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Microstructural characterization and electrochemical properties of Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$ as a novel cathode for SOFCs

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Abstract

The microstructure and electrochemical properties of a novel cathode Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$ were discussed in the paper. After calcination at 1000 °C, a perfect single crystal was obtained. Some diffraction streaks along [200] and [112] patterns in HR-TEM appeared for the ordering of oxygen vacancies, or as the overlap of B-site cations (Co$^{2+}$/Co$^{3+}$ and Mn$^{2+}$/Mn$^{3+}$) with stacking fault-derived scattering of ordering. Area-specific resistance (ASR) was 0.023 Ω · cm$^2$ when Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$ was deposited on the electrolyte as the electrode at 700 °C in air. The maximum power density and the maximum OCV were 592.80 mW cm$^{-2}$ at 650 °C and 0.89 V at 550 °C for a single cell, respectively. Hence, the material Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$ could be considered as an air-electrode in Intermediate-temperature solid oxide fuel cells (IT-SOFC).

Introduction

Recently, more attention has been directed towards IT-SOFCs (Intermediate Temperature Solid Oxide Fuel Cells) for the shorter start-up time, the lower requirements as electrode materials and the low maintenance [1, 2]. However, lower operating temperatures raises some concerns, such as reducing kinetics of oxygen reduction reaction (ORR), which makes the catalytic activity of cathode materials poor [3, 4]. Hence, the most critical technology to develop IT-SOFC’s is searching for suitable electrode materials, especially those suitable for medium-temperature cathode materials [5, 6]. High conductivities of electronic and ionic are necessary for the cathode materials to ensure enough catalytic activity for ORR [7].

Among cathode materials, perovskite oxides [8–11] and Ruddlesden-Popper (R-P) series [12] attract significant attention and have been widely studied as promising electrode materials for IT-SOFC. Bu et al [13] and Tsvetkov et al [14] have reported that Nd replaced La in the LnSrCoMnO$_{6-\delta}$ and Ln$_{1-x}$Sr$_x$MnO$_3$ series of cathode materials (Ln represents the element from La to Gd) can obtain superior oxygen exchange activity. Kim et al [15–17] also proved that the addition of Nd at A-site could enhance the stability and improve the electrochemical performance of single cells. Electrode materials containing cobalt are considered to be the significantly promising since the element Co has three variable valences (Co$^{2+}$, Co$^{3+}$ and Co$^{4+}$) [18]. Double perovskites containing Co, such as PrBaCo$_2$O$_{5+\delta}$ and GdBaCo$_2$O$_{5+\delta}$ have exhibited excellent electrocatalytic activities for ORR at low temperatures [19–21]. However, some properties limit their applications, such as mismatch of thermal expansion coefficients, poor of long-term stabilities [22, 23]. Efforts have been made towards solving these problems. For example, Hong and Wang et al [24–26] proved that electrode materials mixed with Ce$_{0.8}$Sm$_{0.2}$O$_{1.9}$ (SDC) electrolyte in a certain ratio can decrease area specific resistance (ASR) and extend triple phase boundaries between fuel, electrode and electrolyte. Therefore, in this work, elements Nd and Sr were doped in A-site and Co and Mn were substituted with B-site to form Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$ (NSCM). The electrolyte was SDC, and the cathode material was the mixture of SDC with NSCM.
Experimental sections

NSCM electrode material was prepared by the method of EDTA-citric acid-nitrates. Sr(NO₃)₂, Nd(NO₃)₃ · 6H₂O, Co(NO₃)₃ · 6H₂O and C₆H₁₂MnO₈ as the raw materials were mixed in stoichiometric amounts (According to the molar ratio of the elements in the formula) in a beaker, added to deionized water, and stirred until all salts dissolved. In a separate mixture, EDTA and NH₃·H₂O were mixed to obtain EDTA-NH₃ · H₂O. The molar ratio of citric acid as a chelating agent was added in the EDTA-NH₃ · H₂O and then the complex was dropped into the nitrate mixture. Ammonia was added to balance pH value. Heating and stirring the nitrate mixture at 80 °C until sol-gel was obtained. The sol-gel was placed in an oven and continually heated at 200 °C until dry bubble film was obtained. Finally, the film was ground to get powder by ball mill, and then the powder was shifted into a muffle furnace to calcine at different temperatures for 12 h in air.

X-ray diffraction (XRD, Bruker D8) using Cu Kα radiation at RT was carried out to affirm phase formation of the cathode materials after calcination at different temperatures. Diffraction peaks were captured with 2θ range from 10 to 80 degrees when the step size is 0.02 degree.

X-ray diffraction patterns of NSCM electrodes after calcination at different temperatures (Figure 1). (a) XRD patterns (2θ range from 10° to 80°) of Nd₀.₅Sr₀.₅Co₀.₅Mn₀.₅O₃₋ₓ after calcination at different temperatures and PDF#89-0789 corresponding to Powder Diffraction File for Orthorhombic perovskite structure of (Nd₀.₅Sr₀.₅)(MnO₃). (b) The amplification of XRD patterns 2θ range from 20° to 60° (the impurity peaks were labeled with * and the composition was SrMnO₃).

Results and discussion

XRD patterns at RT 2θ from 10° to 80° was seen for Nd₀.₅Sr₀.₅Co₀.₅Mn₀.₅O₃₋ₓ powder which were calcined at an interval of 50 °C from 800 °C to 1000 °C were shown in figure 1(a). Some impurity peaks at 2θ = 27.26°, marked with * . According to the analysis of Jade 5, the impurity phase was SrMnO₃ corresponding to PDF# 84-1612 appeared when the samples were calcined before 950 °C (including 950 °C) as shown in figure 1(b). A perfect single crystal perovskite structure was prepared after calcination at 1000 °C. As shown in figures 1(a) and (b), the NSCM sample after calcination in 1000 °C, has a perfect orthorhombic perovskite structure with space group Imma (74). The diffraction patterns of NSCM samples had three main diffraction peaks at 2θ = 33.08°, 47.53°, 59.1°, corresponding to (200), (202), and (321) for a phase (Nd₀.₅Sr₀.₅)(MnO₃) (PDF#: 89-0789), respectively. Diffraction peaks were shifted 0.29° to the left, caused by increased lattice parameter of NSCM due to the large ionic radius of Co (0.745 Å) replaced partly the smaller ion radius of Mn (0.46 Å). The average crystallite sizes and lattice parameters of NSCM after calcination at 800, 850, 900, 950 and 1000°C calculated are listed in table 1. According to Scherrer’s formula as shown as follow:
Table 1. Crystallite sizes and Lattice parameters of NSCMn calcined at different temperatures.

| Calcination temperatures/°C | Crystallite sizes (degree) | D(nm) | a(Å) | Lattice (Å) | Parameters (Å) | V(Å³) | R(%) |
|-----------------------------|---------------------------|-------|------|-------------|----------------|--------|------|
| 800                         | 16.380                    | 0.514 | 15.93| 5.429       | 7.654          | 5.451  | 226.51 | 7.21 |
| 850                         | 16.496                    | 0.487 | 16.82| 5.419       | 7.649          | 5.438  | 225.40 | 4.97 |
| 900                         | 16.497                    | 0.454 | 18.05| 5.421       | 7.654          | 5.442  | 225.80 | 6.29 |
| 950                         | 16.517                    | 0.358 | 22.88| 5.421       | 7.671          | 5.409  | 224.93 | 6.51 |
| 1000                        | 16.538                    | 0.298 | 27.50| 5.418       | 7.665          | 5.402  | 224.33 | 7.21 |

\[ D = \frac{K\lambda}{\beta \cos \theta} \]  

Where \( D \) is the crystallite size, \( K \) is the Scherrer constant, \( \lambda \) is the x-ray radiation wavelength (Cu Kα; \( \lambda = 0.15406 \) Å), \( \theta \) is the Bragg angle, and \( \beta \) is the full width at half maxima. By fitting the Scherrer formula with equation (2), \( D \) can be obtained.

\[ D = \frac{0.89 \times 0.15406}{(\beta - 0.09/180) \times \pi \times \cos \theta} \]  

It is obvious that \( \beta \) and \( \theta \) for samples NSCM decreased with the raising of calcination temperatures while the particle sizes increased. Moreover, the lattice parameters of NSCM after calcination at 800, 850, 900, 950 and 1000 °C samples at room temperature extracted from Rietveld refinement of XRD data are included in table 1 [29]. These data were in good accordance with the XRD patterns, and they showed the low reliability factors (R).

Energy-dispersive x-ray spectroscopy (EDS) in TEM was used to analyze atomic resolution chemical mapping in the NSCM material as shown in figures 2(a)–(e). Element mapping images (figures 2(b)–(e)) and EDX curves (figure 2(f)) confirmed that the elements of Nd, Sr, Co, Mn in the as-prepared composite were evenly distributed. The microscopic details of the sample were determined by HRTEM. As depicted in figure 2(g), the microstructure of NSCM was cuboid varying in size. The distance of diffraction fringe was 2.749 Å as shown in HRTEM images of NSCM. The result conform to [200] planes. The indexed in orthorhombic lattice of the reflections in FTFs (fast Fourier transforms) can be judged from XRD patterns. The intensity streaks along [112] and [200] planes in (120) diffraction pattern could be found, which can be attributed to ordering of oxygen vacancies [31], or as the overlap of stacking derived fault scattering for B-site cations (Co(II) and Mn(III)) ordering [32, 33]. Generally, in perovskite structure oxides, B-site ionic ordering is based on the differences between charge and size of the two cations [24]. Thus, it has a bearing on the presence of Co^{2+}/Co^{3+} and Mn^{2+}/Mn^{3+} couples in NSCM.

Symmetrical cell NSCM-SDC/SDC/NSCM-SDC was used to assess the effects of the cathode material in the electrocatalytic activity. The symmetrical cell was operating at 450 °C–700 °C in air, and EIS was used to determine ASRs.

In the Impedance spectrum, there are two parts in terms of the frequency occupying low and high: Ohmic resistance (\( R_{\text{ohm}} \)) and cathode polarization resistance (\( R_p \)). \( R_{\text{ohm}} \) is the arc at high frequency which is considered from the connector resistances, electrolyte, the wire resistances, and other contactor’s resistances. \( R_p \) is the arc at an intermediate-to-low frequency, which has a bearing on the electrode process [34–36]. Figure 3(a) gives the curve of the relationship between \( R_p \) and temperature when the cell NSCM-SDC/SDC/NSCM-SDC was operating at different temperatures. With the temperature rising, semicircles formed by an arc intersecting a real axis associated with the cathode polarization process were obviously decreased; it was illustrated that \( R_p \) decreased with increasing of operating temperatures. According to the change of polarization resistance with temperature, it can be seen that the polarization resistance decreases with the increase of temperature, indicating that the higher the temperature is, the greater the surface exchange coefficient and volume phase diffusion coefficient are. In other words, the higher the temperature is, the faster the oxygen ion migration will be. \( R_p \) was 0.023 Ω · cm² at 700 °C. The result is lower than the value (0.18 Ω · cm²) at the same temperature reported from [37].

Commercial NiO was used as the anode material, and SDC with thickness about 19.6 μm was used as the electrolyte. NiO-SDC/SDC/NSCM-SDC (single cell with electrolyte for support) was fabricated to assess the electrochemical properties of cathode material NSCM. Figure 4 gave a cross section SEM image of electrode-electrolyte-electrode.

I-V and I-P curves were obtained under the operating conditions (operating temperatures at 450 °C to 650 °C; anode gas was 100% H₂ and cathode gas was O₂). The electrochemical performance results were provided in figure 5. The maximum power densities were 417.30 mWcm⁻², 565.45 mWcm⁻² and 592.80 mWcm⁻² at 550
Figure 2. (a) and (g) TEM of Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$; (b)–(e) EDX elemental mappings of Nd, Sr, Co, Mn, (f) EDX spectrum of Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$; and (i) HRTEM images of Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$ along [120] plane and the corresponding optical Fourier transform (FT).

Figure 3. (a) ASR of Nd$_{0.5}$Sr$_{0.5}$Co$_{0.5}$Mn$_{0.5}$O$_{3-\delta}$ measured in air by AC impedance of symmetrical cell. (b) Electrochemical impedance spectroscopy (EIS) arcs of the symmetrical cells at 450 °C–700 °C. (c) EIS arcs were amplified at 650 °C and 700 °C.
°C, 600 °C and 650 °C, respectively. Corresponding maximum OCV values were 0.89 V, 0.85 V and 0.81 V. Hence, Nd0.5Sr0.5Co0.5Mn0.5O3-δ can be used as an air-electrode in IT-SOFC.

Conclusion

Nd0.5Sr0.5Co0.5Mn0.5O3-δ for IT-SOFC was successfully synthesized and investigated. A perfect single crystal structure (perovskite-type) was obtained at 1000 °C. Some streaks along [200] and [112] diffraction patterns were observed for the ordering of oxygen vacancies, or as the overlap of B-site cations (Co2+/Co3+ and Mn2+/Mn3+) with stacking fault-derived scattering of ordering. EIS of symmetrical cell elucidated that cathode polarization resistance was responsible for electrochemical degradation. The ARS was 0.023 Ω·cm² when operating temperature was 700 °C. Single cell was operated at 450 °C–650 °C with 100% H2 as anode gas and O2 as cathode gas, 592.80 mWcm⁻² was the maximum power density at 650 °C, and 0.89 V was the maximum OCV at 550 °C. Hence, Nd0.5Sr0.5Co0.5Mn0.5O3-δ can be used in IT-SOFC as an air-electrode.

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