Enhanced Gas Diffusion in Reversible Solid Oxide Cell Fabricated by Phase-Inversion Tape Casting

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> A hydrogen-electrode-supported solid oxide cell (SOC) is fabricated by phase-inversion tape casting technique and its electrochemical performance is investigated with particular focus on high fuel utilization operation and reversible operation. Structure of the hydrogen-electrode support layer is also analyzed. Electrochemical performance of the cell is measured and compared with that obtained from a conventional cell having homogenous porous structure in the hydrogen electrode support. Equivalent circuit fitting analysis is performed for quantitative understanding of impedance spectra. The results indicate that the finger-like pores in the hydrogen-support layer introduced by the phase-inversion process can significantly enhance gas diffusion property of the layer and hence improve cell performance particularly at lower reactant concentrations. Also, asymmetry in cell performance between fuel cell and electrolysis cell modes is suppressed, which is desirable for reversible operation of SOCs.

Introduction

Solid oxide fuel cells (SOFCs) are promising energy conversion devices owing to their high energy conversion efficiency and low emission. However, further improvement in efficiency and cost reduction are still required for their larger-scale introduction into the competitive energy market. Fuel utilization factor, which is defined as the ratio of the amount of fuel consumed to generate electricity to that of the supplied fuel, is among the most important factors that determine the energy conversion efficiency of fuel cell systems. When the cells are operated at high fuel utilization factor, hydrogen in the downstream of the fuel channel is significantly diluted. In addition, because of the concentration gradient formed in the thickness direction within the anode substrate, hydrogen starvation tends to occur at the electrochemical reaction sites near the anodeelectrolyte interface, resulting in drastic increase in the concentration overpotential. Although the hydrogen dilution in the fuel channel is inevitable in operations at high fuel utilization, hydrogen starvation near the anode-electrolyte interface can be avoided by improving gas transport within the anode substrate. Currently, pore formers, such as carbon black and polymer beads, are commonly used to introduce relatively larger pores in the anode substrates to enhance gas diffusivity (1, 2). However, design-based optimization of porous structure is required to further improve gas transport, and hence realize operation at higher fuel utilization.

In addition to electric power generation, SOFCs are potentially operated reversibly to produce hydrogen from water by applying external voltage. In this case they are called solid oxide cells (SOCs). SOCs are recently attracting attention for production of socalled green hydrogen by using renewable energy sources (3). Since SOC are operated in a reversible manner between fuel cell (FC) and electrolysis cell (EC) modes, cost reduction can be realized compared with the cases where power generation and electrolysis are performed in different devices. Reversible operation also expands the application of SOCs to energy storage applications (4-6). However, characteristics of SOCs in FC and EC modes are often asymmetric, meaning that the dependence of overpotential on a current in FC and EC modes is different (7-11). As a result, an SOC showing high power generation performance in FC mode does not always show sufficient electrolysis performance in EC mode, and vice versa. Although physicochemical origin of asymmetric behavior is still unclear, gas diffusion in hydrogen electrode is considered to be one of the important factors (9, 11). Therefore, improving gas transport in hydrogen electrode is also desired for reversible operation of SOCs.

For the fabrication of SOC electrodes, the phase-inversion tape casting technique can be employed to introduce anisotropic pore structures and thereby enhance gas transport properties of the electrodes. Although the phase-inversion technique was originally developed for fabrication of polymer membrane for desalination (12), its application can also be found in the fabrication of porous electrodes for SOCs (13, 14). In the phaseinversion technique, polymer solution consisting of polymer and solvent materials is first tape-casted to form a thin film, and then immersed into water to trigger phase inversion. The solvent is dissolved into water and the remaining polymer forms a film having finger-like pores growing from the immersed surface towards the bottom. The area other than the finger-like pores is occupied by relatively dense microscale sponge-like pores. Although the anisotropic finger-like pores formed by the phase-inversion technique was confirmed to be effective to enhance gas transport within the hydrogen electrode substrates (15, 16), few studies tested the cells at high fuel utilization conditions and in electrolyzer operations. We previously fabricated SOCs using the phase-inversion tape casting and reported enhanced gas diffusion property in the hydrogen electrode (17). However, the fabrication process was not matured enough, resulting in warpage in the fabricated cells during sintering. Also, electrochemical performance in EC mode was not characterized.

In this study, therefore, SOCs are fabricated using the phase-inversion tape casting technique and their electrochemical performance is analyzed at high fuel utilization conditions and in electrolyzer operation. The fabrication process of the Ni-YSZ hydrogen electrode is improved to obtain cells without significant warpage. Their electrochemical performance is evaluated by the electrochemical impedance spectroscopy and compared with that of cells fabricated by the conventional tape casting technique.

Experimental

Cell Fabrication

The hydrogen electrode substrate was fabricated by phase-inversion tape casting. NiO-YSZ powder (Ni:YSZ = 50:50 vol.%) was first prepared by ball-milling NiO

powder (FUJIFILM Wako Pure Chemical Industries, Ltd., Japan) and YSZ powder (TZ-8Y, Tosoh Corp., Japan) with isopropanol and zirconia balls (ϕ 4.0mm) at 300 rpm for 3 h. After evaporating isopropanol, the resultant mixture powder was sieved with 53 µm mesh. Subsequently, polymer solution was prepared by mixing polyethersulfone (PESF, Yamaso Co., Ltd., Japan) as binder, dimethyl sulfoxide (DMSO, Nacalai Tesque, Ink., Japan) as solvent and polyvinylpyrrolidone (PVP, Nacalai Tesque, Ink., Japan) as dispersant using hot stirrer at 60°C. The NiO-YSZ powder was added to the polymer solution and mixed by planetary ball milling at 200 rpm for 48 h to obtain uniform slurry. The composition of the slurry is shown in TABLE I. The prepared slurry was tape-casted on a glass plate with a blade gap of 1 mm and casting speed of 20 mm/s. Subsequently, the casted slurry was immersed into water of approximately 2 L for 12 h to initiate phase inversion. The solidified green sheet was dried at 20°C and 80%rh. The dried green sheet was cut into 30 mm diameter and pre-sintered at 1150°C for 2 h with heating rate of 120°C/h and cooling rate of 200°C/h. A porous zirconia plate with the size of 5x5 cm² and 2 mm in thickness was placed on the green sheet to avoid warpage during sintering.

DMSOPESFPVPNiO-YSZwt.%29.94.20.565.4

TABLE I. Composition of hydrogen electrode paste for phase-inversion tape casting.

On top of the fabricated support layer, hydrogen-electrode function layer was fabricated by spin coating. NiO-YSZ mixture powder was first prepared by mixing NiO powder (Sumitomo Metal Mining Co. Ltd, Japan) and YSZ powder (Ni:YSZ = 50:50 vol.%) using the same procedure described above, and then mixed with terpineol (Nacalai Tesque Inc., Japan) and ethylcellulose (Nacalai Tesque Inc., Japan) using planetary ball milling with zirconia balls (ϕ 10mm) at 500 rpm for 12 h to form homogeneous slurry. The composition of the slurry was NiO-YSZ:terpineol:ethylcellulose = 50:48:2 in weight. The obtained slurry was dropped onto the substrate and immediately spun at 8000 rpm for 1 min. Subsequently, it was dried at 200°C or 10 s.

A YSZ electrolyte was prepared on the function layer by spin coating. A YSZ suspension was prepared by mixing YSZ powder, terpineol, and ethyl cellulose (YSZ:terpineol:ethylcellulose = 25:72:3 in weight) by planetary ball milling to form uniform slurry. The obtained slurry was dropped onto the substrate and immediately spun at 8000 rpm for 1 min. Subsequently, the cell was co-sintered at 1400° C for 5 h with heating rate of 120° C/h and cooling rate of 200° C/h. A GDC (Ce_{0.9}Gd_{0.1}O_{1.95}) barrier layer was also fabricated by spin coating. GDC powder (GDC-10, Shin-Etsu Astech Co. Ltd., Japan), terpineol, and ethylcellulose were mixed (GDC:terpineol:ethylcellulose = 50:48:2 in weight) by planetary ball milling to form uniform slurry. The obtained slurry was dropped on the YSZ electrolyte and immediately spun at 8000 rpm for 1 min. Subsequently, it was sintered at 1250° C for 2 h with heating and cooling rate of 200° C/h.

Finally, an LSCF (La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3- δ}, Kceracell Co. Ltd., Korea) oxygen electrode were fabricated by screen printing. LSCF powder was mixed with an ink vehicle (VEH, Nexceris LLC, Ltd., U.S.A.) at LSCF:VEH = 70:30 in weight using ball milling. The resultant slurry was screen-printed on the GDC barrier layer with a mask thickness of 60 µm and then sintered at 950°C for 5 h with heating and cooling rate of 200°C/h. The apparent geometrical area of the oxygen electrode was ca. 0.785 cm².

In addition, another cell was fabricated using conventional tape-casting for comparison. First, pore former (Polymethyl methacrylate, SSX-108, Sekisui Plastic Co., Ltd., Japan) was added to the NiO-YSZ powder used for the phase-inversion tape-casting and mixed by planetary ball milling with ethanol, Triton X-100 (MP Biomedicals LLC, U.S.A.) and zirconia balls (ϕ 4.0mm) at 200 rpm for 24 h. Subsequently, Polyvinylbutyral (PVB, Mowital B60H, Kuraray, Japan), PEG 400 (FUJIFILEM Wako Pure Chemical Co., Ltd., Japan), and Glycerin (Nacalai Tesque Inc., Japan) were added and ball milled for another 24 h. Composition of the slurry is shown in TABLE II. The obtained slurry was degassed at 0.2 atm for 10 min, and casted on a glass plate with a blade gap of 2 mm and casting speed of 10 mm/s. The casted slurry was dried at 20°C and 80%rh. The dried green sheet was cut into 30 mm diameter and pre-sintered at 1150°C for 2 h with heating rate of 120°C/h and cooling rate of 200°C/h. A porous zirconia plate was placed on the green sheet to avoid warpage during sintering. Fabrication procedure for the other layers were the same as those for the phase-inversion cell.

TABLE II. Composition of paste for conventional tape casting.

| | 1 | 1 | | 1 | 0 | | |
|------|---------|--------------|------|---------------|----------|---------|-------------|
| | Ethanol | Triton X-100 | PVB | PEG400 | Glycerin | NiO-YSZ | Pore former |
| wt.% | 33.8 | 1.06 | 6.36 | 2.89 | 2.89 | 47.7 | 5.30 |

Electrochemical Characterization

Electrochemical performance of the cells was measured using an SOFC button cell evaluation system (Microtrac MRB, Japan). The cells were held in an electronic furnace using alumina tubes, through which hydrogen and air were supplied to the hydrogen electrode and oxygen electrode, respectively, using mass flow controllers. Platinum mesh was used for current collection for both electrodes and platinum wire for wiring. More details of the evaluation system can be found elsewhere (18). Electrochemical characteristics of the cell was analyzed using Solartron 1470E potentio/galvanostat and Solartron 1455A frequency response analyzer (Solatron Analytical, U.K.). For measurement of current-voltage characteristics, terminal voltage of the cell was varied from 1.3 to 0.4 V with an interval of 5 mV. For the impedance analysis, sinusoidal current was applied to the cell with a frequency range from 10^6 to 10^{-1} Hz with an amplitude of 10 mA. Gas composition on the hydrogen electrode side was $H_2:H_2O:N_2 =$ x:x:100-2x (x=10, 20, 30) so that the hydrogen and steam utilization factors become identical when the current densities in FC and EC operation modes are the same. The synthetic dry air was supplied to the cathode. The total flow rate was fixed at 100 mL/min for both electrodes. The cell temperature was maintained at 700°C by an electric furnace.

Equivalent circuit fitting was conducted to quantitatively understand the impedance spectra obtained in this study. The equivalent circuit as shown in Figure 1 was used, which consists of an inductor, a resistor, and two R-CPE (constant-phase element) parallel circuits. Each parallel circuit represents the activation resistance and gas diffusion resistance. Note that the main focus of this study was on the gas transport property within the cell, and hence the activation resistances in the hydrogen electrode and that of the oxygen electrode were not separated in this analysis.



Figure 1. Equivalent circuit for impedance analysis.

Microstructure Characterization

After the electrochemical measurements, microstructure of the hydrogen-electrode was analyzed. First, the porous microstructure of the sponge-like pore in the phase-inversion cell and that of the function layer, which is common in the conventional and phase-inversion cells, were analyzed in 3D using focused ion beam scanning electron microscope (FIB-SEM, NX-9000, Hitachi High-Tech). For the sample preparation, the tested cells were impregnated with epoxy resin (EpoFix, Struers, Denmark) for easier identification of the phases in the electrode. Several hundred images were obtained through the cut-and-see process and they were reconstructed into 3D structures, from which microstructural parameters were quantified. More details of the imaging and quantification procedures were found elsewhere (19, 20).

In addition, structure of the finger-like pores was analyzed by manual polishing and imaging using conventional scanning electron microscope (SEM, Miniscope TM3000, Hitachi Ltd., Japan). The impregnated cells were cut and polished to expose a cross-section parallel to the surface of the electrode. The cross-section was imaged using the SEM to analyze the diameter and the number density of the finger-like pores in the hydrogen electrode. By repeating the polishing and imaging, several cross-sectional images were obtained with an interval of ca. $60 \,\mu\text{m}$.

Results and Discussion

Microstructure

Figure 2 shows the cross-sectional SEM images of the hydrogen electrode support layer. The Cross-sectional images parallel to the electrode surface was taken with an interval of around 70 μ m. The immersed surface during the phase-inversion process corresponds to the interface between the support and function layers. It is clearly seen that finger-like pores grow from the immersed surface towards the bottom. Also, relatively dense skin layer is formed near the immersed surface. The diameter of the finger-like pores increases as they grow, whereas their number density decreases. Also, it is worth noting that there is no merging or branching of the finger-like pores stop growing within the support layer. This indicates that some of the finger-like pores stop growing within the support layer, and remaining pores grow towards the bottom with increasing their diameter. In addition, no finger-like pores penetrate through the layer, which leaves sponge-like region near the bottom of the support layer.



Figure 2. Cross-sectional SEM images of hydrogen electrode support layer fabricated by phase-inversion tape casting.

| Table III. Wherost detailed parameters of porous region | | | | | | | | | |
|---|----------|-----------------------|---------------|----------------------|--|--|--|--|--|
| | | Function layer | Support layer | | | | | | |
| | | | Conventional | Phase inversion | | | | | |
| | | | | (Sponge-like region) | | | | | |
| Porosity | [-] | 0.19 | 0.42 | 0.37 | | | | | |
| Average pore size | [µm] | 0.81 | 1.57 | 1.19 | | | | | |
| TPB density | [µm/µm³] | 3.44 | 1.97 | 2.91 | | | | | |

 Table III.
 Microstructural parameters of porous region

TABLE III shows the microstructural parameters quantified from the cells. The function layer is common in the two cells. Total volume analyzed was at least 2,400 μ m³ for all samples, which is considered to be large enough for the reconstructed structures to represent the structures of the analyzed region. Note that it was difficult to obtain a single large volume from the sponge-like region in the phase inversion cell because of the existence of finger-like pores in the support layer. Therefore, six regions with the volume of around 200–500 μ m³ were extracted and their microstructural parameters were averaged. It is found that porosity of the function layer is relatively small, resulting in small average pore size and larger TPB density. On the other hand, porosities of the support layers are similar and around 40%, indicating that the gas diffusion pathways are likely to be formed (21, 22). Although TPB density in the sponge-like pore region is larger than that in the conventional substrate, total TPB length in the phase-inversion substrate is considered to be comparable to that in the conventional substrate considering the fact that the phase-inversion substrate also has hollow finger-like pores within the substrate.

Figure 3 shows the quantitative information of the finger-like pores. Note that the y axis corresponds to the distance from the immersed surface, and x axis corresponds to the structural parameters. As observed in the SEM analysis, the average diameter of the finger-like pores increases towards the bottom, whereas their number density decreases. In addition, the total porosity of the support layer, including the area fraction of the finger-like pores and the porosity of the sponge-like region, is almost irrespective of the distance from the immersed surface. The value of the total porosity is close to that of the conventional hydrogen electrode support layer fabricated by tape-casting, meaning that mechanical strength of the phase-inversion substrate is comparable to that of the conventional substrate.



Figure 3. Structural parameters of hydrogen electrode support layer fabricated by phase-inversion tape casting.

Electrochemical Performance

Figure 4 shows the current-voltage characteristics of the cells at 700°C with various gas compositions on the hydrogen electrode. The open-circuit voltage was confirmed to be close to the theoretical values estimated from the Nernst equation, which indicates that the gas sealing using glass rings was sufficient and that dense electrolyte was successfully formed within the cell. This was realized because warpage of the cells was suppressed by using zirconia plate during sintering. It is found from Figure 4 that the overpotential, defined as the potential difference from the open-circuit voltage, increases as the gas concentration decreases. Also, the overpotentials are always larger in the EC mode, indicating asymmetric behavior of the cell. Although performance difference between the two cells are not apparent, the phase-inversion cell shows slightly better performance and the difference is more visible in lower gas concentration conditions.

Figure 5 shows the impedance characteristics of the cells at biased conditions in both FC (0.1 A/cm^2) and EC (-0.1 A/cm^2) modes. In the biased conditions, similar trends as in Figure 4 are observed; the total polarization resistance is slightly smaller in the phase-inversion cell, and the difference is more apparent in lower gas concentration. Also, the polarization resistance is larger in the EC mode.



Figure 4. Current-voltage characteristics at 700°C. (a) conventional cell and (b) phase-inversion cell.



Figure 5. Electrochemical impedance characteristics at 700°C. (a) FC mode at 0.1 A/cm² and (b) EC mode at -0.1 A/cm².

To understand the obtained impedance spectra more quantitatively, equivalent circuit fitting analysis was performed to separate the internal resistances. Figure 6 and 7 respectively show the activation and diffusion resistances in the cells as a function of the molar fraction of hydrogen and steam on the hydrogen electrode. Note that the inductance and series resistance components in Figure 1 are found to be irrespective of the operation conditions, therefore, they are omitted from this manuscript.

As shown in Figure 6, the activation resistance increases when the gas concentration is lower, and is always larger in the EC mode. In addition, the difference between the FC and EC modes increases in higher current densities. These trends are common in the two cells fabricated in this study. The absolute values of the activation resistance are slightly larger in the phase-inversion cell, and its reason is not clear at this moment. Since the function layer in the hydrogen electrode was fabricated identically and its thickness (~35 μ m) is considered to be able to cover the active reaction region (23). Therefore, the inhomogeneous porous structure in the support layer of the phase-inversion cell may have an influence on the electrochemical reaction in the function layer; this will be our future study.



Figure 6. Activation resistance at 700°C. (a) conventional cell and (b) phase-inversion cell.



Figure 7. Diffusion resistance at 700°C. (a) conventional cell and (b) phase-inversion cell.

The diffusion resistance shown in Figure 7 shows more remarkable difference between the two cells. In the conventional cell, the diffusion resistance increases in lower gas concentrations and also asymmetry exists between the FC and EC modes. On the other hand, in the phase-inversion cell, the diffusion resistance is significantly smaller than that in the conventional cell. In addition, its value is irrespective of the operation mode and bias current. This indicates that the cell fabricated by the phase-inversion tape casting technique is suitable for high fuel utilization operation of SOCs, and also for reversible operation.

Conclusions

Hydrogen-electrode-supported SOC was fabricated by phase-inversion tape casting technique and its electrochemical performance was investigated with particular focus on high fuel utilization operation and reversible operation. Fabrication process of the layers consisting of the cell was established to achieve cells without warpage and electrolyte pinholes. Structure of the hydrogen electrode was also analyzed in detail. The structural analysis reveals that the finger-like pores start to grow from the immersed surface and grow towards the bottom, with expanding their diameter and decreasing their number density. The electrochemical performance of the cell was measured and compared with that obtained from the conventional cell having homogenous porous structure in the hydrogen electrode support layer. From the current-voltage characteristics, the phaseinversion cell has slightly better performance particularly at lower gas concentration. Asymmetric behavior was observed in both cells between FC and EC operations. Similar trends were also observed in the impedance characteristics. From the equivalent circuit fitting analysis, the activation overpotential show similar trends in both cells. The resistance increases in lower gas concentration and higher current density, and asymmetry exists between the FC and EC modes. Remarkable difference was observed in the diffusion resistance. In the conventional cell, the diffusion resistance increases in lower gas concentration and higher current, and shows asymmetric behavior. On the other hand, in the phase-inversion cell, the diffusion resistance is almost independent of the gas concentration and operation modes, and always smaller than that in the conventional cell. This indicates that the cell fabricated by the phase-inversion tape casting is suitable for high fuel utilization operation and reversible operation of SOCs and hence useful to expand applicability of SOC systems.

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