Symmetrical La$^{3+}$-doped Sr$_2$Fe$_{1.5}$Ni$_{0.1}$Mo$_{0.4}$O$_{6-\delta}$ Electrode Solid Oxide Fuel Cells for Pure CO$_2$ Electrolysis

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Abstract: Electrochemical reduction of the greenhouse gas CO$_2$ in solid oxide electrolysis cells (SOECs) has attracted much attention due to their high energy conversion efficiency and great potential for carbon cycling. Compared with the asymmetrical configuration, symmetrical SOECs with the same material as both the anode and cathode, can greatly simplify the fabrication process and reduce the complication associated with varied interfaces. Perovskite oxides La$_3$Sr$_2$Fe$_{1.5}$Ni$_{0.1}$Mo$_{0.4}$O$_{6-\delta}$ (L$_3$SFNM, $\delta=0.1, 0.2, 0.3$ and $0.4$) are prepared and evaluated as symmetrical electrodes in solid oxide electrolysis cells for electrochemical reduction of pure CO$_2$. The polarization resistances are 0.07 $\Omega \cdot \text{cm}^2$ in air and 0.63 $\Omega \cdot \text{cm}^2$ in 50% CO-50% CO$_2$ for La$_3$SFNM electrode at 800 °C. An electrolysis current density of 1.17 A/cm$^2$ under 800 °C at 1.5 V is achieved for the symmetrical SOECs in pure CO$_2$. Furthermore, the symmetrical cell demonstrates excellent stability during the preliminary 50-hour CO$_2$ electrolysis measurements.

Key words: symmetrical solid oxide electrolysis cells; perovskite; electrode materials

In the past seventy years, the concentration of CO$_2$ in atmosphere has increased by about 0.012%, which is the main cause of greenhouse effect inducing a series of problems like global warming and climate change$^{[1]}$. Capture and utilization of CO$_2$ is of great significance to sustainable development of human society. Nevertheless, high thermodynamic stability and kinetic inertness of CO$_2$ limit its conversion and applications$^{[2]}$.

Solid oxide electrolysis cells (SOECs) are one of the most promising technologies to convert CO$_2$ into high energy density fuels and value-added chemicals using the surplus renewable electricity, promoting CO$_2$ utilization and penetration of renewable electricity in the current energy regime as well$^{[3-4]}$.

Ni-based cermet are the commonly used cathodes in SOECs, but suffer from some limitations such as nickel oxidation in CO$_2$-rich atmosphere and carbon deposition in CO-rich atmosphere at high CO$_2$ conversions, which may prevent their practical applications$^{[5-6]}$. Due to their distinctive features such as brilliant redox stability and excellent resistance against coking formation and sulfur poisoning, mixed ionic-electronic conducting oxides have been widely investigated as alternative cathodes for CO$_2$ electrolysis. Furthermore, some mixed conducting oxides can be simultaneously used as both anodes and cathodes to obtain symmetrical SOECs, which greatly simplifies the fabrication process and reduces complications associated with the different electrode/electrolyte interfaces. Extensive efforts have been made to explore perovskite and double-perovskite electrodes in symmetrical SOECs, such as Sr$_2$Fe$_{1.5}$Mo$_{0.5}$O$_6$-$\delta$,$^{[7-8]}$ La$_{0.75}$Sr$_{0.25}$Cr$_{0.5}$Mn$_{0.5}$O$_3$,$^{[9]}$ La$_{0.5}$Sr$_{0.5}$TiO$_3$,$^{[10-11]}$ and La$_{0.4}$Sr$_{0.6}$Fe$_{0.8}$Ni$_{0.2}$O$_{3-\delta}$. The symmetrical electrodes should be stable with sufficient electrical conductivities (e.g., >1 $\text{S} \cdot \text{cm}^{-1}$) in both air and fuel environments, and have good chemical compatibility with the other cell components. L$_{40.6}$Ca$_{0.4}$Fe$_{0.8}$Ni$_{0.2}$O$_{3-\delta}$ (LCaFN) was employed as the electrodes for symmetrical SOECs, showing a self-recovery from the performance degradation due to formation of carbonates by air treatment$^{[13]}$. Sr$_3$Fe$_{1.8}$Mn$_{0.5}$O$_{6.4}$ (SFM), the symmetrical SOFC electrodes developed by Liu, et al. exhib-
ited high electrocatalytic activities for both CO\textsubscript{2} reduction and oxygen evolution reactions in symmetrical SOECs\textsuperscript{14}. Partial substitution in the A or B site is an effective strategy to optimize the catalytic activity of perovskite oxides. Ce\textsuperscript{4+} was introduced into the A site of La\textsubscript{0.7}Sr\textsubscript{0.3}Cr\textsubscript{0.5}Fe\textsubscript{0.5}O\textsubscript{3-δ} (LSCrF) to increase oxygen vacancies in the lattice by \textit{in situ} reduction under the operational conditions and thereby enhance the catalytic activities\textsuperscript{15}. In this work, La\textsuperscript{3+} doped Sr\textsubscript{2}Fe\textsubscript{1.5}Ni\textsubscript{0.5}Mo\textsubscript{0.4}O\textsubscript{6-δ} (L\textsubscript{2}SFNM) was investigated as the symmetrical SOEC electrodes for direct CO\textsubscript{2} electrolysis. The influence of the La\textsuperscript{3+} content on the oxygen vacancy concentration, electrical conductivity, and catalytic activity were analyzed. Electrochemical performance was evaluated on symmetrical "L\textsubscript{2}SFNM@LSGM|LSGM|L\textsubscript{2}SFNM@LSGM" cells for pure CO\textsubscript{2} electrolysis.

1 Experimental

L\textsubscript{2}SFNM powders were prepared using the Sol-Gel method. The precursor solutions were prepared as follows: stoichiometric amounts of the nitrate salts La(NO\textsubscript{3})\textsubscript{3}-6H\textsubscript{2}O, Sm(NO\textsubscript{3})\textsubscript{3}-6H\textsubscript{2}O, Sr(NO\textsubscript{3})\textsubscript{2}, Ba(NO\textsubscript{3})\textsubscript{2} and Co(NO\textsubscript{3})\textsubscript{3}-6H\textsubscript{2}O were firstly dissolved in distilled water, and then citric acid was added with the molar ratio of the citric acid to the total metal ions at 1.2:1. The precursor solution was subsequently heated at 80 °C until a gel was obtained, followed by drying at 200 °C for 5 h and calcination at 1000 °C in air for 5 h to obtain pure L\textsubscript{2}SFNM powders.

The tri-layer structure of “porous | dense | porous” LSGM (La\textsubscript{0.9}Sr\textsubscript{0.1}Ga\textsubscript{0.8}Mg\textsubscript{0.2}O\textsubscript{3-δ}) structure was prepared by laminating three tape-cast green tapes at 75 °C and 20 MPa, with 40wt% rice starch and graphite used as the pore-forming material in the porous layers. The laminated layers were co-fired at 1380 °C to produce the final ceramic structures. L\textsubscript{2}SFNM catalysts were added into the porous LSGM scaffolds by impregnating the precursor solution with the total concentration of metal ions at 1 mol\textperendash;L\textperendash;\textsuperscript{1}, followed by calcination at 850 °C for 4 h. The impregnation and calcination cycle was repeated for 16 times so as to achieve an L\textsubscript{2}SFNM loading of ~25wt% relative to the LSGM scaffolds.

The X-ray diffraction (XRD) pattern of as-synthesized powders was examined at room temperature on an Rigaku D/Max 2100 Powder X-ray Diffractometer with a monochromatic Cu K\textalpha\ and the diffraction data were recorded in the range 2θ=20°-80° with scan rate of 5°/min\textsuperscript{1}. The cell structure was examined using the scanning electron microscopy (SEM) in a FEI Magellan 400 microscope. The conductivities of L\textsubscript{2}SFNM were tested in air and 50% CO-50% CO\textsubscript{2} using the DC four-probe method. For fuel cell measurements, silver ink (DAD87, Shanghai Institute of Synthetic Resin) and silver wires was applied on the anode and cathode electrode surface as the current collectors. Impedance measurements were performed on the symmetrical fuel cells in a homogeneous environment of air or 50% CO-50% CO\textsubscript{2}. The electrolys performance of the symmetrical fuel cells was evaluated using IM6 Electrochemical Workstation (ZAHNER, Germany), with pure CO\textsubscript{2} fed to the cathodes at 50 sccm and ambient air in the anodes.

2 Results and discussion

The XRD patterns of La\textsubscript{2}Sr\textsubscript{2-x}Fe\textsubscript{1.5}Ni\textsubscript{0.5}Mo\textsubscript{0.4}O\textsubscript{6-δ} oxides at room temperature are shown in Fig. 1(a). All samples showed a pure cubic perovskite structure without any extra peaks of impurities observed. Fig. 1(b) shows that the (110) peak shifts to a higher scattering angle at x=0.1, which can be explained by smaller ionic radius for La\textsuperscript{3+} than that for Sr\textsuperscript{2+} (0.136 nm vs. 0.144 nm). With the La\textsuperscript{3+} content further increasing, the diffraction peak shifted to lower scattering angles. Prior reports showed that the cell expansion was inhibited due to the steric effects associated with anti-site defects\textsuperscript{16-17}. This indicates the existence of a disordered crystal structure in L\textsubscript{2}SFNM double perovskites.

Fig. 2 compares the thermogravimetric curves of L\textsubscript{2}SFNM powders in air, showing a loss of 1%-1.5% in the powder weight due to release of the lattice oxygen with the temperature increasing from 30 to 900 °C. Note that the largest weight loss was observed for L\textsubscript{2}SFNM, indicative of the most abundant presence of oxygen vacancies at high temperatures. Fig. 3(a-b) show the conductivities of L\textsubscript{2}SFNM measured in air or 50% CO-50% CO\textsubscript{2} at 650-800 °C. Due to their p-type
conducting nature, the conductivities were much higher in air than those in 50% CO-50% CO₂ (17.3-38.1 vs. 2.7-5.5 S·cm⁻¹). Fig. 3 also shows that L₀.₃SFNM had the highest conductivities among four samples at all temperatures. For x≤0.3, partial substitution of La³⁺ for Sr²⁺ increased the concentration of anti-site defects and the electron density of Fermi level. It has been reported that the half metallic properties of SFM was quite sensitive to the anti-site defect concentration as it altered the Fe-O-Mo bonding network and the electronic hopping pathway[18]. Due to a decrease in the crystal symmetry and the cation order, the double exchange interaction between Fe-O-Mo may be inhibited at x≥0.4, leading to decreased conductivities[19-21].

![Fig. 2 TGA analysis of L₀.₃SFNM in air](image)

**Fig. 2 TGA analysis of L₀.₃SFNM in air**

![Fig. 3 Electricity conductivities of L₀.₃SFNM at 650-800 °C in (a) the air and (b) the 50% CO-50% CO₂](image)

**Fig. 3 Electricity conductivities of L₀.₃SFNM at 650-800 °C in (a) the air and (b) the 50% CO-50% CO₂**

In order to study the effect of different La³⁺ contents on the activities of L₀.xSFNM oxides toward oxygen evolution and CO₂ reduction reactions, electrochemical impedance measurements were performed on electrolyte-supported symmetrical cells with impregnated L₀.xSFNM electrodes, i.e., L₀.xSFNM@LSGM|LSGM|L₀.xSFNM@LSGM (Fig. 4(a)). The dense LSGM electrolytes were 210 μm in thickness, whereas the porous electrodes were around 20 μm in thickness. Fig. 4(b) shows excellent interfacial bonding between dense electrolytes and the porous scaffolds that was conducive to minimizing the contact resistance. As shown in Fig. 4(c), nano-scale L₀.xSFNM catalysts were deposited onto the internal surfaces of porous LSGM scaffolds to form a well-interconnected network to facilitate electronic conduction and thus enhance their catalytic activities.

![Fig. 4 (a) Cross-sectional SEM image of the tri-layer symmetrical structure of “porous/dense/porous” LSGM and (b) high magnification view of symmetrical cell; (c) High-magnification view of impregnated L₀.₃SFNM catalyst](image)

**Fig. 4 (a) Cross-sectional SEM image of the tri-layer symmetrical structure of “porous/dense/porous” LSGM and (b) high magnification view of symmetrical cell; (c) High-magnification view of impregnated L₀.₃SFNM catalyst**

Electrochemical impedance measurements were performed for symmetrical cells in the homogeneous environment of dry air or 50% CO-50% CO₂, with the typical Nyquist plots of impedance data compared in Fig. 5. Note that the electrolyte resistances were subtracted from the cell impedance, and the polarization resistances were divided by two due to the symmetrical configurations. Fig. 5(a) shows that the anode polarization resistances (R₊) at 800 °C were 0.12, 0.09, 0.07 and 0.10 Ω·cm² for L₀.₁SFNM, L₀.₂SFNM, L₀.₃SFNM and L₀.₄SFNM in dry air, respectively. L₀.₃SFNM exhibited much smaller R₊ values compared with some previously reported air electrodes, such as Sr₂FeMoO₆ (0.1 Ω·cm² at 850 °C)[22], Sr₂Fe₁₋ₓNi₀.₁Mo₀.₉O₆₋ₓ (0.22 Ω·cm² at 750 °C)[23], P₀.₇Sr₀.₃Fe₀.₇Ni₀.₁Mo₀.₁O₄₋ₓ (0.4 Ω·cm² at 800 °C)[24] and La₀.₆Sr₀.₄Fe₀.₃Mn₀.₁O₃₋ₓ-GDC (0.24 Ω·cm² at 800 °C)[25]. Distribution of relaxation time (DRT) analyses were conducted on impedance data so as to identify the number of polarization processes involved in the oxygen evolution reaction. With spectra transferred from the frequency into time domain, the overlapped polarization processes can be clearly distinguished. Fig. 5(b) shows that all DRT curves had three distinctive peaks, corresponding to three elementary reaction processes in the oxygen evolution reaction. The low-frequency peaks (1-10 Hz) were usually assigned to gas diffusion in the porous electrodes, while the high frequency peaks (300-2000 Hz) were related to the charge transfer process across the electrode-electrolyte interfaces. R_LF and R_HI were almost independent of the La³⁺ content. Note that the intermediate frequency peaks (50-200 Hz) dropped pronouncedly in the order of L₀.₁SFNM > L₀.₄SFNM > L₀.₃SFNM > L₀.₅SFNM. Given that the intermediate
frequency peaks were usually associated with the bulk transport of oxygen surface exchange and O\(^{2-}\) bulk diffusion\(^{[26]}\), it can be concluded that L\(_{0.3}\)SFNM had the highest surface oxygen exchange rate. The highest activity of L\(_{0.3}\)SFNM toward oxygen evolution was also consistent with the smallest activation energy of \(R_{P_A}\) among the investigated L\(_x\)SFNM (Fig. 6(a)). It should also be pointed out that the intermediate frequency peaks were much larger than the low- or high-frequency peaks for all L\(_x\)SFNM samples, indicating that surface oxygen exchange is the rate-limiting step in oxygen evolution reaction.

![Fig. 5 Nyquist plots of impedance data measured with L\(_x\)SFNM at 800 °C in (a) air and (c) 50% CO-50% CO\(_2\); (b, d) DRT curves of impedance data shown in (a, c)](image)

Fig. 5(c) shows an order of the cathode polarization resistances (\(R_{PC}\)) - L\(_{0.3}\)SFNM (0.62 Ω\(\cdot\)cm\(^2\))<L\(_{0.2}\)SFNM (0.86 Ω\(\cdot\)cm\(^2\))<L\(_{0.4}\)SFNM (0.98 Ω\(\cdot\)cm\(^2\))<L\(_{0}\)SFNM (1.24 Ω\(\cdot\)cm\(^2\)) measured in 50% CO-50% CO\(_2\), demonstrating that L\(_{0.3}\)SFNM had the highest activities toward CO\(_2\) reduction reactions. Fig. 5(d) shows the DRT analysis results of impedance data in Fig. 5(c), also showing three well-distinguished peaks. The low-, intermediate- and high-frequency peaks were associated with dissociative adsorption of CO\(_2\) molecules on the surface, surface diffusion and the charge transfer process, respectively. \(R_{HF}\) and \(R_{IF}\) remained independent of the L\(_{3+}\) content, while \(R_{LF}\) showed the lowest value at \(x=0.3\). Much higher \(R_{LF}\) values than \(R_{HF}\) and \(R_{IF}\) indicated that the overall CO\(_2\) electro-reduction was limited by dissociative adsorption of CO\(_2\) on the surface of LSFNM. Moreover, the cathode polarization resistances (\(R_{PC}\)) was much larger than \(R_{PA}\), demonstrating that the electrochemical performance of the CO\(_2\) electrolysis cells mainly depended on the catalytic activities of cathodes toward CO\(_2\) reduction reactions. Fig. 6(b) shows that the activation energies of \(R_{PC}\) were about 0.83-0.89 eV.

![Fig. 6 Measured polarization resistances versus the temperature reciprocal in (a) air and (b) 50% CO-50% CO\(_2\)](image)

The electrochemical performance of L\(_{0.3}\)SFNM electrode for pure CO\(_2\) electrolysis was further evaluated using the LSGM-electrolyte supported SOECs at 650-800 °C, with pure CO\(_2\) in the cathodes at 50 sccm and ambient air in the anodes. Fig. 7(a) shows the curves of measured voltages as a function of current densities. At the applied voltage of 1.5 V, the electrolysis current densities increased from 0.4 A\(\cdot\)cm\(^{-2}\) at 650 °C to 1.17 A\(\cdot\)cm\(^{-2}\) at 800 °C. These values were competitive when compared with previously reported results listed in Table 1. Fig. 7(b) compares the corresponding EIS spectra measured at 1.5 V. The ohmic resistance and polarization resistance all increased obviously with reducing the cell operating temperature. The interfacial polarization resistances were 0.13 Ω\(\cdot\)cm\(^2\) at 650 °C and 0.52
\( \text{cm}^2 \) at 800 °C. Fig. 7(c) summarizes the correlation between the doping amount of La\(^{3+} \) ions and the electrolysis current density at 1.5 V, showing that L\(_{0.3}\)SFNM yielded the highest performance at all measurement temperatures. Fig. 7(d) shows the preliminary short-term stability of symmetrical L\(_{0.3}\)SFNM electrode cells measured at 800 °C and 1.3 V in pure CO\(_2\). The current density slightly dropped from 0.68 to 0.64 A·cm\(^{-2} \) during the first 2 h, and then remained stable, indicating good stability of the nano-scale L\(_{0.3}\)SFNM electrodes.

![Graph showing the current density and impedance spectra](image)

Fig. 7 (a) I-V curves and (b) impedance spectra measured at the 1.5 V of a single electrolyte-supported electrolysis cell with L\(_{0.3}\)SFNM at different temperatures with inset in (b) showing corresponding enlarged spectrum of the cell with L\(_{0.3}\)SFNM at 800 °C; (c) Current densities with varied La, SFNM anodes at 1.5 V under different temperatures; (d) Short-term stability test of the symmetrical SOEC with L\(_{0.3}\)SFNM electrode, operating under 800 °C at an applied voltage of 1.3 V

### Table 1

| Electrode | Electrolyte | Performance \(/\text{A·cm}^{-2} \) \(2\) | Ref. |
|-----------|-------------|----------------------------------|-----|
| L\(_{0.3}\)Sr\(_0.7\)Fe\(_{0.7}\)Ti\(_{0.3}\)O\(_3\) | YSZ | 0.52 | [27] |
| L\(_{0.6}\)Sr\(_0.4\)Fe\(_0.9\)Mn\(_{0.1}\)O\(_{3-\delta}\)-GDC | YSZ | 1.107 | [25] |
| L\(_{0.6}\)Sr\(_0.4\)Fe\(_0.8\)Ni\(_{0.2}\)O\(_{3-\delta}\)-GDC | YSZ | 1.03 | [28] |
| L\(_{0.4}\)Sr\(_0.6\)Co\(_{0.3}\)Fe\(_{0.7}\)Nb\(_{0.2}\)O\(_{3-\delta}\) | YSZ | 0.442 | [29] |
| L\(_{0.3}\)Co\(_{0.6}\)Fe\(_0.4\)Ni\(_{0.2}\)O\(_{3-\delta}\)-GDC | YSZ | 0.78 | [13] |
| L\(_{0.75}\)Sr\(_{0.25}\)Ti\(_{0.3}\)Mn\(_{0.5}\)O\(_{3-\delta}\) | YSZ | 0.09 | [30] |
| L\(_{0.3}\)Sr\(_0.7\)Cr\(_{0.3}\)Fe\(_{0.7}\)O\(_{3-\delta}\) | YSZ | 0.32 | [31] |
| (PrBa)\(_{0.95}\)(Fe\(_0.9\)Mo\(_{0.1}\))\(_2\)O\(_{3+\delta}\) | LSGM | 0.51 (1.3V) | [32] |
| L\(_{0.3}\)Sr\(_0.7\)Fe\(_{0.3}\)Ni\(_{0.1}\)Mo\(_{0.4}\)O\(_{6-\delta}\) | LSGM | 1.17 | This work |

YSZ=Zr\(_{0.84}\)Y\(_{0.16}\)O\(_{1.92}\); GDC=Gd\(_{0.9}\)Ce\(_{0.1}\)O\(_{2-\delta}\); LSGM=L\(_{0.3}\)Sr\(_0.7\)Ga\(_{0.3}\)Mg\(_{0.2}\)O\(_{3-\delta}\)

### 3 Conclusions

In summary, a series of perovskite oxides La\(_{x}\)Sr\(_{2-x}\)Ti\(_{1.5}\)Ni\(_{0.1}\)Mo\(_{0.4}\)O\(_{6-\delta}\) (L\(_x\)SFNM, \(x=0.1, 0.2, 0.3 \) and 0.4) have been synthesized and evaluated as symmetrical electrodes for solid oxide electrolysis cells. Impedance measurements show that the electrode performance strongly depended upon the La\(^{3+} \) doping content in L\(_{0.3}\)SFNM. Both of the highest activities for oxygen evolution and CO\(_2\) reduction observed both at \(x=0.3\). LSGM electrolyte-supported SOECs produce an electrolysis current density of 1.17 A·cm\(^{-2} \) at 800 °C and 1.5 V in pure CO\(_2\), and good stability was observed during a preliminary 50-hour measurement. These results demonstrate that L\(_{0.3}\)SFNM could be a promising alternative as symmetrical electrodes in SOECs for pure CO\(_2\) electrolysis.
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La$_{0.3}$Sr$_{0.7}$Fe$_{0.7}$Cr$_{0.3}$O$_{3}$-δ 为电极的电解池表现出最高的电化学性能，800 ℃下，在空气中的极化电阻为 0.07 Ω cm$^2$，在 50% CO-50% CO$_2$ 中的极化电阻为 0.62 Ω cm$^2$。单电池 $La_{0.3}$SFNM@LSGM|LSGM|La$_{0.3}$SFNM@LSGM 在 800 ℃和 1.5 V 电压下的电解电流密度为 1.17 A·cm$^{-2}$，在初的 50 h CO$_2$ 短期电解测试中表现出优异的稳定性，是一种理想的对称电极材料。

**关键词**：对称固体氧化物电解池；钙钛矿结构；电极材料

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