.5 µm grain size (Institute of Electronic Materials, Poland), Ink Vehicle (Fuel Cell Materials, USA) and BSCF prepared as described in [10] were used for the preparation of electrodes. Silver and BSCF pastes suitable for the preparation were made from the powders thoroughly mixed with the Ink Vehicle. Platinum foil was used as a current collector. Samaria doped ceria (SDC) electrolyte discs of 1 mm in thickness and 20 mm in diameter were prepared as described in [11]. Counter electrodes were prepared from the LSCF paste by the screen-printing method. The LSCF paste covered the entire one side of each electrolyte disc. The discs covered with LSCF were sintered for 2 h at 1100 °C in air with a heating and cooling rate of 1 °C·min<sup>-1</sup>, and then cut in half. Two identical BSCF electrodes were made by screen-printing on the other side of the half-discs and sintered using the same route as the LSCF electrode. Obtained by screen-printing circle shapes of 5 mm in diameter electrodes are shown in Fig. 1a, and a schematic view of electrochemical cell is presented in Fig. 1b.

One of the twin electrodes acts as the working electrode and the second one as the reference electrode. Silver electrodes were sintered for 15 min at 800 °C with a heating and cooling rate of 1 °C·min<sup>-1</sup>. The silver electrodes were used for preparation of the Ag|BSCF electrode. The BSCF paste was screen-printed on the silver electrodes and sintered at 800 °C for 15 min with a heating and cooling rate of 1 °C·min<sup>-1</sup>. The Ag|BSCF electrode was used for preparation of Ag|BSCF|Ag electrodes. The Ag paste was screen-printed on the Ag|BSCF electrodes and sintered for 15 min at 800 °C with a heating and cooling rate of 1 °C·min<sup>-1</sup>. The BSCF, Ag, Ag|BSF and Ag|BSCF|Ag electrodes were characterized by electrochemical impedance spectroscopy (EIS) and scanning electron microscopy (SEM).



Fig. 1. Screen-printed BSCF electrodes (a), and schematic view of the electrochemical cell with Ag|BSCF|Ag electrode; 1 – platinum foil, 2 – LSCF counter electrode, 3 – SDC electrolyte, 4 – Ag layer, 5 – BSCF electrode.

#### 2.2. Methods

Cells were mounted in the test fixture which consisted of a gas-tight outer alumina tube of 300 mm in length and an inner alumina tube with 6 channels presented in Ref. 10 in Figs. 5a and 5b. Electrochemical impedance spectroscopy (EIS) measurements were conducted using a Gamry 300 series potentiostat/galvanostat/ZRA at the atmospheric pressure of oxygen and various  $P_{O_0}/P$  ratios in mixtures of oxygen (99.5%, Linde gaz Polska) and argon (99.99%, Linde gaz, Polska) with a flow rate of the gas through the cell of 150 cm<sup>3</sup>·min<sup>-1</sup>. The measuring system was equilibrated before every set of measurements for 8 h at 700°C in oxygen and for 15 min after each change of oxygen concentration. A frequency range used in measurements was 0.01-300,000 Hz at the logarithmic frequency steps of ten points per decade. An amplitude of the sinusoidal voltage signal was 10 mV. SEM images were recorded by means of a JEOL JSM-7500F field-emission apparatus operated at 15 kV.

#### 3. Results and discussion

## 3.1. SEM image

The pictures obtained by SEM showing the morphology of the electrodes are presented in Fig. 2.

The silver layer (Fig. 2a) consists of well-connected silver grains forming a 2D silver net which is easy to be penetrated by gases. This feature ensures a long enough line of triple phase boundary (tpb) where ORR occurs at the silver electrode [12].

The BSCF electrode (Fig. 2b) reveals 3D structures well sintered to the electrolyte. According to Wang *et al.* [4], the

sintering temperature of 1100°C leads to the lowest ASR for the BSCF cathodes.

Due to the melting point of silver being equal to 961°C, the sintering temperature was 800°C for the preparation of Ag|BSCF and Ag|BSCF|Ag. The BSCF grains sintered at 1100°C reveal better contact to each other and lower porosity than in the case of cathodes sintered at lower temperature (Fig. 2c). High density of the material provides good ionic transport conditions but limits the oxygen flux through the electrode layer. In the Ag|BSCF electrode (Fig. 2c) the BSCF structure is connected mainly to the silver layer whereas only in few places is connected to the electrolyte. The low ionic current limited by weakly contacted grains restricts the electrode reaction at the BSCF surface, so at this electrode the ORR reaction which occurs at the tpb gives the considerable contribution to whole ORR. This is also reflected in the similarity of shapes of impedance spectra in case of the Ag and Ag|BSCF electrodes (Figs. 3a and 3b).

The microstructure of the Ag|BSCF|Ag composite cathode is presented in Figs. 2d-2f. The BSCF structure is intertwined with the inner silver net and well enough connected to the electrolyte. This assures that a large number of oxygen ions created in ORR at the BSCF surface can be transported to the electrolyte. It is also reflected in the EIS spectra. The obtained ASR values for the composite electrode are much lower. This electrode reveals also high porosity and greater surface area than the BSCF electrode. A flat silver layer on the top of the electrode improved the electrode-current collector contact. Both silver and BSCF grains are well-sintered what is the reason of good ionic and electron transport.



Fig. 2. SEM pictures determined in the COMPO mode of Ag electrode (a), BSCF electrode (b), Ag|BSCF electrode (c), inner layer of the Ag|BSCF|Ag electrode (d), layer adjoined to the Ag|BSCF|Ag electrolyte (e) and outer layer of the Ag|BSCF|Ag electrode (f).

# 3.2. Electrochemical impedance spectroscopy

The examples of impedance spectra in Nyquist representation of the examined electrodes are presented in Fig. 3.

The whole impedance spectra can be divided in the two or three semicircle-like shapes. EIS spectra depended on the oxygen concentration. For Ag and Ag|BSCF electrodes in the impedance spectra the medium and low frequency semicircles overlap each other whereas spectra of BSCF electrodes reveal two distinguishable semicircles. In Fig. 3d for low frequencies and at higher oxygen concentrations, there are visible two semicircles. However, for lower oxygen concentrations, there are visible well separated three semicircles. Equivalent electrical circuits (EECs) consisting of a resistor  $R_0$  and three (or two) parallel connected resistor and capacitor pairs ( $R_i$ ,  $C_i$ ), connected in series, were used for fitting the impedance spectra. Each pair reflects at least one step of overall ORR. Constant phase elements (CPEs) were used instead of capacitors.

The impedance of the CPE may be expressed by the formula [13]:

$$Z_{CPE} = \frac{1}{2\pi f_0 C_0} \left(\frac{f_0}{jf}\right)^{\alpha}$$
(1)

where *f* is the frequency,  $f_0$  is the frequency of the reference,  $C_0$  is the capacitance at the frequency of reference, *j* is the imaginary unit and  $\alpha$  is a coefficient equal to 1 for a capacitor and assuming a value between 0.7 and 1.0 for non-ideal capacitance and close to 0.5 for a diffusion process. The EEC used for fitting impedance spectra and a sample of the fitted results are presented in Fig. 4.

The fitting errors were smaller than 0.7% in all cases. The assessment of the error was carried as described in detail in Ref. 12. In Fig. 4  $R_0$  and the ( $R_1$ , CPE<sub>1</sub>) pair represent the



Fig. 3. EIS spectra in the Nyquist representation at 700 °C and oxygen concentrations  $P_{o_2}/P$  of 1 (black square), 0.5 (black triangle), 0.2 (black circle), 0.1 (black diamond), 0.05 (filled, black triangle), 0.02 (filled, black circle), 0.01 (filled, black diamond), 0.005 (grey triangle), 0.002 (grey circle), 0.001 (grey diamond): a) BSCF electrode, b) Ag electrode, c) Ag|BSCF electrode, and d) Ag|BSCF|Ag electrode.



Fig. 4. Equivalent electrical circuits used for fitting impedance spectra (a); an example of the fitted spectrum of Ag electrode at 700 °C and  $P_{o,r}/P = 0.2$  (b); crosses – measured data, circles – fitted data, the numbers near filled symbols denote the logarithm of the frequency.

impedance of the electrolyte and the geometrical impedance of the cell.

The resistance connected to each slow step of the reaction depends on oxygen concentration:

$$\log(R) = a - m \log(P_{O_2}) \tag{2}$$

where *a* and *m* are coefficients. The resistance of the cathodic reaction (ASR) is equal to  $R_2$  or to the sum of the  $R_2$  and  $R_3$  for two or three R-CPE pairs used in EEC. The dependence of resistances on the oxygen partial pressure at 700° C are shown in Fig. 5a. Arrhenius plots of the electrode reaction resistances as a function of temperature is presented in Fig. 5b.

The Ag|BSCF|Ag cathode reveals the lowest ASR in the whole examined temperature range whereas higher ASR of the other examined cathodes are similar to each other. In the case of the Ag|BSCF|Ag cathode, a large amount of catalytic active sites both on the inner and outer silver layer facilitates ORR. The values of  $\alpha$  and m (eqs. (1) and (2)) can be used for the determination of a corresponding process type [10, 14, 15, 16, 18]. The calculated m and  $\alpha$  values are shown in Table 1. For the process responsible for the main part of the Ag|BSCF|Ag electrode reaction resistance described by  $R_3$  the coefficient *m* being close to -0.5 may point to a charge transfer reaction with molecular oxygen involved [10,16], the dissociative adsorption of molecular oxygen at the electrode surface [18], or the surface diffusion [19]. However,  $\alpha$  parameter varies between 0.7 and 0.83. This indicates the charge transfer reaction. For the BSCF and Ag|BSCF electrodes parameters *m* concerning both  $(R_2, CPE_2)$  and  $(R_3, CPE_3)$  processes are close to -0.25. According to Wang et al. [18] this value can be ascribed to the transport of oxygen ions from the site on the electrode surface through the electrode to the electrode|electrolyte interface.

The apparent activation energy of the ASR was determined from the Arrhenius plot presented in Fig. 5b. The activation energies of the BSCF, Ag, Ag|BSCF and Ag|BSCF|Ag cathodes are similar and yield 55.1 kJ·mol<sup>-1</sup>, 47.7 kJ·mol<sup>-1</sup> , 47.7 kJ·mol<sup>-1</sup> and 37.9 kJ·mol<sup>-1</sup> respectively. All activation energies are close to 55 kJ·mol<sup>-1</sup> which was reported for the Ce<sub>0.9</sub>Gd<sub>0.1</sub>O<sub>1.95</sub> oxygen ionic conductivity in Ref. 20. It is additional confirmation that in cases where m = -0.25 (the BSCF and Ag|BSCF cathodes) the transport of oxygen ions through the bulk of the cathode is the rate determining step. In each electrode the low *m* coefficient for  $R_0$  and  $R_1$  being close to 0 (an exception is  $m_1$  for the Ag|BSCF|Ag electrode) corroborates that these resistances represent the ohmic resistance and the geometrical impedance of the cell. The silver electrode does not reveal ionic conductivity and the ORR occurs mainly at the tpb [12]. The obtained activation energy is slightly lower than 89 kJ·mol<sup>-1</sup> reported for the silver point electrode [12].

#### 4. Conclusions

Ag, BSCF electrodes and Ag|BSCF, Ag|BSCF|Ag composite electrodes were obtained and tested. The electrodes reveal well connected structure and reasonable catalytic activities in ORR. The presence of silver in the cathodes improved the electric conductivity of the cathode and reduced the ASR. The Ag|BSCF|Ag electrode reveal the lowest ASR. The rate determining step for this electrode is found to be the charge transfer reaction whereas the transport of oxygen ions from the electrode surface to the electrode|electrolyte interface in the case of BSCF and Ag|BSCF electrodes.



Fig. 5. ASR versus oxygen partial pressure (a) and Arrhenius plot of the electrode reaction resistances for the BSCF (circles), Ag (squares), Ag|BSCF (triangles) and Ag|BSCF|Ag (diamonds) cathodes (b).

Table 1. Coefficients m and  $\alpha$  ( $m_{x'} \alpha_x$  respectively for  $R_{0_1} R_{1_2} R_2$  and  $R_3$ ) of the BSCF, Ag, Ag/BSCF and Ag/BSCF/Ag cathodes measured at 700 °C and P/P<sub>02</sub> = 0.2.

	m <sub>o</sub>	<i>m</i> <sub>1</sub>	α,	<i>m</i> <sub>2</sub>	α <sub>2</sub>	m <sub>3</sub>	α <sub>3</sub>
BSCF	- 0.054	- 0.051	0.5-0.58	- 0.23	0.65-0.75	-	-
Ag	- 0.048	- 0.017	0.56-1.0	- 0.26	0.50-0.57	- 0.27	0.60-0.73
Ag BSCF	- 0.034	- 0.009	0.5-0.72	- 0.25	0.50-0.60	- 0.23	0.73-0.79
Ag BSCF Ag	- 0.017	- 0.217	0.5-0.53	- 0.11	0.53-0.84	- 0.46	0.70-0.83

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