Chromium deposition and poisoning of La$_2$NiO$_4$ cathode of solid oxide fuel cell

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Chromium deposition and poisoning of La$_2$NiO$_4$ cathode of solid oxide fuel cell were studied. La$_2$NiO$_4$ cathode showed stable performance in the presence of metallic interconnects. Comparing with the polarization resistance ($R_p$) of La$_2$NiO$_4$ cathode in the absence of metallic interconnects, $R_p$ did not change in the presence of metallic interconnect. After electrical conductivity relaxation method, La$_2$NiO$_4$ with high surface oxygen diffusion coefficients working under Cr atmosphere improved the oxygen reduction kinetics and increased cathode $O_2$ reduction reaction rates. No chromium deposition was observed on the La$_2$NiO$_4$ cathode surface after polarization for 20 h at 800°C. The chemical compatibility of La$_2$NiO$_4$/Cr$_2$O$_3$ and La$_2$NiO$_4$/Gd$_{0.1}$Ce$_{0.9}$O$_{1.95}$ (GDC) study indicates that La$_2$NiO$_4$ did not react with Cr$_2$O$_3$ and GDC under the operating temperature. The results indicate that La$_2$NiO$_4$ cathode is a potential chromium-tolerant material of solid oxide fuel cell.

1. Introduction

The intermediate-temperature (600–800°C) solid oxide fuel cells (SOFCs) is the high-efficiency and low-cost power generation system due to the feasibility of using less expensive metallic interconnect materials, such as chromia-forming alloys [1–3]. However, the deposition of Cr eventually leads to a serious polarization loss of the cathode and performance degradation of SOFCs [4–8]. The chromium deposition and poisoning on the cathode is becoming one of the most critical issues for the commercial viability and application of SOFC technologies [9,10]. Thus, development of cathodes with high resistance towards chromium poisoning becomes great challenge for the metallic interconnect based IT-SOFCs.
Until now, many researchers have already tried to develop several cathode materials to be tolerant towards chromium poisoning. Cheng et al. [11] studied the novel MIEC (La0.24Sr0.16Ba0.6)(Co0.5Fe0.44Nb0.06)O3 (LSBCFN) cathode by direct mixing synthesis of BCFN and LSCF powders. The results indicated that LSBCFN cathode showed an excellent stability and tolerance toward chromium deposition and poisoning under SOFC operation conditions. Zhen & Jiang [12] investigated that Sr-free (La,Ba)(Co,Fe)O3 had a potential to be a high performance cathode with high Cr-deposition resistance and Cr-tolerance for IT-SOFCs, in comparison with a (La,Sr)(Co,Fe)O3 (LSCF) electrode. Moreover, it had also been found that La(NiFe)O3-based cathodes had higher resistance to chromium poisoning [13,14]. Huang et al. [15] even reported that an LNF cathode impregnated with Gd0.1Ce0.9O1.95 (GDC; AGC Seimi Chemical Co. Ltd, Japan) made it possible to have good stability for long-term operation under Cr exposure due to very little Cr deposition.

Nowadays, of interest are Ni-based mixed conducting materials, e.g. the Ruddlesden–Popper phases with the K2NiF4-type structure. The mixed ionic–electronic conductor, La2NiO4+δ (LNO) is also being considered as a potential cathode material because of its high oxygen diffusion coefficients and good electronic conductivity [16–19]. Skinner & Kilner [17] showed that the oxygen tracer diffusion coefficient of LNO is higher than that of LSM and LSCF, which indicates also high ionic conductivity for LNO. Therefore, this Sr-free LNO cathode material will be paid much attention on the electrochemical performance, as well as stability in the future. In this paper, the fabrication and cathode behaviour of the LNO cathode have been investigated. The electrochemical performance of the LNO cathode for oxygen reduction is studied for the first time in the presence of chromium-forming alloy interconnects under the operating temperature.

2. Material and methods

Powders of La2NiO4 were prepared using a citrate–nitrate route described before [20]. After calcination at 950°C for 3 h, the as-prepared powders were confirmed by X-ray diffraction (XRD) to be single-phase, tetragonal La2NiO4. La2NiO4 powders were then pressed into a rectangular bar at 300 MPa and sintered at 1350°C for 4 h in air to form dense La2NiO4 bar with relative density higher than 96%, which was satisfied with the requirement of electrical conductivity relaxation (ECR) method [21]. The size of the sintered La2NiO4 bar samples had dimension of 25 mm × 6.6 mm × 0.62 mm. Measurement of oxygen surface exchange coefficient (Kchem) was performed using ECR method. The effect of in situ chromium deposition and poisoning on the LNO bar samples were then studied at 800°C for a period of 24 h.

The polarization resistance (Rp) of the La2NiO4 cathode was measured in the three-electrode cell configuration [22]. Electrolyte pellets were prepared by die pressing GDC powder, followed by sintering at 1500°C for 5 h. The pellets were 0.6–0.9 mm in thickness and 19–21 mm in diameter. Then La2NiO4 powders were dispersed in a solution of 20 wt% terpineol and 80 wt% cellulose to obtain the cathode ink, which was screen printed onto the electrolyte, followed by sintering in air at 1050°C for 3 h. The thickness of the La2NiO4 electrodes was 20–30 mm and the geometric area was 0.5 cm².

A commercial Fe-Cr alloy RA446 (23–27% Cr, 1.5% Mn, 1% Si, 0.2% C, 0.12% N and the remaining Fe; Rolled Alloy Co., Ontario, Canada) was used as the interconnect materials. The alloys were machined into coupons (12 × 12 × 4 mm) with channels (1.2 × 1.2 mm) cut on one side of the coupons. Air was directed to the channels through an alumina tube. Two Pt wires were spot-welded to the coupon to serve as the voltage and current probes, respectively. The interconnect, which also acted as the current collector, was polished to expose the fresh surface and directly placed onto the surface of the La2NiO4 electrode. Detailed cell and interconnect configurations were reported elsewhere [23]. Electrochemical impedance responses were measured using a Gamry Reference 3000 Potentiostat. Impedance curves were recorded under open circuit with frequency range from 0.1 Hz to 100 kHz and the signal amplitude of 10 mV. The Rp was then measured by the differences between the high- and low-frequency intercepts. To determine the phase composition and chemical compatibility of La2NiO4 and GDC, Cr2O3, respectively, and pressed into pellets for XRD characterization at 1050°C, 800°C, respectively. The microstructure of the cells with and without chromium atmosphere was examined by scanning electron microscopy (SEM) and energy dispersive spectrometer using a Zesis EVO with 15 keV.

3. Results and discussion

3.1. XRD analysis

Figure 1 shows XRD patterns of the La2NiO4 and La2NiO4/Cr2O3 mixture after heated at 800°C. It can be clearly seen from figure 1b that a single-phase, tetragonal La2NiO4 (JCPDS PDF 00-034-0314) was formed.
and the result was in accordance with previous papers [24]. As shown in figure 1a, no new peaks were observed at 800°C. So it indicates that LNO and Cr$_2$O$_3$ did not react with each other at certain temperature. The results show that LNO could prevent from the deposition of chromium at 800°C. However, more works would still need to be done for the detailed understanding of the relationship between the LNO and chromium deposition in the future. No reaction or decomposition for LNO/CGO (JCPDS PDF 01-075-0162) was also observed by XRD for 24 h of treatment at 1050°C, as shown in figure 2. It also means that LNO prepared by citrate–nitrate method did not easily react with CGO at certain temperature, which was in accordance with the previous reported papers [25]. Other authors have also reported the compatibility of LNO with both YSZ and CGO at the mentioned temperatures for different periods of time [26,27].

3.2. Microstructure of LNO bar samples

Figure 3 shows the SEM micrographs of LNO sintered at 1350°C. It can be clearly seen that the generally uniform and dense microstructure composed of well-grown grains around 1 μm was achieved at the sintering temperature of 1350°C. The specimen sintering at this temperature attains 96% of the theoretical density. Figure 4 shows SEM micrographs of the LNO surface heat treated in the presence of Cr$_2$O$_3$ for 24 h. It can be clearly seen that the size of the particles did not change quickly, comparing with the SEM micrograph without Cr$_2$O$_3$ of LNO fresh sample. Then the element distribution of selected point on the surface of LNO samples was examined by energy dispersion spectra (EDX) and all the results were given in figure 4c. It can be seen that for LNO bar samples treated at 800°C for 24 h in the presence of Cr, only La and Ni peaks were detected when they were measured on LNO grain.
surface. It indicates that these particles might be able to have a high tolerance towards chromium deposition; moreover, this result was also in accordance with the previous XRD result. Hildenbrand et al. [28] have reported that LNO was a potentially Cr-poisoning resistant cathode material.

3.3. ECR of LNO samples

Figure 5 shows typical normalized conductivity of LNO bar samples with Cr atmosphere at 800 °C when the oxygen partial pressure changed from 0.05 bar to 0.21 bar. It can be clearly seen that the experimental data match well with the fitting curve. The relaxation time was about 100 s. After being exposed to the chromium for 24 h, the calculated $K_{\text{chem}}$ and oxygen diffusion coefficient ($D_{\text{chem}}$) of LNO at 800 °C was $4 \times 10^{-3}$ cm s$^{-1}$ and $5 \times 10^{-4}$ cm$^2$ s$^{-1}$, respectively. It shows that the surface exchange under such condition was fast and the overall relaxation process was already purely controlled by bulk diffusion of oxygen. There is still some literatures report about the fast surface exchange rate of LNO without Cr.
atmosphere. It shows that the sample took 50 s and 150 s to reach equilibrium at 900°C and 750°C, respectively. $K_{chem}$ and $D_{chem}$ at 750°C were $3.9 \times 10^{-5}$ cm$^2$ s$^{-1}$ and $1.6 \times 10^{-3}$ cm s$^{-1}$, respectively. It also points out that the fast surface exchange at high temperature was in good agreement with the literature on tracer and permeation measurements reporting higher activation energy for surface exchange than for diffusion in LNO [17,29]. It indicates that the ECR method used in this study was a reliable technique to study the exchange coefficient for LNO bar samples. Finally, it can also be concluded that there was no significant effect of the chromium atmosphere on the relaxation kinetics of LNO samples, which was in agreement with the previous EDX results.

Figure 5. Normalized conductivity versus time at 800°C upon a step change of $P_{O_2}$ from 0.05 bar to 0.21 bar for the La$_2$NiO$_4$ cathode in the presence of CrO$_3$.

Figure 6. Polarization and impedance behaviour of La$_2$NiO$_4$ cathode in (a and b) the absence and (c and d) presence of metallic interconnects as a function of polarization time at 200 mA cm$^{-2}$ for 20 h and 800°C. Impedance curves were measured at open circuit.
slowly with the polarization time. The \( R_p \) of the LNO cathode under exposure of Cr for 20 h was nearly 0.6 V cm\(^2\), which was slightly higher than that of the same cathode after polarization for 0 h. Compared with the LNO cathode working in the absence of Fe–Cr alloy, the \( R_p \) did not change too much. Meanwhile, the varying trend of the overpotential (\( \eta \)) of LNO cathode working in the absence and presence of Fe–Cr alloy remained the same. \( \eta \) increased from 50 to 90 mV in the absence of Fe–Cr alloy, however, \( \eta \) increased from 50 to 100 mV in the presence of Fe–Cr alloy. So, this indicates that little effect on the chromium poisoning of the metallic interconnect on the activity and performance of LNO cathode. Sayers et al. [30] reported that the cathode area-specific resistance (ASR) of LNO working on CGO was 1.0 \( \Omega \) cm\(^2\) at 700°C. What is more, Pérez-Coll et al. [31] studied that the cathode ASR of LNO working on Co doped, samarium doped ceria was 0.4 \( \Omega \) cm\(^2\) at 800°C. It is commonly believed that the deposition process of (La,Sr)MnO\(_3\) and LSCF was related to the chemical dissociation of the gaseous Cr species, and is likely to be limited by the nucleation reaction between these Cr species and the nucleation agents (Mn\(^{2+}\)) or Sr-segregation, and then it could lead to the increase in \( R_p \) and performance degradation [23]. Also, \( R_p \) increased significantly for BSCF cathode working in the same condition [32]. However, in this study the much lower increase in \( R_p \) for the \( \text{O}_2 \) reduction reaction on the LNO cathode means that this Sr-free cathode material might not easily react with Cr species. It can be explained that for K\(_2\)NiF\(_4\)-type structure, LNO with high surface oxygen diffusion coefficients may improve the oxygen reduction kinetics and increase cathode \( \text{O}_2 \) reduction reaction rates. Then LNO cathode might have a great potential in improving the ability of Cr-resistance in the future. More studies and explanations about SEM images of LNO cathode working in the presence of metallic interconnect under a current density of 200 mA cm\(^{-2}\) for 20 h will be discussed in detail.

Figure 7 shows the SEM images of the cathode surface under the rib and channel of the metallic interconnect after tested under a current density of 200 mA cm\(^{-2}\) for 20 h (\( a \) and \( b \)). The surface of a La\(_2\)NiO\(_4\) cathode tested without interconnect is shown in (c).

Figure 7. SEM micrographs of the surface of La\(_2\)NiO\(_4\) cathode under rib and channels of the metallic interconnect after tested under a current density of 200 mA cm\(^{-2}\) for 20 h (\( a \) and \( b \)). The surface of a La\(_2\)NiO\(_4\) cathode tested without interconnect is shown in (c).
However, there were other reports about the chromium deposition on the LSCF cathode surface under the same conditions [33]. It clearly showed that the mechanism of the chromium deposition and poisoning at these cathodes was affected by the deposition process. LNO cathode material with Sr-free element could have a good tolerance towards chromium.

Figure 9 shows SEM image of the LNO/GDC interface in the presence (a) and absence (b) of metallic interconnect after tested under a current density of 200 mA cm$^{-2}$ for 20 h. It is obvious that GDC electrolyte thin film was essentially dense, with a continuous and crack free surface morphology and

Figure 8. EDX spectra of the La$_2$NiO$_4$ cathode surface under rib and channel of the metallic interconnect tested after under a current density of 200 mA cm$^{-2}$ for 20 h.

Figure 9. SEM micrographs of the fracture surfaces of La$_2$NiO$_4$ cathode in the presence (a) and absence (b) of metallic interconnect after tested under a current density of 200 mA cm$^{-2}$ for 20 h, (c) selected points of EDX spectra of the La$_2$NiO$_4$ cathode in the presence of metallic interconnect, (d) porous La$_2$NiO$_4$ cathode.

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Figure 9 shows SEM image of the LNO/GDC interface in the presence (a) and absence (b) of metallic interconnect after tested under a current density of 200 mA cm$^{-2}$ for 20 h. It is obvious that GDC electrolyte thin film was essentially dense, with a continuous and crack free surface morphology and
no pinholes. The porous structures were still kept in the presence of Cr atmosphere (figure 9b). Cr vapours in air were supplied inside the porous cathodes without significant microstructure change. The microstructures at the cathode/electrolyte interface show no voids and perfect adhesion of porous vapours in air were supplied inside the porous cathodes without significant microstructure change. So it also exhibits mixed electronic and ionic conductivity properties, which was highly desirable for expanding the reaction zone beyond three-phase boundaries [34]. Secondly, according to the results of LNO bar sample with Cr atmosphere tested at 800°C for 24 h, it clearly shows that LNO with high surface oxygen diffusion coefficients might improve the oxygen reduction kinetics and increase cathode O2 reduction reaction rates. Thirdly, some researchers have reported that LNO had higher ionic conductivity, comparing with LSCF and LSM [35–38]. So, it can improve the transport of oxygen ions from the cathode to the electrolyte. Therefore, LNO could probably become a Cr-resistance cathode material. It still needs further study about the stability of LNO cathodes operating in the presence of metal interconnect in the future.

4. Conclusion

Chromium deposition and poisoning of LNO cathode of SOFC are investigated. The Rp did not change too much with the polarization time at 800°C in the presence of metallic interconnect. After ECR method, LNO with high surface oxygen diffusion coefficients working under the Cr atmosphere may improve the oxygen reduction kinetics and increase cathode O2 reduction reaction rates. No Cr deposition is observed in the region of the LNO/GDC interface after operating under a current density of 200 mA cm−2 for 20 h. The chemical compatibility of LNO/Cr2O3 and LNO/GDC study indicates that LNO did not react with Cr2O3 and GDC under the operating temperature. The results demonstrate that La2NiO4 cathode is a potential chromium-tolerant material of SOFC.

Data accessibility. This article does not have any additional data.

Authors’ contributions. B.X.H. and C.C.W. carried out experiments, participated in data analysis and wrote the paper. X.M.C. participated manuscript correction; Y.G.C. and C.C.W. conceived and designed the study. Competing interests. We have no competing interests.

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