YSZ/LSM Composite Cathode Deposited by Solution Precursor Plasma Spraying

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Abstract: In this paper, solution precursor plasma spraying (SPPS) was employed to prepare a porous YSZ/LSM composite cathode for solid oxide fuel cell (SOFC). The surface morphology and microstructure of the composite cathode deposits were characterized using SEM. The effect of annealing treatment on SPPS YSZ/LSM microstructure was examined. The results showed that the as-sprayed YSZ/LSM deposits presented a porous aggregate with a size range of 10–60 µm when the alcohol was used as the solvent and the spraying distance was 60 mm. The porous aggregate was found to be composed mainly of small particles ranging from 0.2–2 µm, the YSZ/LSM composite cathode showed a finely porous microstructure with grain sizes from micrometers to sub-micrometers. A further annealing treatment at 1050 °C for 2 h in air resulted in a continuous microstructure porous coating with a perovskite phase. The polarization test results demonstrated that the minimum polarizations were 1.26 and 0.083 Ω·cm² for the composite cathode at 800 and 1000 °C, respectively.

Keywords: solid oxide fuel cells; solution precursor plasma spraying; polarization resistance; YSZ/LSM composite cathode

1. Introduction

Solid oxide fuel cells (SOFCs) are highly efficient chemical fuel cells, which have been widely used in submarines [1], automobiles [2], aircraft [3], and other fields [4]. The successful application of this technology is of great significance for alleviating the energy crisis and protecting the ecological environment [5]. However, in the process of commercializing solid oxide fuel cells, the high manufacturing cost of SOFC power generation systems, low output power density, and low long-term stability of the cell stack have become the main obstacles to their large-scale commercial development [6–8].

In recent years, many studies have focused on the materials [9–11], the preparation process [12,13], and the structure optimization [14–16] of SOFC power generation systems to establish a competitive SOFC system compared with the existing power generation technology. In SOFC power generation systems, one method of saving costs is to reduce the operating temperature. However, as the operating temperature decreases, the electro-chemical performance of the SOFC also decreases, which is mainly due to the increase in the polarization resistance of the electrodes and the decrease in the conductivity of the electrolyte [17].

While, with the thinning of electrolytes, the wide application of new electrolyte materials in SOFC, and the optimization of the anode structure, cathode polarization has become the main bottleneck restricting the power output of batteries [18–20]. Therefore, the focus of attention has transferred from the electrolyte to the cathode. Recently, many new cathode materials have been evolved and shown high catalytic activity [12,21]; however, their long-term stability is still a challenge to overcome.
When it comes to long-term stability, the traditional $\text{Y}_2\text{O}_3$-stabilized $\text{ZrO}_2/\text{La}_{1-x}\text{Sr}_x \text{MnO}_3$ (YSZ/LSM) material system has its advantages and therefore attracts special research interest as one of the solutions to commercialize SOFC [22,23]. Solution precursor plasma spraying (SPPS), an easy and low-cost process, has the potential to prepare coatings with submicron or nanoscale porous structures [24,25].

According to our previous studies [26,27], $\text{La}_{0.8}\text{Sr}_{0.2}\text{MnO}_3$ (LSM) cathodes have been successfully fabricated using SPPS. However, the polarization properties still need to be improved. In an earlier report, Prakash et al. [28] prepared LSM/YSZ-based solid oxide fuel cell cathodes by SPPS; however, the effect of an annealing treatment on the surface morphology of YSZ/LSM was not considered in that study. In addition, the microstructure and polarization properties of YSZ/LSM composite cathodes and the single LSM cathode prepared by SPPS were also not investigated systematically.

Herein, the traditional YSZ/LSM composite cathode was prepared by solution precursor plasma spraying. The microstructure control law of YSZ/LSM composite cathode and its structure–activity relationship with performance were clarified to establish a low-cost preparation method of the high-performance cathode. The microscopic morphology and polarization properties of YSZ/LSM were compared with those of a single LSM cathode prepared by the plasma spraying of solution precursors to provide an effective way of solving the current commercial development of SOFC.

2. Materials and Methods

2.1. Materials

The nominal compositions of the YSZ and LSM in this paper were 0.08 mol $\text{Y}_2\text{O}_3$-stabilized $\text{ZrO}_2$ and $\text{La}_{0.8}\text{Sr}_{0.2}\text{MnO}_3$, respectively. The molar ratio of YSZ and LSM in the YSZ/LSM composite cathode was 1:1. In a typical preparation, 0.16 mol $\text{Y(NO}_3)_3 \times 6\text{H}_2\text{O}$, 1 mol $\text{Zr(NO}_3)_4 \times 5\text{H}_2\text{O}$, 0.8 mol $\text{La(NO}_3)_3 \times 6\text{H}_2\text{O}$, 0.2 mol $\text{Sr(NO}_3)_2$, and 1 mol $\text{Mn(NO}_3)_3$ (solution, 50 wt.%) were dissolved in distilled water. The raw materials used in the experiment were obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). After adding an appropriate amount of absolute ethanol, the mixture was stirred with a magnetic stirrer until obtaining a uniform solution. The amount of ethanol added should ensure that the concentration of nitrate ions in the solution is 0.1 mol/L. The obtained solution was used as a spraying precursor solution. A molded YSZ ceramic sheet was used as substrate material, with a thickness of about 1 mm and a diameter of about 20 mm. Before spraying, the substrate surface was ground and polished.

2.2. Preparation and Characterization of YSZ/LSM Composite Cathode

The YSZ/LSM composite cathode was sprayed by SPPS technology. The surface and cross-sectional morphology of the YSZ/LSM cathode coating was observed by scanning electron microscope (SEM, VEGA II-XMU, TSCAN, Brno, Czech Republic), the phase structure was characterized by X-ray diffraction (XRD, Rigaku D/max-2400, Tokyo, Japan), and the porous structure and thickness of the coating were analyzed by an imaging method. The SPPS deposition system was composed mainly of a traditional plasma spray gun and a liquid feeding system.

The plasma spraying equipment was an XM-80SK high-energy plasma spraying system manufactured by Shanghai Xiuma Spraying Machinery Co., Ltd. (Shanghai, China). The liquid material can be fed into the plasma flame by a peristaltic pump through a small hole with a diameter of 0.4 mm in the nozzle. In the experiment, Ar was used as the main gas and $\text{H}_2$ as the auxiliary gas. The arc power used to generate the plasma jet in this experiment was 36 kW (60 V × 600 A), the pressure and flow rates of Ar were 0.8 MPa and 40 L/min, and the pressure and flow rates of $\text{H}_2$ were 0.4 MPa and 30 L/min, respectively as shown in Table 1.
Table 1. The SPPS spraying process parameters.

| Parameter                  | Value      |
|----------------------------|------------|
| Spraying power             | 36 kW      |
| Arc current                | 600 A      |
| Arc voltage                | 60 V       |
| Main gas (Ar)              | 40 L/min   |
| Auxiliary gas (H₂)         | 30 L/min   |
| Substrate temperature      | 400 °C     |
| Liquid delivery speed      | 80 rpm     |
| Spraying distance          | 60 mm      |

During the spraying process, the spray gun was fixed on the manipulator, and the spraying distance and the moving speed of the spray gun can be controlled by the manipulator. To prepare a circular composite cathode with a diameter of 6.5 mm, a mask with an opening diameter of 6.5 mm was used to cover the YSZ substrate, and the axis of the plasma spray gun was adjusted to align with the center of the mask. To increase the adhesive strength between the deposited particles and the substrate, prior to the spraying process, the backside of the substrate was heated slowly by a flame gun, and an infrared thermometer was employed to measure the surface temperature of the substrate. Coating deposition began when the substrate temperature reached 400 °C.

2.3. Characterization of Polarization Properties of Composite Cathode

Figure 1 shows a schematic diagram of the polarization test device. The geometrical area of the porous YSZ/LSM composite cathode was 0.33 cm². As the counter electrode, Pt paste was painted symmetrically to the composite cathode on the opposite side of the electrolyte pellet. As the reference electrode, a Pt paste with a width of 2 mm was painted at the perimeter of the pellet. Reference and counter electrodes were fixed to the pellet through firing at 850 °C for 0.5 h.

The distance between Pt reference electrode and composite cathode was about 5 mm. The impedance measurements were conducted in air from 800 to 1000 °C at an interval of 50 °C. A frequency response analyzer (Solartron1260) and an electrochemical interface (Solartron 1287, Solartron analytical, Hampshire, UK) were used for the electrochemical impedance measurements. The tests were performed in a frequency range from 0.1 to 10⁵ Hz with the amplitude of the AC signal of 100 mV at equilibrium potential. The three electrode configuration and test approach was proposed in the reference [29] in detail.

Figure 1. Schematic diagram of the polarization resistance test device.
3. Results and Discussion

3.1. Phase Structure of YSZ/LSM Deposits

In SPPS, metal nitrates were used as the raw materials for the starting precursor. During deposition, the droplets injected into the plasma jet will undergo rapid solvent evaporation, solute precipitation, decomposition, chemical reaction and synthesis, melting, solidification, and crystallization. Then, these particles are collected to form a deposit. The X-ray diffraction patterns of the YSZ/LSM composite deposits are shown in Figure 2. We observed that the as-sprayed deposit presented a partially crystallized phase.

Although the temperature of the plasma flame is very high, the dwelling time of droplets in the plasma jet is very short [30]. If the droplet dwells for insufficient time within the plasma flame or passes through the periphery of the plasma jet, incomplete evaporation of the solvent and condensation of the precursor materials occurs; this will result in incomplete crystallization. After annealing in air at 1050 °C for 2 h, complete crystallization to the desirable phase was observed, which is evident from the increased XRD peak intensity of the annealed sample.

![Figure 2](image2.png)

**Figure 2.** XRD pattern of YSZ/LSM deposits: (a) as-sprayed and (b) after annealing at 1050 °C for 2 h in air.

3.2. Microstructure of YSZ/LSM Prepared by SPPS

Figure 3 shows the surface morphology of YSZ/LSM prepared by SPPS with a spraying distance of 60 mm. From Figure 3a, the deposited particles present aggregates with sizes ranging from 10 to 60 µm. It can be inferred that each agglomerate corresponds to a large droplet in the plasma flame. These agglomerates have a loose and porous structure, with large pores between the agglomerates.

![Figure 3](image3.png)

**Figure 3.** The surface morphology of YSZ/LSM prepared by ethanol SPPS at a spraying distance of 60 mm: (a) Low magnification. (b) High magnification.
From the higher magnification of the surface, as shown in Figure 3b, the deposited agglomerates are composed of small particles of 0.2–1.5 µm, which can help to increase the triple phase boundaries (TPB) length of the cathode, thereby, reducing the polarization resistance. The polarization of the cathode is related to the length of the TPB, Deng et al. [31] established a calculation model for the length of the TPB in SOFC, and the various influencing factors on its length were also simulated and calculated. Under the condition of constant porosity, the length of the TPB increased with decreasing particle size, resulting in smaller polarization resistance.

3.3. Effect of Annealing Treatment on the Microstructure of YSZ/LSM Composite Cathode

Figure 4 shows the surface morphology of the YSZ/LSM composite cathode prepared by SPPS after annealing treatment at 1050 °C for 2 h in air. Compared with the surface morphology of the untreated sample, the surface edges and corners of the large agglomerates of the heat-treated sample became smaller, as shown in Figure 4a, while the small particles in the agglomerates were almost the same for both samples, as shown in Figures 3b and 4b.

This indicates that the microstructure of the sprayed YSZ/LSM deposits remained stable during the annealing treatment process, and there was no obvious particle sintering and growth phenomenon. This feature indicates that the YSZ/LSM composite cathode would exhibit good structural stability under high temperature operating conditions.

Figure 5a shows the as-sprayed cross-sectional morphology of the YSZ/LSM cathode prepared by SPPS. The YSZ/LSM showed a porous structure with a thickness of about 40 µm; however, the layered structure features of LSM prepared by plasma spraying of aqueous precursors were not observed [26,27] as shown in Figure 5b.

Figure 4. Surface morphology of the YSZ/LSM composite cathode after annealing treatment at 1050 °C for 2 h: (a) Low magnification. (b) High magnification.

Figure 5. The as-sprayed cross-sectional morphology of the YSZ/LSM cathode and LSM cathode prepared by ethanol SPPS: (a) YSZ/LSM cathode. (b) LSM cathode.
Figure 6 shows the cross-sectional morphology of the YSZ/LSM cathode prepared by SPPS after annealing treatment at 1050 °C for 2 h. From Figure 6a,b, the cross-sectional structure of the coating after annealing treatment was not greatly different from that of the untreated coating. The YSZ/LSM cathode after annealing treatment was mainly composed of agglomerates containing spherical particles.

The agglomerates had a porous structures with micron to submicron and even nanometer scales. In order to obtain a clearer view of the porous structure, the YSZ/LSM cathode was impregnated with commercial E-7 glue, and then the polished section was prepared after the pores were immobilized. It can be seen, from Figure 6c,d, that there were large pores between some of the large agglomerates, and most of the pores were between 0.2 to 2 μm in size.

Figure 6. The cross-sectional morphology of the SPPS YSZ/LSM cathode after annealing treatment: (a,b) As-sprayed: (a) Low magnification. (b) High magnification. (c,d) Impregnating and polishing: (c) Low magnification. (d) High magnification.

3.4. The Electrochemical Performance of YSZ/LSM Composite Cathode

The polarization resistance properties of the cathode were tested using the AC impedance method. Figure 7 shows the Nyquist diagrams of the YSZ/LSM cathode after annealing at 1050 °C for 2 h, which were tested at 800–1000 °C. Each curve intercepted the real axis at a low and a high frequency, respectively. The high frequency intercept $R_0$ is the overall ohmic resistances, including the YSZ ionic resistance, LSM electronic resistance, contact resistance between the electrode and Pt mesh, and external cell connection resistance. The intercept difference between the low and high frequency of the semicircle represents the cathode polarization $R_p$, as shown in Figure 7. The polarization resistance decreases between the electrolyte and electrode as the temperature increases.

Table 2 shows the polarization resistance values of the LSM [27] and YSZ/LSM cathodes at different temperatures. As indicated in Table 2, the cathodic polarization resistance decreased with increasing temperature. This is mainly due to the increase of cathodic
activity and electrochemical reaction rate at the interface with the increase of temperature and thus decrease of the cathodic activation polarization [27]. From Table 2, the YSZ/LSM prepared by SPPS had a lower polarization resistance. When the temperature increased from 800 to 1000 °C, the polarization resistance decreased from 1.26 to 0.083 Ω·cm². These values are about 0.31–0.55 times that of the LSM prepared by ethanol SPPS. This shows that the YSZ/LSM composite cathode had better performance than the single LSM cathode.

Table 2. Comparison of polarization properties of YSZ/LSM and LSM.

| Cathode Types | Polarization Resistance (Ω·cm²) |
|---------------|---------------------------------|
|               | 800 °C  | 850 °C  | 900 °C  | 950 °C  | 1000 °C |
| YSZ/LSM       | 1.26    | 0.33    | 0.18    | 0.11    | 0.083   |
| LSM [27]      | 2.55    | 1.04    | 0.4     | 0.21    | 0.15    |

4. Conclusions

In this paper, the microstructure of a YSZ/LSM composite cathode deposited by alcohol SPPS was studied. The effect of an annealing treatment on the morphology of the composite cathode was investigated, and the electrochemical performance of a YSZ/LSM composite cathode was also preliminarily studied. The results showed that the as-sprayed deposits presented a partially crystallized phase. After an annealing treatment at 1050 °C for 2 h, the deposits crystallized completely, and the morphology with a porous structure did not change greatly. The YSZ/LSM composite cathode was mainly composed of agglomerates containing spherical particles with a size of 0.2–2 μm. The polarization resistances of the YSZ/LSM composite cathode were 1.26 and 0.083 Ω·cm² at 800 and 1000 °C, respectively, which were lower than the LSM cathode prepared under the same conditions. The addition of the YSZ electrolyte phase was beneficial to improving the cathode performance.

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