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Thin 8 mol% yttria stabilized zirconia interlayer for intermediate-temperature solid oxide fuel cells

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A layer of zirconia coting on anode by precision spin-coating was used to prepare thin electrolyte layer of nano-sized (ca. 60 nm) powder and modify the contact resistance of a cell. The powder was ground, dispersed and spin-coated on a (NiO+8 mol% yttrium stabilized zirconia) anode disk for flattening the electrolyte surface. Bi-layer electrolyte, consisted of the nano-zirconia and Tosho zirconia powders, for planar fuel cells were made in thickness $5.4 \pm 0.2 \mu m$ for comparison to the cells only with one layer Tosho zirconia electrolyte. The electrolyte layers are characterized on several processing factors and the polarization resistance of half-cells by electrochemical impedance spectra (EIS) analysis. The results show that the addition of nano-zirconia thin layer greatly decreases the surface roughness, interfacial resistance, and effectively improves short-term performance of the cell power output.

Key words: Zirconia, Interlayer, Electrolyte, Interfacial resistance.

Introduction

Fuel cells (FCs) are one of the promising devices for generating electric power and reduce environmental impact as the cells are operating. Solid oxide fuel cells (SOFCs) are one of the FCs made without using precious Pt and effectively converting chemical energy into electrical energy at temperatures as low as 600 °C. The devices show high energy convergent efficiency, fuel flexibility and low pollutant emission [1, 2]. The cells mainly consist of a cathode, possibly multiplelayer electrolyte and anode, and can be made in planar type by facile processing methods, e.g. tape casting or spin-coating [2]. In addition, some other components, such as an additional inter-layer of the electrolyte, were developed to improve the performance of the cells [3, 4].

The control of the electrolyte thickness and use of reliable process are two important issues to get consistent properties of the cell, of which the electrolyte is mainly the source of the resistance (or polarization) of the made cells. One of the powder processes, spin-coating method is often used to fabricate ceramic thin films in electronic industry, and often used for the electrolyte layer in SOFCs. The film thickness and the quality are difficult to control, but influenced by many processing parameters, e.g. slurry viscosity (h), slurry solid loading (C), spin-coating rotating speed (R_s), coating cycle time (t),

evaporation conditions and sintering conditions. (The last two conditions are treated constant in this study.)

Emslie *et al.* [5] derived the relationship of the thickness of a spin-coated film to the following processing parameters by considering a flow of a viscous liquid (a Newtonian fluid) on a rotating, dense disk. They gave the relationship as below.

$$h(t) = h_0 \left(\frac{1 + 4\rho_L \omega^2 h_0^2 t}{3\eta}\right)^{-1/2}$$
(1)

where h(t) is the film thickness, *t* is spinning time, h_0 is initial height of a fluid layer containing a constant solid phase, ρ_L is density of the liquid, and ω is angular velocity (rad.s⁻¹) of spin condition. While the viscosity of a low solid-loaded slurry did not change much with the solid loading, the viscosity could be taken as a constant. Therefore, several inconsistencies of the relationship in Eq. (1) have been reported in literature. Hui *et al.* [7] suggested that the thickness of sintered spin-coated layers from SEM observation was different from the original value (green state). Since the drying and sintering processes were carried out, the comparison should be based on the same processing conditions. Besides, the relationship can be simplified [6] to depict the relationship as below.

$$\mathbf{h}(C) = \alpha \times C \times \omega^{-1} \tag{2}$$

where α is an empirical constant.

In this study, planar type SOFCs of anode supported

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is selected. We use porous anode of constant porosity and thickness $(300 \pm 5 \,\mu\text{m})$ as a substrate. Precision spin-coating method in tight control of the coating parameters and consistent (good quality) slurries for spinning is used to prepare thin-layer electrolytes and cathode on the anode layer. Since the anode consists of micro- to nano-sized pores, which make the interface of the anode/electrolyte uneven, a thin electrolyte prepared from well-dispersed 8YSZ-N slurry is added between the anode/electrolyte interfaces. This study consistently practices the spin-coating parameters, and therefore precisely controls the electrolyte thickness and reduces the variation (STD) of the layer. The effects of the solid loading and spin-cycle time to the thickness are investigated. As a result, the interfacial conditions are improved and the resistance of the electrolyte/anode is reduced.

Material and Methods

Dispersion of nano-8YSZ slurry

An 8 mol% yttrium stabilized zirconia powder (8YSZ-N, Tsinghua University, China) was dispersed into de-ionized water with 1.74 wt% D-134 (ammonium salt homo-polymer with 2-propenoic acid group, Daiichi Seiyaku Co. Ltd., Japan) as a dispersant [8]. The slurry with a solid loading of 1.74 wt% or 3.5 wt% (based on power) was ball-milling for 100 min by Φ 0.2 mm 3YSZ media, then changed to attrition milling (Buhler, Switzerland) for additional 0 min-230 min. The other case was using Tosho 8YSZ powder (8YSZ-T, Tosho Corp., Japan) which was ball-milling for 0-72 hr. The particle size of the ground slurries was measured by a laser diffraction particle size analyzer (LS230, Beckman Coulter, US) to assure the dispersive conditions of the 8YSZ slurries.

Cell fabrication

Nickel oxide powder (NiO, F grade, NICO Ltd., Japan) and the 8 mol% yttria stabilized zirconia (8YSZ) powder were separated dispersed in ethanol (95% purity, Taiwan Tobacco & Liquor Corporation, Taiwan) and ball-milled for 72 hr and additional 20 hr by attrition milling, respectively. The dispersed and milled slurries are named 8YSZ-T and 8YSZ-N. Then the dispersed NiO and 8YSZ-T slurries were mixed together in a mass ratio of 1.0:1.0. The mixed slurry added with 20 wt% carbon powder (meso-carbon micro-black, as abbreviated "MCMB", National Chung-Shan Institute of Science & Technology, Taoyuan, Taiwan). After additional ball-milling for 1.0 hr and rotary drying, the dried anode powder was sieved through a 100 mesh sieve and die-pressed into disks with a 30.0 mm diameter and 0.50 mm thickness. After that, the disks were pre-sintered at 1000 °C for 1.0 hr in air to burn the carbon species (MCMB) and showed an average

linear shrinkage of 4.12%. Finally, the disks were cosintered with a coated electrolyte at 1400 $^{\circ}$ C for 1.0 hr to an average porosity of 26.4%.

Two different electrolytes are prepared. The first electrolyte is a bi-layer electrolyte with one layer of nano 8YSZ-N and a second layer of 8YSZ-T. The other electrolyte consisted of only one layer of 8YSZ-T. The dispersed 8YSZ-N slurry was diluted to 0.10 wt%-1.00 wt% for the following spin-coating test. The 8YSZ-N slurry were spin-coated on the anode substrate (pre-sintering at 1000 °C for 1 hr in air) by the conditions of 3000 rpm, lasting for 60 s, and repeating 1 to 3 cycles.

Additional electrolyte film was made from the 8YSZ-T slurry to get a thin layer in a range of 5-10 μ m. In order to get a desired electrolyte film (for instance, $5.4 \pm 0.2 \mu$ m), three spin conditions with different solid loadings of the slurry (5.0 wt % spinning 2 cycles, 1.0 wt% spinning 3 cycles, or 1.0 wt% spinning 6 cycles) were carried out to check the water leakage of the sintered half cells.

For the per-sintered anode disk, the diameter reduced from 30.00 mm to $28.76 \pm 0.51 \text{ mm}$. For the half cell (anode/electrolyte) sintered at $1400 \text{ }^{\circ}\text{C}$ for 1 hr, the diameter was $20.45 \pm 0.59 \text{ mm}$, thus the average linear shrinkage during the co-firing was 28.9%. In order to prevent the warpage of the half cells, a porous alumina block was left on the top of the cells during the co-firing process.

A cathode powder $Bi_{0.7}Sr_{0.3}Fe_{0.5}Mn_{0.5})_{3-\delta}$ (BSFMn0.5) [9] mixed with same volume fraction CeO₂ powder (99.9% pure, Yon-Song, Taoyuan, Taiwan) was dispersed into PEG 200 (Average Mw = 200, >99%, Hayashi Pure Chemical Ind, Ltd., Japan) in a solid loading of 50 wt%. After ball-milling for 72 hr, the slurry was spin-coated on the 1400 °C sintered half-cell in the selected conditions of 1000 rpm, lasting for 20 s. The coated cell was sintered at 950 °C for 1 hr to get a desired cathode film.

Characterization

Scanning electron microscope (SEM, JSM6510, JEOL, Japan) was used to analyze the microstructure (thickness and grain size) of sintered layer cells. A secondaryelectron image (SEI) mode was used to observe the surface morphology. The surface altitude was imaged and quantified by a digital microscope (VHX-5000, Keyence, Japan). Thickness from 10 random locations of the sintered cells was measured and calculated the average thickness and standard deviation.

Total conductivity (σ) of sintered samples from 600 °C to 800 °C is measured by a two-probe DC technique [10]. Two surfaces of the disk samples were coated a thin Pt layer (<50 nm), then connected to an Ag wire by Ag paste (Sinetek Co., Ltd, Taiwan). The samples are binder-burn-out at 700 °C for 30 min. The

conductivity of the samples is calculated as below.

$$\sigma = \frac{S}{R \times A} \tag{3}$$

where S is the distance between two electrodes, A is the projected area of the electrodes, and R is the resistance of sample.

The effects of the 8YSZ-N layer on the contact resistance of the half cells were investigated by an electrochemical impedance spectrometer (EIS, ZAHNER IM6eX, Germany). The applied frequency range was from 100 mHz to 1 MHz, and the voltage amplitude was 10 mV.

The cell test was conducted based on the document published by US Fuel Cell Council [11]. Some different procedures and the testing tools are described as below. The cell was sealed by a high-alumina ceramic paste (Sinetek Co., Ltd, Taiwan) on a 8YSZ zirconia tube to separate the fuel on anode side from the air on cathode side. The effective area of the cell was 3.28 ± 0.19 cm² and the current collection was going through the connection by Ag paste and wires. The flowing rate of H_2 fuel was 50 ml min⁻¹ with 3% H₂O (passing though water bath at 25 °C) at the anode side, and 250 ml.min⁻¹ air at the cathode side. Therefore, the ratio (λ) of the oxygen to fuel was 2.0. Before the test started to collect data, a reduction process holding at 600 °C for 1 hr was conducted and able to reduce NiO to Ni completely in the porous anode. The heating rate of the tests to testing temperature was 5 °C min⁻¹.

Results and Discussion

Thin electrolyte layer

Fig. 1(a) shows the particle size distributions of the 8YSZ-N and 8YSZ-T slurries ball-milling as long as 80 hr and attrition milling for another 6 hr. For the 8YSZ-T slurry, a milling time longer than 60 hr is effectively to reduce the average particle size of 1.0 µm to 0.2 μ m-0.3 μ m. The medium particle size (D₅₀) of the 8YSZ-N powder ball-milled for 1.5 hr is 0.3-0.4 µm, but additional attrition for 230 min results in a minimal size ca. 60 nm. For longer milling, the size (D_{50}) slightly increases to 65 nm, implying an increase of agglomeration of the particles. Therefore, the slurries for the coating test were made by the milling for 60 hr (8YSZ-T) and 230 min (8YSZ-N), respectively.

The slurry was diluted to different solid loadings, and spin-coated on the anode substrate at a rate of 3000 rpm, and sintered at 1400 °C for 1 hr. Fig. 1(b) shows the thickness of sintered 8YSZ-N electrolyte and how it is controlled by the solid loading (*C*) of the 8YSZ-N slurry. Additional layers of the 8YSZ-T are also coated on the anode substrate with and without the pre-coating 8YSZ-N layer. The thickness results are shown in Fig. 1(c). The film thickness shows a linear relationship to the



(c)

Fig. 1. (a) Average particle size of 8YSZ-N (N) and 8YSZ-T (T) slurries milling for different periods; Sintered thickness of (b) sintered 8YSZ-N layer on the anode co-sintered at 1400 °C for 1 hr varied with the slurry solid-loading, (c) sintered electrolyte thickness with and without a layer of the 8YSZ-N layer plotted against different spin-cycles using 1 wt% 8YSZ-T slurry.

slurry concentration and the number of the coating cycles, which confirm the relationship, as shown in Eq. (2).



Fig. 2. (a) SEM images and (b) scanning surface morphology of a typical anode surface sintered at 1000 °C for 1 hr, (c) SEM and (d) scanning surface morphology of one layer of an 8YSZ-N flat layer on the anode sintered at 1400 °C for 1 hr.

(a) 1000 °C pre-sintered anode



(b) Half cell: after slurry-added



Fig. 3. Schematic diagrams showing the states of spin coating. (a) A porous anode with surface roughness and porosity, and (b) a drop of the slurry been adsorbed on the anode forming a thin layer of particle cake, and draining the water (the route is indicated by the arrows) as the anode is spinned.

After the spin-coating of a thin 8YSZ layer on the anode substrate, the surface of the 8YSZ-N layer

becomes much smoother than that of the original anode surface. The initial anode surface (Figs. 2(a) and 2(b)) shows some residual pores that are caused by burn-out of the meso-carbon particles, which are used as a pore-former. The typical surface after the coating process reveals that the pores are filled by the thin layer 8YSZ-N and hardly visible, as shown in Fig. 2(c). The anode with the 8YSZ-N layer (Figs. 2(c) and (d)) shows a surface roughness (R_a) of 6.9 µm, reducing from the original 11.6 µm in Fig. 2(b). The 8YSZ-N layer shows the advantage of surface flattening of the electrolyte.

Schematic diagrams in Fig. 3 illustrates the formation sequence of a thin layer formed on the porous anode. The porosity (p) of the anode is 44% which is the major driving force to the water draining from the slurry, then instantly forms a thin layer of the cake (a thickness of h(C)) on the top of the anode (Fig. 3(b)). The instant thickness h(C) of the cake is controlled by the amount of the slurry adsorbed into the porosity. Therefore, the relationship is formulated as below.

$$h(C) = \frac{0.03pC}{\rho_z \rho_c} \tag{4}$$

where p is area of the cake (cm²), ρ_c is the relative density (0.56) of the cake, and ρ_z is the density of 8YSZ (6.02 gcm⁻³). If the solid concentration of the slurry *C* is 0.2 wt%, then the expected h (0.2 wt%) is 0.12 µm, which is far less than the data (1.0 ± 0.2 µm) shown in Fig. 1(b). Therefore, the anode with a thin nano-8YSZ layer should be treated as a nearly dense



Fig. 4. SEM images of (a) surface morphology of 8YSZ-N spin-coated for 3 cycles and sintered at 1400 °C for 1 hr, and (b) surface morphology of an 8YSZ-T layer spin-coated for 2 cycles on the top of one 8YSZ-N flat layer and (c) the cross-section of (b).



Fig. 5. (a-d) EIS spectra of the half cell (anode / electrolyte) with and without a flat layer (abb. as "flat layer") measured from 600 °C to 750 °C; (e) Proposed equivalent circuit model (R+RC+RC) of the electrochemical property of the half-cell (electrolyte/anode).

substrate, and the spin-coating of the layer can be formulated in consideration of Eqs. (1) and (2), shown

as below

$$h(C) = 1 + C \left(\frac{\eta \omega h_0}{\rho_L}\right)^{1/2}$$
(5)

The last term $\left(\left(\frac{\eta \omega h_0}{\rho_L}\right)^{\nu_2}\right)$ is the slope of the line in Fig. 1(b).

Too much cycles of the 8YSZ-N coating is not favored. Thick 8YSZ-N layer is difficult to dry without cracks. Surface cracking after densification at 1400 °C is easily formed for three-layer 8YSZ-N samples, as shown in Fig. 4(a). These fine cracks possibly cause some degree of fuel leakage. Therefore, one layer of the 8YSZ-N with an additional 8YSZ-T to form the electrolyte layer are used to assure the densification and free leakage of the electrolyte layers with the same thickness in the cells. The results are shown in Fig. 4(b). No surface cracks and pores are found in this case. The cross-sectional microstructure shown in Fig. 4(c) also indicates the intimate contact between the anode/electrolyte layers. The contact condition between the anode and the electrolyte is perfect, and no marks are found between the 8YSZ-N and 8YSZ-T layers after 1400 °C sintering.

Interfacial resistance

The EIS spectra of the half cells (anode/electrolyte) are shown in Fig. 5. The equivalent circuit model is shown in Fig. 5(e). The Ohmic loss (R_0) corresponds to the combination of Ohmic loss of Ni+YSZ anode (R_{0^{-} anode}), electrolyte layer ($R_{0^{-}YSZ}$) and the resistance of the interface of anode/electrolyte. On the other hand, the polarization resistances (R_{p1} and R_{p2}) include the interfacial polarization due to the transport of oxygen vacancies and point defects [9]. The results tested at 600 °C and 650 °C are distinguished into two semicircles, which correspond to the equivalent circuit model shown in Fig. 5(e). However, when the testing temperature arises to 700 °C or higher, the semi-circles at high frequency disappears, only matched to an equivalent circuit of ($R_0+R_{p2}C_2$).

According to our previous work [12], the semicircle at high frequency R_{p1} was interpreted as a grain-boundary impedance, while the semicircle at low frequency R_{p2} was a grain impedance. As the testing temperature increased, R_{p1} and R_{p2} first reduced, then R_{p1} disappeared at \geq 700 °C, and the grain impedance (R_{p2}) dominated the electrical conductivity. The values of the R_0 and two R_p are analyzed and summarized in Table 1.

Arrhenius plot of the Ohmic resistance (R_0) is shown in Fig. 6. The activation energies of the half-cell either with or without the 8YSZ-N layer are not significantly different, $61.2 \pm 3.5 \text{ kJmol}^{-1}$ and $59.7 \pm 3.2 \text{ kJmol}^{-1}$, respectively. The results illustrated the R_0 decreases ca. 20% if using the 8YSZ-N layer based on the same electrolyte material and similar thickness. According to SEM microstructural analysis (Fig. 4(c)), the thickness of both samples is in the same range $5.4 \pm 0.2 \mu m$. Therefore, the R_0 is reduced very likely due to a good adhesion of the 8YSZ-N flat layer to the porous anode surface.

Table 1. Ohmic resistance and polarization resistance of half-cell with and without flat layer measured from $600 \text{ }^{\circ}\text{C}$ to $750 \text{ }^{\circ}\text{C}$.

| | | 600 °C | 650 °C | 700 °C | 750 °C |
|---------------------------|---------------------------------------|--------|--------|--------|--------|
| With a 8YSZ-N layer | R ₀ (ohm.cm ²) | 20.4 | 15.8 | 10.3 | 6.6 |
| | $R_{p1}(ohm.cm^2)$ | 10.8 | 1.2 | | |
| | R_{p2} (ohm.cm ²) | 53.8 | 17.3 | 4.9 | 2.1 |
| without the layer | R ₀ (ohm.cm ²) | 25.6 | 19.2 | 12.3 | 8.1 |
| | $R_{p1}(ohm.cm^2)$ | 13.7 | 1.8 | | |
| | $R_{p2}(ohm.cm^2)$ | 82.6 | 26.6 | 5.9 | 2.1 |



Fig. 6. Arrhenius plot of Ohmic loss (R_o) for the half-cell (anode and electrolyte) with or without the 8YSZ-N layer (flat layer) measured from 600 °C to 750 °C.

Multiple layer structure

According to Ding and his co-workers' report [13], an ideal electrolyte thickness is between 5 μ m to 10 μ m. The results shown in Table 2 illustrate that the samples by 5 wt% 8YSZ slurry for 2 cycles (5W-2) or 1 wt% slurry for 6 cycles (1W-6) treatments are fabricated to have the electrolyte of a desired thickness of 5.4 μ m or 5.0 μ m, in a standard deviation of 0.2 μ m and 0.18 μ m, respectively (Table 2). Both half cells show no leakage. Because the 1w-6 (6 cycles) process is relatively timeconsuming, optimal preparation steps for the electrolyte used in the following sections was practiced: preparing 1

Table 2. Electrolyte film thickness and leakage test results prepared by different solid loading and cycles of the spin-coating. The half cells were sintered at 1400 °C for 1 hr.

| | Leakage test | Thickness (µm) | |
|--|-----------------|----------------|----------------------------|
| Spinning conditions using 8YSZ-T slurry | | Average | Standard Devia- tion |
| 5.0 wt% spinned for 2 cycles (5W-2) | Watertight | 5.4 | 0.2 |
| 1.0 wt% spinned for 3 cycles (1W-3) | Leak | 3.1 | 0.2 |
| 1.0 wt% spinned for 6 cycles (1W-6) | Watertight | 5.0 | 0.2 |



Fig. 7. SEM images of one cell. (a) a cross section of one cell; (b) top view of the cathode spin-coating for 1 cycle and sintered at 950 °C for 1 hr, and (c) porous Ni+8YSZ-T anode surface after H_2 reduction and I-V test.

layer of 0.2 wt% 8YSZ-N slurry, then 2 cycles of 5.0 wt% 8YSZ-T slurry on a 1000 °C pre-sintered anode substrate, then put to co-sintering at 1400 °C for 1 hr.

The cross-section and surface morphology of a complete cell (from top, cathod/electrolyte/anode) cell are shown in Fig. 7. The average thickness and standred deviation of the cathode are $9.4 \pm 0.7 \mu m$ after spin-coating for 1 cycle. As concerning the electrochemical reaction and current collection of a cathode layer [1], Ivers-Tiffee *et al.* suggested a desired cathode film in the range of 8-15 μm to be appropriate for current collection. Therefore, such a cathode film was produced in this study, by the conditions of spinning rate at 1000 rpm for 20 s and 1 cycle.

I-V test and fuel utilization of the cell

An anode-support, planar SOFC cell appears the thickness of each layer is $300 \ \mu\text{m}$ (anode), $5.4 \ \mu\text{m}$ (electrolyte) and $9.4 \ \mu\text{m}$ (cathode), respectively. The anode after H₂ reduction at 600 °C for 1 hr is porous (Fig. 7(c)). Plenty of pores in sizes of ca. 10 μm to less



Fig. 8. Cell voltage output (OCV) and power density plotted against current density of the cell operated at specified temperature using a fuel of 3% water humidified H_2 in anode side at a flow rate of 50 ml.min⁻¹ and air in cathode side at a flow rate of 250 ml.min⁻¹.

than 1 μ m is noted. The amount of the anode porosity after H₂ reduction is about 36.6% analyzed by the image analyzing program (Image J). This is suitable for fuel channel of the anode of SOFCs.

Fig. 8 shows the power density and I-V curve of the SOFC cell tested between 600 °C-800 °C. The open circuit voltage (OCV) is 0.95 V and the maximum power output (P_{max}) 119 mW.cm⁻² at 800 °C by using humidified H₂ as a fuel. The performance of 0.95 V is inferior to our previous study [14] on similar cell consisted of Ni+8YSZ/8YSZ/La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-d}+CeO₂ with OCV 1.1 V and P_{max} equal to 362 mW·cm⁻². The reasons have been investigated and show the interaction of cathode/8YSZ and form m-phase ZrO₂, of which the grains always associated with nano-sized flaws and induce fuel slightly cross-over. The detail study and the improvements for this case will be reported in our next paper.

Conclusions

An 8YSZ-N slurry of using nano-sized particles was prepared and spin-coated on NiO+8YSZ-T anode substrate as a flat layer to improve the contact condition between anode and electrolyte. The thin and dense YSZ electrolyte layer in a tight control of the thickness 1.0 μ m improves the surface roughness and resistivity properties of the cells. EIS analysis revealed the addition of the 8YSZ-N layer decreased the interfacial resistance (R₀) of the half cells for 20%.

Thin and high-quality electrolyte layer for the IT-cells was fabricated by 1 cycle of 0.2 wt% 8YSZ-N slurry and 2 cycles of 5.0 wt% 8YSZ-T slurry on NiO+8YSZ anode substrate, and co-sintered at 1400 °C for 1 hr. The half-cell was spin-coated a layer of $(Bi_{0.7},Sr_{0.3})(Mn_{0.5},Fe_{0.5})O_{3-8}$ cathode. The cell with anode/electrolyte/cathode in the thickness of 300 µm, 5.4±0.2 µm, and 9.4±0.2 µm can be achieved through the spin-coating method. The cell test showed an open circuit voltage (OCV) of 0.95 V and a maximum power output of 119 mW.cm⁻² at 800 °C.

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