



BaCo_{0.7}Fe_{0.2}Nb_{0.1}O_{3-δ} as cathode material for intermediate temperature solid oxide fuel cells

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ABSTRACT

BaCo_{0.7}Fe_{0.2}Nb_{0.1}O_{3-δ} (BCFN) has been synthesized and characterized as cathode material for intermediate temperature solid oxide fuel cells (IT-SOFCs) using La_{0.8}Sr_{0.2}Ga_{0.83}Mg_{0.17}O_{3-δ} (LSGM) electrolyte. X-ray diffraction results show that pure cubic BCFN perovskite phase can be obtained at 950 °C through solid state reactions of BaCO₃, Co₃O₄, Fe₂O₃ and Nb₂O₅. The electrical conductivity of BCFN increases with the increase in oxygen partial pressure, indicating that BCFN is a p-type semiconductor. The polarization resistance of the BCFN cathode with LSGM electrolyte is only 0.06 Ω cm² at 750 °C in air under open-circuit conditions. The overpotential at a current density of 1 A cm⁻² in oxygen was only about 0.04 V at 750 °C. Peak power densities of 550, 770 and 980 mW cm⁻² have been achieved on LSGM-electrolyte supported single cells with the configuration of Ni-Gd_{0.1}Ce_{0.9}O_{1.95}|La_{0.4}Ce_{0.6}O₂|LSGM|BCFN at 700, 750 and 800 °C, respectively. These results indicate that BCFN is a very promising cathode candidate for IT-SOFCs with LSGM electrolyte.

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1. Introduction

In recent years, great efforts have been devoted to develop intermediate temperature solid oxide fuel cells (IT-SOFCs) operating at 650–800 °C [1]. The decrease in the SOFC operating temperature is expected to reduce reactivity among the cell components and therefore improve fuel cell durability. In addition, much cheaper metallic interconnects and gas manifold materials can be used, leading to more cost-competitive SOFC systems. However, reducing the SOFC operating temperature typically leads to a significant decrease in the electrode kinetics and substantial increase in the electrode interfacial polarization resistances, especially for the cathode [2]. In order to lower the cathode polarization resistance, materials with mixed electronic and ionic conductivity as well as high oxygen reduction catalytic activity have been extensively studied as cathode for IT-SOFCs [3]. Most of these compounds are perovskites, such as ABO₃-type oxides in which A site is occupied by Ln, Sr and Ba while the B-site is occupied by Cr, Mn, Fe, Co, and Ni [4]. Many mixed conductors with high oxygen permeability at elevated temperatures perform well as cathodes of IT-SOFCs, such as Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-δ} [5]. However, cobalt containing perovskite materials typically lack chemically stability and com-

patibility; BSCF can be easily disintegrated, especially under CO₂ rich atmosphere at elevated temperatures. Recently, it has been reported that introduction of Nb in the B-site of cobalt-based perovskites can significantly improve chemical stability and enhance oxygen permeability [6]. BaCo_{0.7}Fe_{0.2}Nb_{0.1}O_{3-δ} (BCFN) has been evaluated as promising oxygen transport membrane material [7] as well as cathode material for IT-SOFCs with Ce_{0.9}Gd_{0.1}O_{1.95} (GDC) electrolyte [8]. BCFN has been reported to show a high oxygen permeation flux of greater than 20 ml min⁻¹ cm⁻² during a reaction period of 300 h [9]. Further, a maximum power density of 618 mW cm⁻² has been achieved for IT-SOFCs using BCFN as cathode with the cell configuration of Ni-GDC|GDC|BCFN at 800 °C [8].

Although promising performance of IT-SOFCs using BCFN as cathode with GDC electrolyte has been demonstrated, there is an urgent need in evaluating the cathode performance of BCFN with other potential IT-SOFC electrolyte since the oxygen reduction behavior of the cathode/electrolyte interface plays a vital role on the cell performance for IT-SOFCs [10]. La_{0.8}Sr_{0.2}Ga_{0.83}Mg_{0.17}O_{3-δ} (LSGM) has been considered as excellent electrolyte for intermediate temperature SOFCs due to its high ionic conductivity and predominately oxide ionic conductivity over a broad oxygen partial pressure and operating temperature range in comparison with that of GDC electrolyte which is considered as the electrolyte for SOFCs operating below 650 °C since GDC has substantially high electronic conductivity at temperature higher than 650 °C [11]. Therefore, it will be valuable to study the performance of BCFN as cathode for IT-SOFCs using LSGM electrolyte.

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Furthermore, there has been a lack of detailed study on the synthesis and characterization of BCFN as cathode for IT-SOFCs in the literature. BCFN with a pure cubic structure has been typically obtained at high calcination temperatures of 1050–1150 °C using a solid state reaction process [7,8]. If pure cubic BCFN structure can be obtained at lower calcination temperature through the solid state reaction approach, BCFN powders will have smaller particle sizes and consequently higher activity, which will be beneficial for BCFN as a cathode material for IT-SOFCs. Lower calcinations temperature to achieve BCFN pure phase will also reduce processing cost since less energy input is needed for the calcination process.

In this work, synthesis and the cathode performance of BCFN on LSGM electrolyte have been systematically studied. Pure cubic BCFN perovskite can be obtained at a much lower temperature of 950 °C. BCFN has been found to have low over-potential losses and high performance at around 650–800 °C, indicating that it can be a very promising cathode candidate for IT-SOFCs based on LSGM electrolyte.

2. Experimental

2.1. Material synthesis and characterization

BCFN was prepared using a solid-state reaction method. Stoichiometric amount of BaCO₃, Co₃O₄, Fe₂O₃ and Nb₂O₅ was mixed by ball milling. Calcination of the precursor powders was performed at 650 °C, 750 °C, 800 °C, 850 °C, 900 °C, 950 °C and 1050 °C for 4 h in air, respectively. Samples calcined at 950 °C were ball milled with ethanol using zirconia balls, dried, and then compacted into pellets (25 mm in diameter) and bars (64 mm × 4 mm × 6 mm), respectively, using uniaxial pressing at 250 MPa. Dense samples (above 95% relative density) were obtained by sintering the compacts at 1130 °C for 6 h in air. Thermogravimetric-differential scanning calorimetry (TG-DSC) of the precursor powders was performed using a Netzsch STA 409C. The particle size distributions of precursor powders were determined using a laser particle size analyzer (SEISHIN LMS-30), and *d*₅₀ was found to be 1.3 μm. Phase structures of the powders calcined at different temperature were determined using X-ray diffraction (XRD) in the 2θ range of 10–80°, and Cu-Kα radiation was used as the X-ray source. The crystal structure and lattice parameter were obtained through analysis of the XRD patterns.

The conductivities of BCFN under different atmospheres (air, O₂ and Ar) were measured by direct current (DC) four probe method using Arbin Electrochemical Test Station (BT2000). Microstructure of BCFN cathode was observed using a thermally assisted field emission scanning electron microscope (FEI XL30 S-FEG).

2.2. Cell fabrication and testing

LSGM electrolyte was prepared by a solid state reaction method. The starting materials were La₂O₃, SrCO₃, Ga₂O₃, and MgO. The ball-milled mixture of the starting materials was calcined in air at 1250 °C for 10 h. LSGM powder was pressed uniaxially at 200 MPa to form pellets and then sintered at 1450 °C for 12 h. BCFN, La_{0.4}Ce_{0.6}O₂ (LDC) and NiO-Gd_{0.1}Ce_{0.9}O_{1.95} (Ni-GDC) (60 wt%: 40 wt%) powders were mixed with a Heraeus binder V006 (weight ratio of 1:1) to form electrode inks.

For symmetrical cells (shown in Fig. 1a), BCFN ink was printed on both sides of the LSGM pellets (having a diameter of ~13 mm and a thickness of about ~0.5 mm) and fired at 1000 °C for 2 h. The area of BCFN electrode was 0.33 cm² and the thickness was ~20 μm. Ag paste was used as current collectors on the BCFN electrodes.

The overpotential of BCFN cathode was tested with a three-electrode configuration (shown in Fig. 1b), similar to that reported

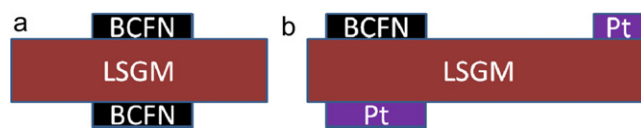


Fig. 1. Schematic diagrams of (a) a symmetrical cell and (b) a three-electrode cell.

in the literature [12]. BCFN working electrode (WE) was screen-printed on the LSGM electrolyte and sintered at 1000 °C for 2 h. Pt reference electrode (RE) and Pt counter electrode (CE) were subsequently applied on the LSGM electrolyte and sintered at 900 °C for 2 h. The distance between the WE and RE was ~3 mm. The areas of both WE and CE were 0.33 cm² and the thickness was ~20 μm. The area of RE was 0.1 cm². The overpotential was measured by monitoring the current in the cell when a potential step was applied to the WE, and was determined by the following equation:

$$\eta_{WE} = U_{WR} - iR_{el} \quad (1)$$

where η_{WE} is the cathode overpotential, U_{WR} is the applied voltage between WE and RE, i is the current flowing through the cell and R_{el} is the resistance of the electrolyte obtained from the impedance spectrum of the cell.

Single cells were fabricated using LSGM electrolyte pellets with thickness of ~300 μm. A layer of LDC was first printed on one side of the LSGM pellet and fired at 1350 °C for 2 h. NiO-GDC ink was subsequently printed on the surface of the LDC buffer layer and fired at 1150 °C for 2 h. BCFN ink was then printed on the other side of the LSGM pellet and fired at 1000 °C for 2 h to form LSGM-electrolyte supported single cells with the configuration of NiO-GDC|LDC|LSGM|BCFN cells. The BCFN cathode with an area of 0.33 cm² was about 20 μm thick after firing. Pt paste was used as current collectors for both electrodes and the cells were tested in a four-probe configuration. H₂ at a flow rate of 50 ml min⁻¹ was used as the fuel and ambient air was used as the oxidant. All the electrochemical characterizations were performed with a Versa STAT 3-400 test system (Princeton Applied Research), with frequency range of 0.01–100 kHz and input sinusoidal signal amplitude of 10 mV for the impedance measurement.

3. Results and discussion

3.1. Synthesis process

Fig. 2 shows the TG-DSC plots of BCFN precursor powders. TG plot shows a fairly sharp decrease in weight at 700–900 °C, coincident with the main reaction temperature zone. Three endothermic

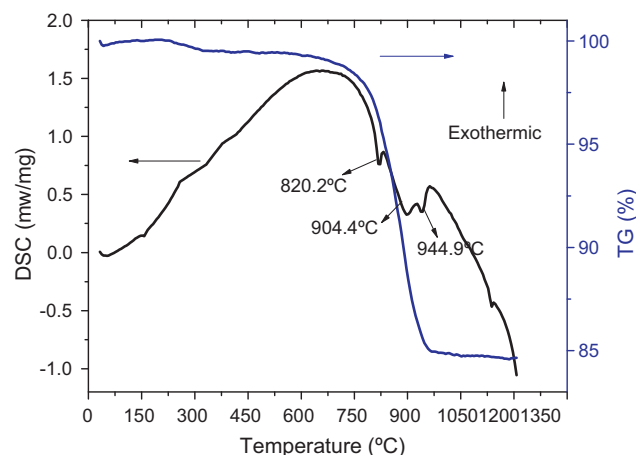


Fig. 2. TG-DSC plot of BCFN precursor powders.

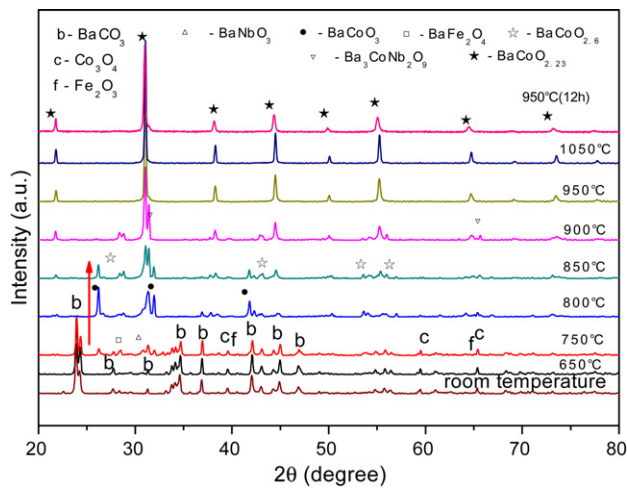
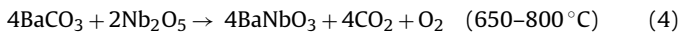
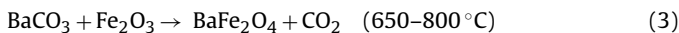


Fig. 3. XRD patterns of BCFN precursor powders calcined at different temperatures.

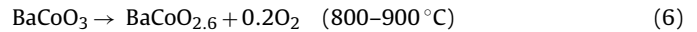
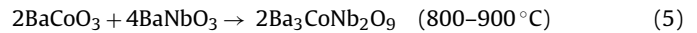
peaks were found at 820.2, 904.4 and 944.9 °C in the DSC plot, which are corresponding to the formation of new phases. Fig. 3 shows the XRD patterns of the precursor powders calcined at 650 °C, 750 °C, 800 °C, 850 °C, 900 °C, 950 °C and 1050 °C. As shown in Fig. 3, no significant change was observed from room temperature to 650 °C, and the main phases were the same as those of the precursor powders: orthorhombic BaCO₃ (71–2394), cubic Co₃O₄ (74–1656) and rhombohedral Fe₂O₃ (89–0599). Nb₂O₅ is not observable in the XRD pattern due to its relatively low weight ratio in the raw material mixture.

As shown in Figs. 2 and 3, the BCFN synthesis process can be divided into three steps. Firstly, several intermediary reaction products were formed from 650 °C to 800 °C, including BaNbO₃ (87–0248), BaFe₂O₄ (70–2468) [13] and BaCoO₃ (74–0902). The amount of BaCoO₃ increased from 650 °C to 800 °C, coincident with the fairly sharp decrease in weight as seen in Fig. 2. The weight loss observed in Fig. 2 can be primarily ascribed to the following reactions [14]:

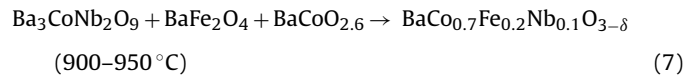


Subsequently, hexagonal Ba₃CoNb₂O₉ (46–0997) and hexagonal BaCoO_{2.6} (71–2453) were formed when the temperature was increased from 800 °C to 900 °C [15]. It was observed that the amounts of BaCoO₃ decreased as the temperature was raised from

800 °C to 850 °C, and BaNbO₃ disappeared. Therefore, it showed that BaCoO₃ and BaNbO₃ were transformed into Ba₃CoNb₂O₉ with a corresponding endothermic peak centered at 820.2 °C in the DSC plot. Meanwhile, some BaCoO₃ was transformed into BaCoO_{2.6}, leading to a release of O₂ and a corresponding exothermic peak at 904.4 °C in the DSC plot, as shown in the following reactions:



Finally, when the sintering temperature was increased to 950 °C, both Ba₃CoNb₂O₉ and BaCoO_{2.6} disappeared and a single cubic BCFN phase of BaCoO_{2.23} (75–0227) perovskite structure was formed, corresponding to an exothermic peak at 944.9 °C in the DSC plot. The reaction may be written as:



Consequently, pure cubic BCFN perovskite can be obtained at 950 °C under a suitable sintering procedure. This is the lowest synthesis temperature being reported to obtain pure cubic BCFN perovskite phase. Hexagonal and cubic phases of BCFN were reported to co-exist even after the sample was calcined at 950 °C [8,16], indicating that the raw material and calcination process are critical to obtain single cubic BCFN perovskite phase. BCFN XRD peak intensity increased remarkably when the calcination temperature was increased to 1050 °C, indicating that BCFN powders with higher crystallinity have been obtained.

3.2. Electrical conductivity

Dense BCFN bar samples were used to measure the BCFN electrical conductivity. The surface and fracture SEM images of BCFN samples after the conductivity test are shown in Fig. 4a and b. It can be seen that the grain size in the BCFN sample is about 2–5 μm and the BCFN sample has a uniform dense microstructure with good particle-to-particle contact. The electrical conductivity of BCFN as a function of temperature under different atmospheres is shown in Fig. 5. For BCFN material, the oxygen ion conductivity is at least two orders of magnitude lower than the electronic conductivity [17]. Therefore, the electrical conductivity shown in Fig. 5 can be assumed to be primarily electronic conductivity. As shown in Fig. 5, the electrical conductivity increases with increasing oxygen partial pressure, indicating that BCFN is a p-type semiconductor with electronic holes as charge carriers. An electrical conductivity value of 12.4 S cm⁻¹ was obtained at 800 °C in air, similar to that of BaCo_{0.7}Fe_{0.2}Nb_{0.1}O_{3-δ} oxide (12 S cm⁻¹) reported

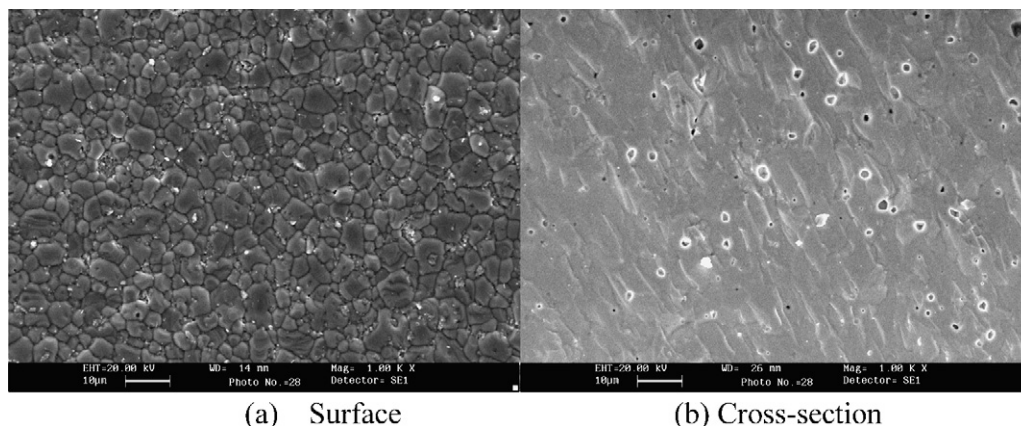


Fig. 4. (a) Surface and (b) fracture SEM images of BCFN sintered at 1130 °C.

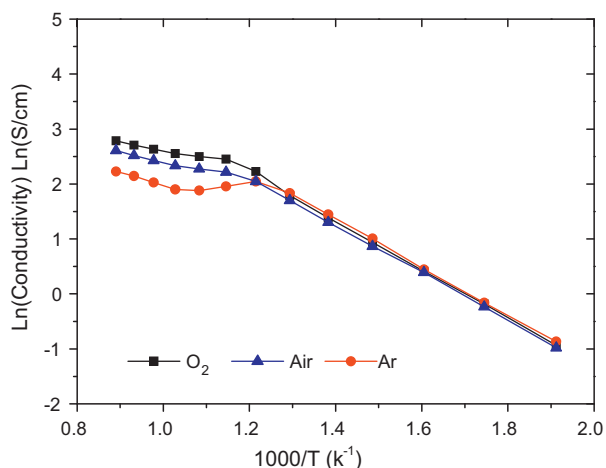


Fig. 5. Electrical conductivity of BCFN under air, O₂ and Ar atmospheres.

in the literature [7]. As it can be seen in the following section, BCFN cathode exhibits attractive electrochemical performance. In fact, several perovskite electrode materials with low electrical conductivity have been reported to possess excellent electrochemical performance. For example, the maximum conductivity of BSCF was about $\sim 35 \text{ S cm}^{-1}$ at $\sim 800^\circ\text{C}$ in air but BSCF has been shown to have very good electrochemical catalytic activity for oxygen reduction [18]. Generally, the conductivity increased gradually with the increase in temperature. However, there is some fluctuation in conductivity at $400\text{--}600^\circ\text{C}$, which may be due to the loss of the lattice oxygen leading to more oxygen vacancies as a result of the thermally induced lattice oxygen losses [17].

3.3. AC impedance study

Fig. 6a shows the interfacial resistances on BCFN symmetrical half cells tested at $600, 650, 700$ and 750°C in air under open-circuit conditions. In order to clearly show the polarization resistance, all bulk resistances were normalized to zero. Electrode polarization resistance values of $0.45, 0.26, 0.12$ and $0.06 \Omega \text{ cm}^2$ were obtained at $600, 650, 700$ and 750°C in air, respectively, comparable to the state-of-the-art intermediate temperature SOFC cathode of $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ (LSCF, cathode polarization resistance of $0.12 \Omega \text{ cm}^2$ at 700°C in air) [19]. From the polarization resistance versus temperature relationship shown in Fig. 6b, the activation energy of the BCFN electrode can be determined to be 100 kJ mol^{-1} , much lower than those of other cathode materials. For example, the activation energy for LSCF was estimated to be $135\text{--}142 \text{ kJ mol}^{-1}$ from symmetrical half cell results [19]. Thus, BCFN is expected to

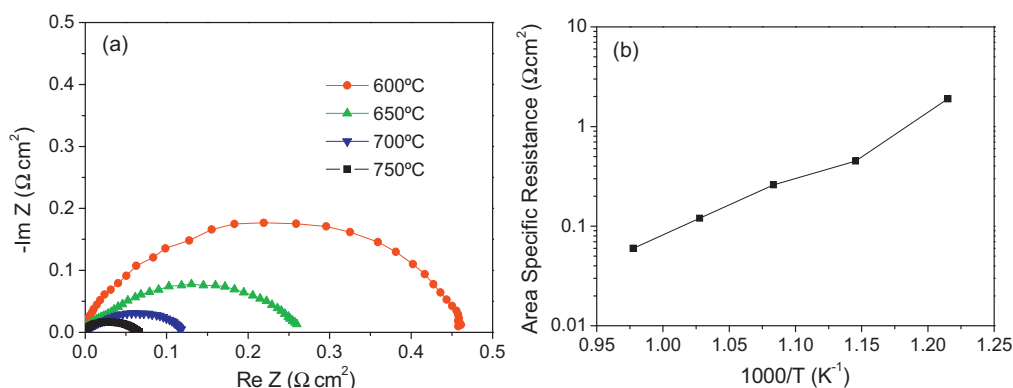


Fig. 6. (a) Impedance spectra and (b) area specific resistance of BCFN cathode as a function temperature.

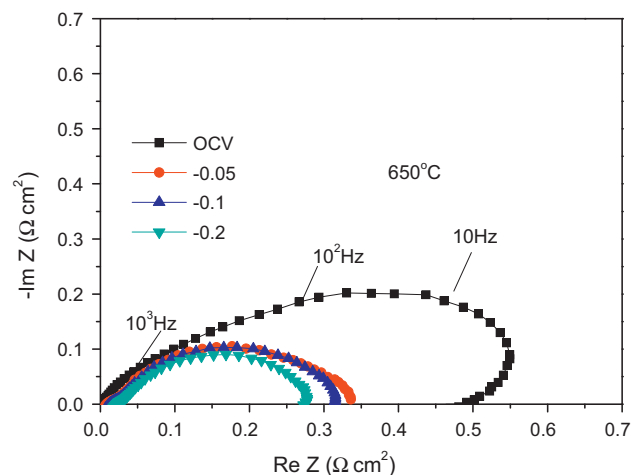


Fig. 7. Impedance spectra of BCFN cathode under DC polarization in air at 650°C .

have higher catalytic activity than LSCF, especially at lower operating temperature.

In order to study factors affecting the polarization resistance of BCFN cathode, the impedance spectra of BCFN cathode was recorded under DC polarization in air with a three-electrode configuration. Fig. 7 shows the impedance spectra of BCFN under DC polarization in air at 650°C . All DC polarization experiments were recorded when a steady state was reached, and the time required in establishing an equilibrium state for each DC polarization measurement varied with the experimental conditions. As can be seen in the figure, the cell resistance decreases with increasing polarization overpotential at 650°C , indicating that the rate-limiting step of the electrode reaction is an electrochemical, not a pure chemical reaction. The assembly shows rather high activity for oxygen reduction, especially under DC polarization [20], indicating that BCFN is a very promising cathode material for intermediate temperature SOFCs.

3.4. Cathode overpotential

Fig. 8 shows the cathode overpotential of BCFN as a function of current density at different temperatures in air obtained by linear sweep voltammetry (LSV) using a three-electrode configuration as presented in Fig. 1b. LSV experiments were carried out from 0 to -0.8 V , with a 10 mV s^{-1} potential scan rate at various temperatures. The ohmic drop of the cell was determined from the impedance spectra, and then subtracted from the polarization

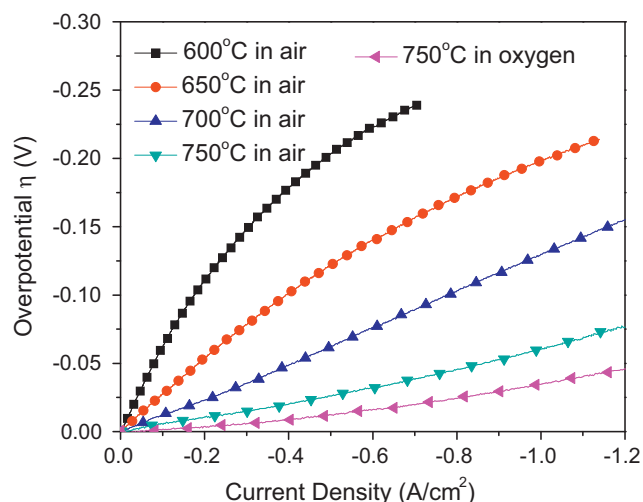


Fig. 8. BCFN cathode overpotential measured using a three-electrode configuration at various temperatures in air and oxygen.

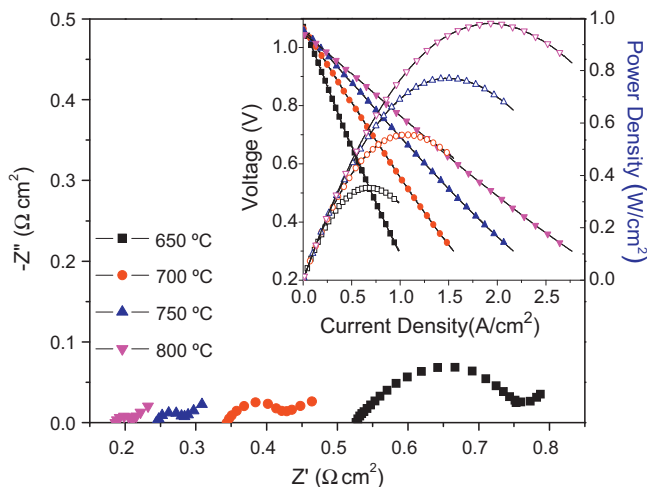


Fig. 9. Performance of the single cell (Ni-GDC|LDC|LSGM|BCFN) with H₂ as fuel and ambient air as oxidant at different temperatures.

potential. Generally, the overall oxygen reduction reaction over a BCFN electrode can be expressed as:



where $\text{V}_\text{O}^{\bullet\bullet}$ represents oxygen vacancy and $\text{O}_\text{O}^{\text{X}}$ represents oxygen ion in the electrolyte, respectively. BCFN shows rather high activity for oxygen reduction, especially with oxygen as the oxidant. For example, the overpotential at a current density of 1 A cm⁻² in oxygen is only about 0.04 V at 750 °C as shown in Fig. 8, even lower than that of high performance composite cathode of $\text{Sm}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$ and $\text{La}_{0.8}\text{Sr}_{0.2}\text{Ga}_{0.8}\text{Mg}_{0.15}\text{Co}_{0.05}\text{O}_3$ in similar testing conditions (0.08 V at 750 °C in oxygen) [12].

3.5. Single cell performance

The performance of BCFN cathode was tested in LSGM electrolyte supported single cell with Ni-GDC as the anode. LDC interlayer was applied between the anode and electrolyte to prevent reactions between LSGM and Ni. Current–voltage (I – V) and

current–power (I – P) curves obtained at different temperatures with H₂ as fuel and ambient air as oxidant are shown in the inset of Fig. 9. It can be seen that the maximum power densities are 550, 770 and 980 mW cm⁻² at 700, 750 and 800 °C, respectively, similar to those of the cells of Ni-GDC|LDC|LSGM|LSC-LDC (LSC: $\text{La}_{0.9}\text{Sr}_{0.1}\text{CoO}_3$, 1100 mW cm⁻² at 800 °C) with the thickness of the LSGM electrolyte of ~60 μm [21].

The impedance spectra of the single cell obtained under open-circuit conditions at different temperatures are shown in Fig. 9. The cell polarization resistance is 0.12 Ω cm² at 750 °C, only half of the cell ohmic resistance (0.24 Ω cm²), indicating that the electrolyte thickness limits the cell power output. Consequently, the cell performance is expected to be further improved by decreasing the thickness of the electrolyte to reduce the ohmic losses. All these results suggest that BCFN is a very promising cathode candidate for IT-SOFCs using LSGM electrolyte.

4. Conclusions

Cubic BCFN perovskite structure single phase has been synthesized at 950 °C using a solid state reaction method. The electrical conductivities of BCFN under different oxygen partial pressures were measured, and a conductivity of 16.2 S cm⁻¹ was obtained at 850 °C in oxygen. The polarization resistance varied with temperature, and the lowest polarization resistance value of 0.06 Ω cm² was obtained in air at 750 °C. The overpotential at a current density of 1 A cm⁻² in air was only about 0.04 V at 750 °C. In addition, single cells with the configuration of Ni-GDC|LDC|LSGM|BCFN show peak power density of 980 mW cm⁻² at 800 °C using H₂ as the fuel and ambient air as the oxidant. These results indicate that BCFN is a very promising cathode for intermediate-temperature solid oxide fuel cells with LSGM electrolyte.

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