Correlation of ohmic resistance and interfacial adhesion strength between cathode and electrolyte for solid oxide fuel cells

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ABSTRACT

In this work, a quantitative correlation between the adhesion strength and the ohmic resistance at the interfaces between the electrolyte and the cathode was established. A cell consisting of LSM/YSZ/LSM was used to tune the interface adhesion strength by changing the sintering temperature. Electrochemical impedance spectroscopy was used to measure the interfacial resistance. The interfacial adhesion strength was obtained by using a mechanical tensile test. The result shows that the ohmic area specific resistance decreases gradually with the increase of the adhesion strength at the interfaces and reaches a plateau at a certain value. **It seems that the polarization resistance has the similar changing trends with that of the ohmic resistance.**

Keywords: Interfacial adhesion strength; Interface area specific resistance; Cathode-electrolyte interface; Solid oxide fuel cells

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1. Introduction

Solid oxide fuel cells (SOFCs) play a key role to provide a sustainable energy in the future economy largely because of (1) the flexibility of the fuels to power SOFCs, ranging from CO, H₂ to hydrocarbons and alcohols and (2) the overall high efficiency > %75 in the combined heat and power systems. In order to commercialize this technology, SOFC stacks must exhibit superior stability and activity [1]. Previous studies have showed that the interfaces between SOFC components governs the cell performance inside a stack repeating unit [2-6], including the electrode-interconnect and the electrode-electrolyte interfaces. We recently reported that the performance of a stack was determined by the performance of a repeating unit when the stack was well sealed with an intimate interfacial contact [7]. In order to further improve and stabilize the cell performance, it is necessary to understand the key factors that influence the cell performance. However, because of the complexity of operating conditions and the cell structure, it is difficult to quantify the contribution of each interface to the cell performance. In 2014, our group developed a method to obtain the quantitative contribution by placing a Pt voltage probe between the electrode and electrolyte [8]. The experimental results showed that there was 90% of cell performance accounts for the interface between electrode and electrolyte. And the interface between cathode-electrolyte and anode-electrolyte contributed about 50% to the cell performance.

Comparing with the SOFC anode, the cathode materials have a lower electrical conductivity and the adhesion between cathode and electrolyte is weak due to a lower sintering temperature to prevent cathode densification. As a result, the cathode is easy to peel off from the electrolyte [9-10]. Consequently, many literature reports on the cell interface focused on the cathode side [11-13]. From these literatures, it was found

that the cell electrochemical performance was mainly dependent on the cathode/electrolyte interface. Many advancements have been obtained through the surface modification of the cathode and the design of the electrode materials [14-18]. It can be known that increasing the reaction sites or areas between LSM and YSZ electrolyte is critical to enhance the performance of the LSM cathodes [17, 19]. Through the modification of cathode surface or design of the interface structure to improve the cathodic contact, it is possible to extend the length and to increase the contact area of the interface. However, no matter what kinds of the contribution mechanisms are, it seems that the bonding effect between cathode and electrolyte interface will be enhanced.

During the past few years, our team has studied the interface between the cathodes and interconnect. It was shown that the essence of the improvement of cathode/interconnect interface bonding is to increase the interfacial adhesion strength [20-22]. The higher the interfacial adhesion strength, the better the interface bonding, thus the smaller the interfacial ohmic resistance, and the higher the cell performance. Similarly, when the interface between the electrode and the electrolyte was improved, the key is to enhance the interfacial adhesion strength. In this work, two thick electrodes were sandwiched by the electrolyte, which were sintered at various temperatures to obtain various different interface bonding strengths between electrolyte and cathode. Electrochemical impedance spectroscopy (EIS) was used to measure the resistance of the interface. A mechanical tensile machine was used to measure the interfacial adhesion strength. The quantitative correlation between the interfacial adhesion strength and the ohmic area specific resistance was established to provide a reference for understanding and improving the interface between electrolyte and electrode.

2. Experimental

Powders of $(\text{La}_{0.75}\text{Sr}_{0.25})_{0.95}\text{MnO}_3$ (LSM) was used as the cathode materials ($200 \text{ S}\cdot\text{cm}^{-1}$ at 750°C [23]), 8YSZ (TZ-8Y, Tosoh Co., Ltd., Japan) as the electrolyte materials ($3.4\times 10^{-2} \text{ S}\cdot\text{cm}^{-1}$ at 750°C [24]). LSM powders were uniaxial pressed under 180 MPa to form disks with approximately 20 mm in diameter and 4 mm in thickness. The 8YSZ pastes were screen-printed on one side of the LSM disk and stacked to another LSM disk to form a symmetrical sandwich structure of LSM/YSZ/LSM. The schematic of the sample preparation is shown in Fig. 1. The sandwich cell was dried in a hot chamber to reduce the mobility of the electrolyte paste and then sintered at various schedules as listed in Table 1, during which the sandwich samples were loaded under a 100 g/cm^2 pressure. The sintered specimens with various bonding strengths were obtained by controlling the heating rate and holding time. The heating rate, maximum temperature and holding time at different sintering processes are shown in Table 1. The electrolyte thickness of the sandwich structure sample is $\sim 10\text{-}15\mu\text{m}$ after sintering, as shown in Fig. 2.

A layer of silver paste was coated on both sides of the symmetrical specimen and sintered at 850°C . After that, the temperature was lowered to 750°C for the electrical measurements. We used a four-probe method and CS2100 electrochemical workstation to measure its impedance performance, as shown in Fig. 3. The effective area of the specimen in this work is about 2 cm^2 , the measurement frequency range and the applied voltage were 1MHz to 0.1Hz and 20mV, respectively. The theoretical

area specific resistance (ASR) of the specimen can be calculated by using the following formula (1), (2):

$$Rs = \sum(1/\sigma_j)(L_j/S) \quad (1)$$

$$ASR = R_s \times S \quad (2)$$

Where L_j (cm) is the thickness of component j, S (cm²) is the contact area, and σ_j is the conductivity of component j (σ_{ji} -ionic conductivity, σ_{je} -electronic conductivity).

The specimens were taken out from the furnace after the EIS test was finished. Then, the Ag pastes on both sides of the sample were removed. An E7 epoxy resin adhesive (commonly known as AB glue, quality ratio 10:1) was used to mount the specimen on the tensile test mold, as shown in Fig. 4. The mechanical tensile test was then carried out by using a universal testing machine (CMT5105) controlled with a microcomputer. The interfacial bonding strength can be calculated by the following formula [25]:

$$\sigma = F_p/S \quad (3)$$

σ is the average bonding strength (MPa) of the interface; F_p is the interfacial adhesion force. After the tensile test, the interface microstructure and the element distribution in cathode-electrolyte interface were characterized by using a Scanning Electron Microscopy (SEM, FEI Quanta FEG 250) with X-ray energy dispersive system (EDS). The constituent phases of mixed powders sintered under various sintering processes were identified by X-Ray Diffraction (XRD, D8 Advance).

3. Results and discussion

Fig. 5(a) shows the EIS results of the specimens, P1, P2, P3, and P4, measured at 750°C in the first experiment. The ohmic resistance R_s is obtained from the high

frequency intercept, whereas the total resistance R_t is determined from the low frequency intercept. The polarization resistance R_p is obtained by subtracting R_s from the total resistance R_t . The ohmic resistance of P1, P2, P3, and P4 is $0.7 \Omega \cdot \text{cm}^2$, $1.6 \Omega \cdot \text{cm}^2$, $2.3 \Omega \cdot \text{cm}^2$ and $2.8 \Omega \cdot \text{cm}^2$, respectively. The trends of the total R_t are consistent with the ohmic R_s . The polarization resistance of P1, P2, P3 and P4 is $3.64 \Omega \cdot \text{cm}^2$, $6.63 \Omega \cdot \text{cm}^2$, $20.73 \Omega \cdot \text{cm}^2$ and $25.64 \Omega \cdot \text{cm}^2$, as shown in Fig. 5(a). According to the Eq. (1), the theoretical ohmic resistance of the electrolyte in this study is $\sim 0.06 \Omega \cdot \text{cm}^2$, which is negligible compared with that of the total ohmic resistance. Therefore, the ohmic resistance of the cell is mainly determined by the interfacial resistance.

Comparing all the specimens P1 to P4, we observed that the heating rate played a key role on the interfacial resistance. A decrease in the heating rate can significantly decrease the interface resistance, which may be related to the diffusion and mass transfer during sintering. With an increase in the sintering temperature, rapid grain growth occurs, resulting in the presence of porosity at the interfaces. Hence, the slower heating rate results in the more uniform mass transfer of the materials, thus creating an intimate contact between the cathode and the electrolyte. At elevated sintering temperatures (e.g. $> 1300^\circ\text{C}$), $\text{La}_2\text{Zr}_2\text{O}_7$ (LZO) and SrZrO_3 (SZO) were reported to form at the LSM/YSZ interface, which resulted in a decrease in cell performance [2-4, 26]. In this work, the LSM/YSZ/LSM specimen was sintered at 1300°C , and the phases of LZO and SZO can potentially be formed. In order to validate this reaction, an equal amount of LSM and 8YSZ powders were mixed and then sintered with the same protocols listed in Table 1. Fig. 6 shows the phase composition of the powders after sintering. In detail, in Fig. 6, open triangle (∇) and closed ($\bullet$) and ($\blacklozenge$) respectively indicate peaks of the XRD patterns originating from 8YSZ, LZO and LSM. A small amount of LZO was detected in the powder sintered

by the procedure to make P1 sample; while the new phase was not observed in the powders treated with the procedures to make either P2, P3, P4 specimens. It seems that the formation of the insulating phase is so little that it can't be detected or no new phase was formed under the sintering procedure. Comparing with Fig. 5(a), the ohmic resistance of P2 is higher than that of P1, and the sample of P4 has the maximum ohmic resistance. There was almost no insulating phase in the sample of P2, P3 and P4 (Fig. 6). Therefore, the contribution of the insulating phase to the resistance is very small, while the contribution of the interface bonding to the ohmic resistance governs the interfacial resistance. The EDS line scan profile, as shown in Fig. 7, also shows the element distribution in cathode-electrolyte interface is uniform in the sample of P1, P2, P3 and P4. The influence of the insulating phase can be neglected.

Fig. 8 shows the adhesion strength of the LSM/YSZ/LSM specimens. Because all specimens were fragile and easy to fracture during the tensile test, we used the maximum loading to characterize the interface adhesion strength. The adhesion strengths of P1, P2, P3 and P4 are 0.276 kN, 0.202 kN, 0.189 kN, and 0.183 kN, respectively. The interfacial bonding strength is determined by the heating rate. The specimen sintered with a slowest heating rate exhibits the highest adhesion force. According to Eq. (3), we can obtain the average bonding strength, as shown in Fig. 9. The curve 1 in Fig. 9 (a) shows that the ohmic resistance decreases with increasing interfacial bonding strength, following an exponential relationship, Eq. (4):

$$y = \exp\{-a(x - 1.02)\} + 0.6 \quad (4)$$

The coefficient, a , is ~ 7 and the error of the fitting curve is as less as 0.05, when substituting the other two experimental points into the equation. The curve 1 in Fig. 9 (a) shows that the ohmic resistance decreases gradually and reaches to a minimum value with increasing interfacial bonding strength. According to the aforementioned

fitting results, the bonding strength and the ohmic resistance of the interface follow Eq. (5):

$$y = \exp\{-7(x - 1.02)\} + 0.6, x \in R, x > 0, |\Delta| \leq 0.05 \quad (5)$$

where x represents the interfacial bonding strength and y is the ohmic resistance.

The fitting results matched well with the experimental data.

To further validate the relationship between the ohmic resistance and the bonding strength of the interface, the experiment was repeated under the same condition and the results are shown in the curve 2 in Fig. 9 (a). Comparing the results in the curve 1 and the curve 2, we found that the ohmic resistance and interfacial bonding strength exhibited similar trend. The relationship between ohmic resistance and interfacial bonding strength still satisfies an exponential curve. The fitting curve 2 shown in Fig. 9 (a) follows Eq. (6):

$$y = \exp\{-6(x - 0.71)\} + 0.65 \quad (6)$$

Hence, the ohmic resistance and interfacial bonding strength between cathode and electrolyte follows an exponential relationship. The formula can be expressed:

$$y = \exp\{-a(x - b)\} + c; a, b, c > 0; a, b, c \in R \quad (7)$$

Therefore, the improvement of the interfacial bonding strength can reduce the ohmic resistances of the SOFC. The variation in polarization resistances versus bonding strength in the experiments are shown in Fig. 9(b). With the increase of the bonding strength, the polarization resistance also decreased, and has the similar changing trends with that of the ohmic resistance. According to the early results [27-28], the increase of the resistances is the major factor causing the cell degradation as the SOFC is working at the operating condition. Therefore, one can consider to reduce the resistances by improving the interfacial bonding strength.

Fig. 10 shows the surface morphology of the samples after tensile test. In

comparison with Fig. 2, all specimens were separated from the cathode/electrolyte interfaces. The fracture surface is shown at the electrolyte side. Fig. 10 (a) shows the contact between LSM/YSZ is much more uniform in the P1 specimen than all the others. This is consistent with the highest interface bonding strength and the lowest ohmic resistances observed in P1.

4. Conclusion

The cell configuration of LSM/YSZ/LSM was used and sintered at various heating rates to prepare the specimens with different interface strengths. The ohmic resistance was measured at the temperature of 750 °C by EIS. The corresponding bonding strength between cathode and electrolyte was measured by using a mechanical tensile equipment. The results showed that the ohmic resistance of the samples decreases with the increasing interfacial bonding strength, following an exponential relationship, where generally it can be expressed as $y = \exp\{-a(x - b)\} + c$; $a, b, c > 0$; $a, b, c \in R$. And the polarization resistance has the similar changing trends with that of the ohmic resistance.

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Figures captions

Fig. 1 - A schematic diagram showing the process to make LSM/YSZ/LSM specimens.

Fig. 2 - A cross sectional SEM image of P1 symmetrical specimen after tensile test.

Fig. 3 - Schematic diagram of EIS test for symmetrical samples. CE: counter electrode; WE: working electrode; RE: reference electrode.

Fig. 4 - A schematic of the vertical tensile test setup (a) and the image of the tensile test setup (b).

Fig. 5 - Nyquist plots of P1, P2, P3, and P4 measured at 750°C in the first experiment (a) and the second experiment (b).

Fig. 6 - X-ray diffraction patterns LSM and 8YSZ mixed powders sintering at conditions to make P1, P2, P3 and P4 specimens.; LZO= $\text{La}_2\text{Zr}_2\text{O}_7$; YSZ= $\text{Y}_{0.08}\text{Zr}_{0.92}\text{O}_2$; LSM= $\text{La}_{0.65}\text{Sr}_{0.35}\text{MnO}_3$.

Fig. 7 - EDS scan profile showing the distribution of the elements in the cathode/electrolyte interface of samples P1 (a) and P3 (b).

Fig. 8 - A plot of the interfacial adhesion strength and displacement during the tensile tests.

Fig. 9 - Experimental results and the fitting curves of the ohmic resistances R_s versus interfacial adhesion strength (a) and variation in polarization resistances R_p versus bonding strength (b).

Fig. 10 - SEM images of the interfaces after the tensile test for P1 (a), P2 (b), P3(c) and P4 (d).

Table captions

Table 1 - Sintering parameters of different samples.

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Table 1 - Sintering parameters of different samples.

Samples	heating rate - maximum temperature - holding time
P1	0.25°C /min-1300°C -0h
P2	0.5°C /min-1300°C -4h
P3	1°C /min-1300°C -2h
P4	1°C /min-1300°C -4h