

One step synthesis of $\text{Sr}_2\text{Fe}_{1.3}\text{Co}_{0.2}\text{Mo}_{0.5}\text{O}_{6-\delta}\text{-Gd}_{0.1}\text{Ce}_{0.9}\text{O}_{2-\delta}$ for symmetrical solid oxide fuel cells

Yanru Yang¹, Shishuai Li¹, Zhibin Yang^{1,z}, Yu Chen^{2,z}, Panpan Zhang¹, Yuhao Wang³, Fanglin Chen⁴, Suping Peng¹

¹ Research Center of Solid Oxide Fuel Cell, China University of Mining and Technology-Beijing, People's

Republic of China

² School of Environment and Energy, South China University of Technology, Guangzhou 510006, People's

Republic of China

³ Department of Mechanical and Aerospace Engineering, Hong Kong University of Science and Technology, Clear

Water Bay, Hong Kong SAR, People's Republic of China

⁴ Department of Mechanical Engineering, University of South Carolina, Columbia, South Carolina 29208, United

States of America

Abstract

Homogenous distribution of individual phases in the composite electrode is critical to the overall electrode performance. In this work, we report a one-step synthesis method to fabricate $\text{Sr}_2\text{Fe}_{1.3}\text{Co}_{0.2}\text{Mo}_{0.5}\text{O}_{6-\delta}\text{-Gd}_{0.1}\text{Ce}_{0.9}\text{O}_{2-\delta}$ (SFCM-GDC) composite electrode material for symmetrical solid oxide fuel cells (SSOFCs). X-ray diffraction analysis reveals that the characteristic peaks of the perovskite and fluorite phases are obtained in the SFCM-GDC powder via the one-step synthesis method without any observable impurities. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) indicate a better phase distribution of the composite electrode of SFCM-GDC via one-step synthesis than that via a mechanically mixing process. As a result, the area specific resistance (ASR) of SFCM-GDC electrode is effectively decreased to $0.036 \Omega \text{ cm}^2$ and $0.047 \Omega \text{ cm}^2$ at 850°C in air and hydrogen, respectively, compared to that of $0.041 \Omega \text{ cm}^2$ and $0.074 \Omega \text{ cm}^2$ of the mechanically mixed SFCM and GDC (SFCM+GDC). Symmetrical solid oxide fuel cells using the one-step synthesis method with a cell configuration of SFCM-GDC||LSGM||SFCM-GDC are successfully fabricated and evaluated using wet H_2 (3% H_2O) as fuel. The cell maximum power density can reach 0.986 W cm^{-2} , higher than that with mechanically mixed electrode of 0.894 W cm^{-2} at 800°C . This work shows that one step synthesis method is an effective way to fabricate composite electrode

with enhanced electrode performance.

Key words: Solid oxide fuel cells; One-step synthesis; Composite electrode

1. Introduction

Solid oxide fuel cells (SOFCs) can generate electricity by efficiently converting chemical energy in the fuel without combustion [1-3]. NiO-YSZ (yttria-stabilized zirconia) electrode has been widely applied as anode material because of its high electrochemical activity for fuel oxidation and high electrical conductivity [4, 5]. However, the redox instability of Ni-based cermet anode is still a major problem to limit the SOFC commercialization [6, 7]. One of the reasons for the performance degradation has been attributed to the microstructural changes of the anode during redox cycles [8]. Consequently, redox stable electrode, especially those which can function as both anode and cathode, might be a better choice. In fact, symmetrical solid oxide fuel cells (SSOFCs) using the same material as both anode and cathode have attracted considerable attention in the past decade [9-11]. Noticeably, the electrode fabrication process would be simpler due to the reduction of the number of components for the SSOFCs, and the similar electrolyte–electrodes interfaces [12].

Early on, $\text{La}_{0.75}\text{Sr}_{0.25}\text{Cr}_{0.5}\text{Mn}_{0.5}\text{O}_{3-\delta}$ (LSCM) was used as cathode and anode simultaneously by J.C. Ruiz-Morales [13]. However, the electrode performance is relatively low. Later on, $\text{Pr}_{0.7}\text{Sr}_{0.3}\text{Cr}_{1-y}\text{Mn}_y\text{O}_{3-\delta}$ and $\text{Sr}_2\text{Fe}_{1.5}\text{Mo}_{0.5}\text{O}_{6-\delta}$ (SFM) have been evaluated as electrode material for SSOFCs [14, 15]. However, for most of these materials, although the electrical conductivity and stability are good enough to meet the SOFC electrode requirement, their catalytic activity is still insufficient compared to the state-of-the-art electrode materials such as Ni-YSZ. Precious metal materials, such as Pt, Pd, and Au, or non-precious metal alloy catalyst of Co-Ni-Mo have been integrated to improve the catalytic activity [16], which will undoubtedly increase the complexity of fabrication processes. Therefore, it is of great significance to explore new electrode materials for SSOFCs.

In our prior work, high performance $\text{La}_{0.4}\text{Sr}_{0.6}\text{Co}_{0.2}\text{Fe}_{0.7}\text{Nb}_{0.1}\text{O}_{3-\delta}$ (LSCFN) and $\text{Pr}_{0.4}\text{Sr}_{0.6}\text{Co}_{0.2}\text{Fe}_{0.7}\text{Nb}_{0.1}\text{O}_{3-\delta}$

(PSCFN) as both cathode and anode were developed [17, 18]. Mixing the PSCFN powder with $\text{Gd}_{0.2}\text{Ce}_{0.8}\text{O}_{2-\delta}$ (GDC) will further improve the electrode performance. For example, the area specific resistance (ASR) of PSCFN-GDC composite electrodes were $0.044\ \Omega\text{cm}^2$ (as cathode) and $0.309\ \Omega\text{cm}^2$ (as anode) at $800\ ^\circ\text{C}$, respectively, indicating that the composite electrodes demonstrated excellent electrochemical performances for both oxygen reductions and fuel oxidation reactions [19]. In recent work, Co-substituted $\text{Sr}_2\text{Fe}_{1.5}\text{Mo}_{0.5}\text{O}_{6-\delta}$ (SFCM) as anode materials for solid oxide fuel cells have achieved high performance via nanoparticle exsolution [20]. The effects of Co and Mo doping on the properties of $\text{SrFeO}_{3-\delta}$ perovskite as cathode in SOFCs have also been analyzed [21]. Therefore, it is reasonable to conclude that SFCM might be a good candidate electrode material for SSOFCs.

On the other hand, ceria-based oxide has been typically used to further increase the ionic conductivity and extend surface reaction sites in SOFC anodes. Ceria oxide may also be able to remove the coking when SOFCs are fueled by hydrocarbons due to its unique catalytic activity. For instance, a composite of $\text{Sr}_2\text{FeMo}_{0.65}\text{Ni}_{0.35}\text{O}_{6-\delta}$ (SFMN)-GDC as a promising anode material shows a good performance when operated in carbon-containing fuels such as syngas or methane [22]. Nonetheless, the distribution of individual phases in the composite electrode is also critical to the cell performance. The electrochemical performance of cermet anodes may be improved by increasing the number of triple phase boundaries, i.e., the electrochemical reaction sites. Therefore, development of novel chemical syntheses routes to produce homogeneous nano-composite powders, and further optimization of the cermet microstructure are highly desirable [23].

One-step synthesis method is simple and versatile, and has a wide range of applications in the fabrication of ceramic ionic transporting membranes [24], which can offer more uniform mixing of multi-phases than the traditional methods. In addition, it can provide more catalytic reaction sites and extend TPBs in the SOFC electrode [25]. Here, we fabricated the composite electrode of SFCM-GDC via one-step synthesis, and further evaluated its electrochemical performance as electrode of SSOFCs. Our results show that composite powder obtained via the one-

step synthesis has a more homogeneous phase distribution. More importantly, the performance is improved significantly by one step synthesis method.

2. Experimental procedure

2.1 Fabrication of powders and cells

Two different routes of the so-called “one-step synthesis” and “two-step synthesis” were used to prepare $\text{Sr}_2\text{Fe}_{1.3}\text{Co}_{0.2}\text{Mo}_{0.5}\text{O}_{6-\delta}$ (SFCM) and $\text{Gd}_{0.1}\text{Ce}_{0.9}\text{O}_{2-\delta}$ (GDC) composite electrode in this study.

The composite electrode via one step synthesis was denoted as SFCM-GDC. According to the mass ratio of SFCM and GDC (1:1), stoichiometrically amounts of SrCO_3 , $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Fe_2O_3 , $(\text{NH}_3)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ were used as precursors for solid state reaction. The precursors were mixed by ball milling for 48 h in ethanol, dried at 150 °C, and calcined in air at 1100 °C for 5 h.

The composite electrode via two-steps synthesis was denoted as SFCM+GDC. Firstly, SFCM is prepared by a conventional solid-state reaction; the details about the fabrication can be found in our prior work [20]. SrCO_3 , Fe_2O_3 , $(\text{NH}_3)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were mixed at a stoichiometric ratio. The mixtures were then ball-milled and fired at 1100 °C in air to obtain the perovskite phase. Similarly, the GDC powder was also prepared by solid-state reaction method. $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ were mixed at the stoichiometric ratio. The mixtures were then ball-milled and calcined at 800 °C for 15 h. Finally, the SFCM and GDC powder were mechanically mixed with a mass ratio of 1:1.

Both the symmetrical half cells and full cells in this work are electrolyte ($\text{La}_{0.8}\text{Sr}_{0.2}\text{Ga}_{0.83}\text{Mg}_{0.17}\text{O}_{3-\delta}$, LSGM) supported cells. LSGM powders are synthesized by solid state reaction method. Dense LSGM pellets with 18 mm in diameter and 240 μm in thickness are fabricated by dry-pressing and sintered at 1450 °C, as previously reported [26]. The two components of composite electrode powders were mixed with glue (V-737) to form electrode ink. The

electrode ink was finally applied on both sides on the LSGM electrolyte by a screen-printing method and fired at 1100 °C for 2 h to form symmetrical half cells and full cells. The effective area of both electrodes is 0.5 cm² with a thickness of approximately 40 μm.

2.2 Materials characterization and electrochemical performance test

Phase identification of the synthesized composite powder was analyzed by X-ray diffraction (XRD) (PANalytical X'Pert PRO, Netherlands) with a scanning step of 0.02° in the 2θ range of 10-90°. The size and morphology of particles were characterized using scanning electron microscopy (SEM, FEI Quanta 200).

The single cells were sealed on the alumina tube with a ceramic bond 552 and were firstly reduced at 850 °C for 2 h in H₂. The electrochemical performance was typically evaluated in the temperature range of 700 to 850 °C, using H₂ humidified with 3% H₂O as fuel (with a flow rate of 50 ml min⁻¹), and ambient air as oxidant.

3. Results and discussion

3.1 XRD analysis

Shown in Fig. 1 are the XRD patterns of the synthesized SFCM and GDC composite powders (one-step or two step) calcined at 1100 °C for 5 h in air, as well as individual SFCM and GDC powders. It can be seen that the characteristic fluorite structure peaks and perovskite structure peaks are distinctly detected in the SFCM-GDC powder, which is similarly observed from the pattern of SFCM+GDC powder. All the diffraction peaks are matched well with SFCM and GDC. No impurity phase peaks were found, indicating that SFCM and GDC can be synthesized in one step. Furthermore, it is also suggested that these two materials are chemically compatible, with no other undesirable reactions.

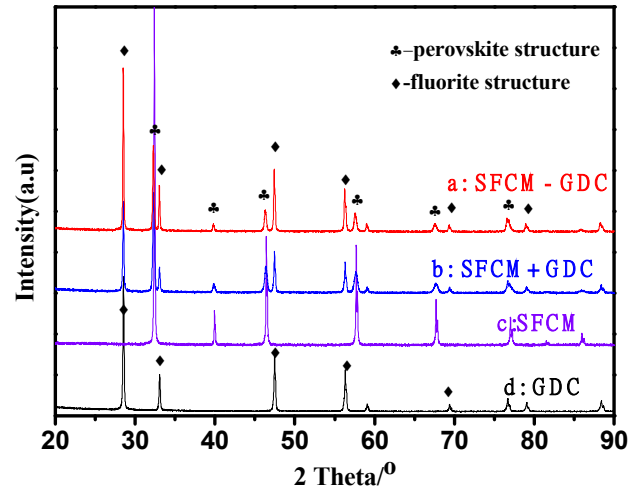


Fig. 1. X-ray diffraction patterns of (a) one step synthesized SFCM-GDC, (b) two step synthesized SFCM+GDC, (c) SFCM and (d) GDC powders.

3.2 Microstructure characterization

Shown in Fig 2 are the cross-sectional scanning electron microscopic (SEM) images of the full cells with SFCM-GDC (Fig. 2a) and SFM+GDC (Fig. 2b) electrodes. It can be seen that the adhesion between porous electrodes and dense electrolytes is very well. The thickness of the electrode was about 40 μm and the thickness of the LSGM electrolyte films was about 240 μm .

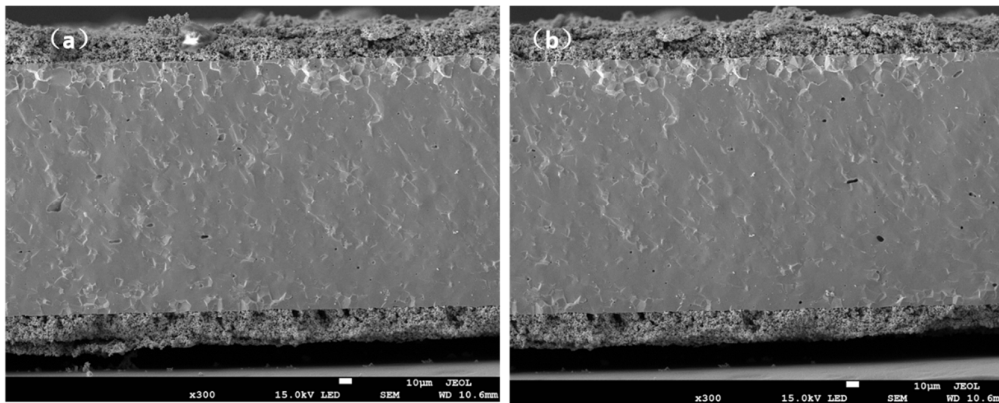


Fig. 2. The cross-sectional scanning electron microscopic (SEM) image of the full cells (a: SFCM-

GDC/LSGM/SFCM-GDC, b: SFCM+GDC/LSGM/ SFCM+GDC)

Shown in Fig.3. are the morphology and the corresponding EDS mappings (Sr- and Ce-) of the surface of the SFCM-GDC and SFCM+GDC electrode. Significant difference of the particle size and electrode morphology can be observed; much finer particles and more uniformly dispersed microstructure of SFCM-GDC electrode are obtained using the one-step synthesis method compared with the two-step synthesis one. Obvious aggregations were found in the two-step synthesis one.

Further, the backscatter images of Fig.3 (c, d) clearly demonstrated differences between these two electrodes in morphology, particle size and particles distribution. The dark particles are SFCM while the bright ones are GDC. It is shown that much smaller particle size of both GDC and SFCM, and more uniformly dispersed electrode microstructure are obtained using the one step synthesis method. These will potentially increase the active sites for electrochemical reactions when used as electrodes for SSOFCs.

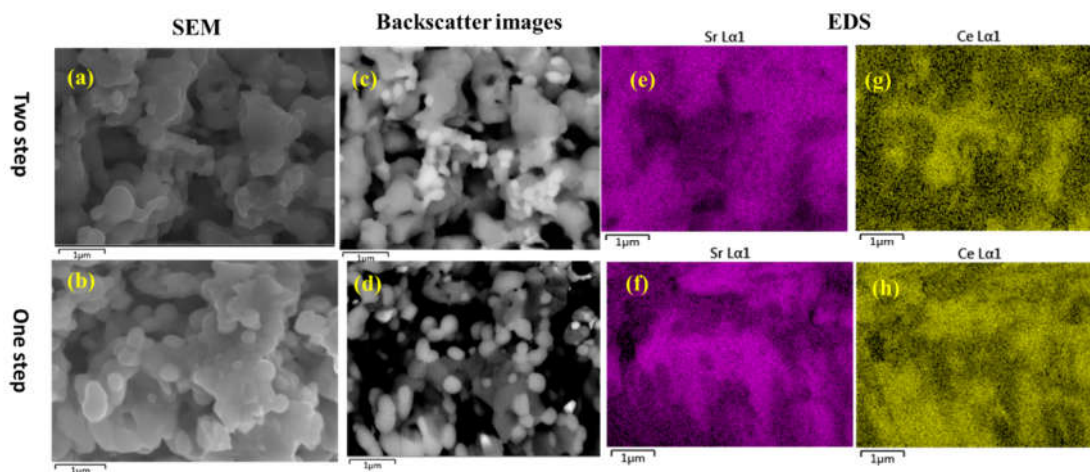


Fig.3. SEM secondary electron images, backscatter electron images and the corresponding EDS mapping of the surface of the as-prepared SFCM+GDC (two-step, a, c, e, g) and SFCM-GDC (one-step, b, d, f, h) electrode.

3.3 Electrochemical characterization of the SFCM and GDC composite electrode

Shown in Fig. 4 are the impedance spectra of SFCM, SFCM-GDC and SFCM+GDC electrode in symmetrical

half-cells when tested in air (a, b) or hydrogen (c, d). The ohmic resistances were subtracted from the total electrode impedance for clear comparison of electrode area specific resistance (ASR, or R_p). As expected, the composite SFCM-GDC and SFCM+GDC electrode showed reduced R_p values compared to the SFCM electrode ($0.052 \Omega \text{ cm}^2$ in air at 850°C). Therefore, the addition of GDC can promote the electrode reactions. In particular, the SFCM-GDC electrode in air showed the promising R_p values of $0.036 \Omega \text{ cm}^2$, $0.05 \Omega \text{ cm}^2$ and $0.073 \Omega \text{ cm}^2$ at 850°C , 800°C and 750°C , which are much lower than that of the $\text{Pr}_2\text{NiO}_4\text{-Pr}_{0.2}\text{Ce}_{0.8}\text{O}_{1.9}$ composite cathode ($0.09 \Omega \text{ cm}^2$ at 800°C)[27] and $\text{La}_{0.8}\text{Sr}_{1.2}\text{CoO}_{4+\delta}\text{-Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$ composite cathode ($0.21 \Omega \text{ cm}^2$ at 750°C)[28]. The significantly reduced R_p value for the SFCM-GDC electrode can be ascribed to its extended boundary length between the two phases, facilitating the electrode reactions at triple phase boundaries. Similar results were found when the electrodes were tested in hydrogen. All these results indicate that the SFCM-GDC via one step synthesis method could significantly decrease the R_p and further increase the fuel cell performance.

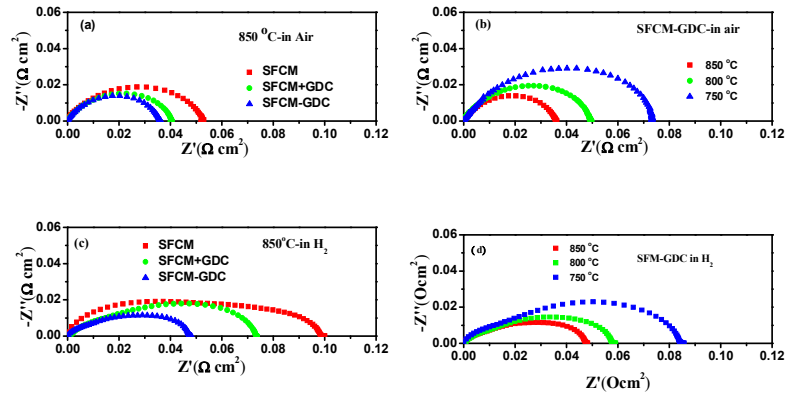


Fig.4. Impedance plots obtained for SFCM, SFCM-GDC and SFCM+GDC for symmetrical half-cells tested in air (a, b) and hydrogen (c, d)

Shown in Fig. 5(a, b) are the typical IV and IP curves of cells using SFCM, SFCM-GDC or SFCM+GDC as electrode when tested in wet H_2 at different temperatures. From Fig.5a, the maximum power density of the cells with the SFCM-GDC electrode is 0.986 W cm^{-2} at 800°C , which is higher than that of the cells with SFCM+GDC electrode,

which is only 0.894 W cm^{-2} at 800°C . Since the cell configuration, compositions, and testing conditions are the same, the improved cell performance could be directly attributed to the lower electrode polarization resistance values of the SFCM-GDC electrode as shown in Fig. 4. Maximum power densities are 749 , 986 and 1305 mW cm^{-2} at 750°C , 800°C , 850°C respectively, and the polarization resistance values of the cell are only 0.27 , 0.35 , and $0.52 \Omega \text{ cm}^2$ at 850 , 800 and 750°C , respectively, as shown in Fig. 5(b, c), suggesting that SFCM-GDC composite electrode has good catalytic activity to both oxygen reduction and fuel oxidation.

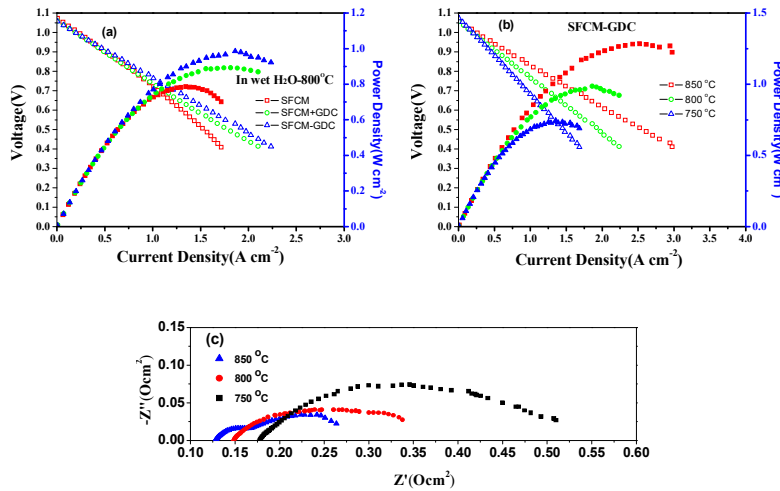


Fig. 5. (a) IV and IP curves of cells with electrode of SFCM, SFCM-GDC or SFCM+GDC for SSOFCs when using wet H₂ as fuel and ambient air as oxidant at 800°C ; (b,c) IV and IP curves and the polarization resistance value with the SFCM-GDC electrode at different temperatures

4. Conclusions

In this study, performance of symmetrical solid oxide fuel cells using one-step synthesis method to fabricate SFCM-GDC composite electrode was systematically evaluated. Compared with the SFCM electrode or mechanically mixed SFCM+GDC composite electrode, SFCM-GDC composite electrode fabricated from the one-step synthesis method showed significantly improved performance. More homogeneous microstructure and extended triple phase

boundary length may be the main reason to the electrode performance enhancement, while the addition of GDC also promoted the electrode reactions. This study shows that the SFCM-GDC composite electrode obtained from the one-step synthesis method is a promising electrode candidate for practical symmetrical solid oxide fuel cell application.

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