Metal-supported microtubular solid oxide fuel cells with ceria-based electrolytes

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Metal-supported solid oxide fuel cells (SOFCs) are expected to lower operating temperature and improve reliability. The residual crushing strength and electrical conductivity of porous metallic nickel microtubes were higher than those of nickel-gadolinia-doped ceria (Ni-GDC) cermet. The maximum power density of the metal-supported microtubular SOFC with GDC electrolyte was approximately three times higher than that of a conventional anode-supported microtubular SOFC with yttria-stabilized zirconia electrolyte at a relatively low temperature of 550°C. The metal-supported microtubular SOFCs have a potential to be applied not only in stationary devices, such as residential combined heat/power systems, but also in portable power sources and vehicles.

Key-words : Solid oxide fuel cell (SOFC), Microtube, Metal-supported cell, AC impedance, Distribution of relaxation times (DRT)

1. Introduction

Solid oxide fuel cells (SOFCs) are expected to be power generation systems with high energy conversion efficiency. SOFCs, which mainly consist of ceramics, can design electrolyte-supported cells (ESCs) and anode-supported cells (ASCs). The ESCs use an electrolyte substrate with 0.1–1 mm in thickness to obtain mechanical support for thin layers of anode and cathode. Bloom Energy is designing 200 kW-class stationary power generation systems by using ESCs with scandia-stabilized zirconia (ScSZ) substrates made by Nippon Shokubai.1) The ionic conductivities of yttria-stabilized zirconia (YSZ) and ScSZ show Arrhenius dependence on temperature.2) Generally, the ESCs are operated at high temperatures of 850–1000°C. However, the ESCs are operated at temperatures lower than that of ESCs.

Recently, two approaches have been proposed to lower the operating temperature of SOFCs to 600–800°C. One approach is to use new electrolyte materials that have a high ionic conductivity at low temperature, such as lanthanum gallate3) and Gd-doped ceria (GDC),4) for ESCs. Ishihara et al.5) obtained the maximum power density of 1.53 W/cm² at 800°C for ESCs with La0.8Sr0.2Gd0.8Mg0.115Co0.085O3 electrolyte. However, the mechanical strengths of lanthanum gallate and GDC are lower than those of YSZ and ScSZ.6,7) The other approach is to thin down the YSZ electrolyte to 5–20 μm on the anode thick substrate. Kyocera has successfully commercialized residential combined heat/power systems using flatten-tubular ASCs with YSZ electrolyte since 2011,8) and the other manufacturers are also developing SOFC systems using planar and tubular ASCs.9–12)

However, the huge compressive stress of approximately 650 MPa was observed in the electrolyte for ASCs by X-ray stress measurement at room temperature,13) which is caused by the mismatch of coefficients of thermal expansion between dense YSZ thin-film (10 × 10⁻⁶ K⁻¹)14) and porous Ni-YSZ substrate (12 × 10⁻⁶ K⁻¹).15) Furthermore, the internal stress is variable during thermal and reduction–oxidation (redox) cycles.16) The tensile internal stress was observed in the electrolyte thin-film during re-oxidation cycle by in-situ X-ray stress measurement at SPring-8 BL02B1,17) which was caused by anomalous expansion of the NiO-YSZ anode substrate.18) The tensile stress sometimes produces microcracks and micropores in the electrolyte thin-film for ASCs.19–21) The reliability of ASCs is relatively lower than that of ESCs.

Metal-supported SOFCs (MSCs) use ceramic layers as thick as necessary for the electrochemical reaction. The porous metallic substrate has a function of mechanical support. Better mechanical robustness, redox tolerance, rapid thermal cycling and cost reduction are expected, when MSCs are employed.22) Imperial College London and Ceres Power developed MSCs with Ni-GDC anode, GDC electrolyte and (La, Sr)(Co, Fe)O₁₋ₓ₄ (LSFC)-GDC cathode thin-film on a ferritic stainless steel substrate, and the maximum power density of 300 mW/cm² was demonstrated at 600°C.23) The O₂⁻ ionic conductivity of GDC is higher than those of YSZ and ScSZ.24) However, electron is also conducted through GDC under low oxygen partial pressures.25) The current leakage causes the decrease in open circuit voltage (OCV)26) and power generation efficiency.27–29) Several researchers developed the MSCs with YSZ single layer30,31) or YSZ/GDC bi-layer32,33) electrolytes to prevent the current leakage. The solid solution of YSZ-GDC is formed at the interface between YSZ and GDC after sintering at high temperature above 1200°C.34) The solid solution has significantly lower conductivity compared with YSZ and GDC.5) Furthermore, the MSCs cannot be sintered at high temperatures, because the heat-resistivity of porous metallic substrates is lower than those of ceramic and cermet substrates. Electrophoretic deposition (EPD),35) electrochemical vapor deposition (EVD),36) and atmospheric plasma spray (APS)37) were employed to form dense electrolytes to prevent sintering at high temperatures.
The authors found that Y-doped barium cerate (BCY) had a function of blocking the electron conduction for GDC electrolyte under low oxygen partial pressures.\textsuperscript{39} The OCV increased by inserting the BCY blocking layer between GDC electrolyte and Ni-GDC anode for ASCs. Authors also developed anode-supported microtubular SOFCs for low operating temperatures.\textsuperscript{39-41} On the other hand, Park and Virker\textsuperscript{42} prepared the oxide substrate of Fe\textsubscript{2}O\textsubscript{3}-NiO composite. The NiO-GDC anode, GDC electrolyte and (La,Sr)CoO\textsubscript{3}–GDC cathode were formed on the oxide substrate by conventional ceramic manufacturing processes including high temperature sintering process. Then, the Fe–Ni metallic substrate was obtained by reduction treatment after the setup for power generation tests. In this work, the metal-supported microtubular SOFCs of Ni | Ni-GDC | BCY | GDC | LSCF-GDC were developed, and its electrochemical properties were compared with conventional ASCs’ ones by the distribution of relaxation times (DRT) analysis.\textsuperscript{40,41,43,44}

2. Experimental

Microtubular substrates were made from NiO (Sumitomo metal mining), (Ce\textsubscript{0.9}Gd\textsubscript{0.1})O\textsubscript{1.95} (GDC; Anan Kasei), pore former (acrylic resin; Sekisui Plastic) and binder (Cellulose; Yuken Kogyo) powders. The weight ratios of NiO to GDC were 6:4 and 10:0 for ASCs and MSCs, respectively. A pore former of acrylic resin with a grain size of approximately 5 \( \mu \)m was added before sintering to increase substrate porosity. These powders were mixed mechanically by kneading machine with addition of the proper amount of water for 2 h. The microtubes were extruded using a piston cylinder with a metal hole of 4 mm (outside diameter) and 2 mm (inside diameter). After extrusion, the tubes were dried overnight in air at room temperature. An anode slurry was prepared by mixing NiO, GDC, a binder (polyvinyl butyral; Sekisui Chemical) a dispersant (tallow propylene diamine; Kao) and a plasticizer (diocyl adipate; Wako Pure Chemical Industries) into ethanol and toluene solvents for 48 h. The NiO-GDC anode was formed by dip-coating on the NiO microtubular substrate for MSCs. The NiO anode was pre-sintered in air for 1 h at 800°C. BaCe\textsubscript{0.9}Y\textsubscript{0.1}O\textsubscript{3-\textdelta} (BCY) blocking layer was formed in the same manner for MSCs. After coating (Y\textsubscript{2}O\textsubscript{3})\textsubscript{0.00}Zr\textsubscript{2}O\textsubscript{1.92} (YSZ) or GDC electrolyte for ASCs or MSCs, respectively, the microtubes were sintered in air for 3 h at 1450°C. Then, the cathode of La\textsubscript{0.6}Sr\textsubscript{0.4}CoO\textsubscript{3} (LSCF)-GDC was sintered in air for 1 h at 950°C. The weight ratio of LSCF to GDC was 7:3. The outside diameter of microtube was 3 mm, and the cathode length was 10 mm after sintering. After the setup for power generation test, the mixture of 20%H\textsubscript{2}-80%N\textsubscript{2} was supplied to the inside of microtubular cells for 4 h at 550°C for reduction treatment of the Ni or Ni-GDC substrate and the Ni-GDC anode.

Figure 1 shows the cross-sectional scanning electron microscopic images of the ASC with YSZ electrolyte and MSC with GDC electrolyte after reduction treatment. The porosity of the anode and metal substrates before and after reduction treatment is shown in Table 1.

Radial crushing strengths of anode and metal substrates were evaluated by a method based on ISO-2739 “Determination of radial crushing strength for sintered metal bushings” in air at room temperature. Five microtubes with 10 mm in length after sintering were set in compression testing machine, and the radial crushing strength, S, was calculated from the following equation:

\[
S = \frac{F(D - t)}{Lt^2}
\]  

where \( F \) is the maximum load, \( D \) is the outside diameter, \( t \) is the thickness, and \( L \) is the length of the microtube. The electrical conductivity of the anode and metal substrates were measured by four-terminal method applying an AC voltage of 1 mV during the cooling procedure from 700°C to room temperature in 10%H\textsubscript{2}-90%N\textsubscript{2}. Electrochemical properties were measured with a potentiostat/galvanostat (Solartron Analytical 1470E) and an impedance analyzer (Solartron Analytical 1455). Silver wires and paste were used as a current collector.\textsuperscript{40} A mixtures of H\textsubscript{2}:H\textsubscript{2}O:N\textsubscript{2} = x:3.97-x (x = 5, 10, 20) were supplied as the fuel with a flow rate of 100 mL/min to the inside of microtubes, and air was supplied as the oxidant at 100 mL/min to the outside of microtubes. The operating temperature was 550°C. Current-voltage (i-V) characteristics were measured from OCV to 0.2 V at a sweep rate of 5mV/s. The AC impedance was measured at OCV in the frequency range from 1 MHz to 0.03 Hz with 20 steps per logarithmic decade. Impedance spectra are generally analyzed...
by the complex non-linear least squares (CNLS) method on the assumption of an equivalent circuit. The polarization impedance capacitance element (RC) has a single relaxation time \( \tau \) defined by the appropriate \( R \) and \( C \) values (\( \tau = RC \)). Generally, the relaxation response of electrochemical processes cannot be perfectly fitted by one RC when the relaxation time shows a distribution. The polarization resistances are fitted to resistance-constant phase element (R-CPE). However, it is sometimes difficult to assume an exact equivalent circuit for CNLS fitting, because several electrode processes generally overlap within the actual impedance spectra of SOFCs. In this study, the DRT related to the electrochemical processes were directly calculated from the experimental impedance spectra as similar to the previous papers. The polarization impedances \( Z_{\text{pol}}(\omega) \) are considered to be an equivalent circuit composed of an infinite number of RC elements in the following equations:

\[
Z_{\text{pol}}(\omega) = R_{\text{pol}} \int_0^\infty \frac{\gamma(\tau)}{1 + j\omega \tau} d\tau \quad \text{with} \quad \int_0^\infty \gamma(\tau) d\tau = 1
\]

(2)

Here, \( R_{\text{pol}} \) is the total DC polarization resistance, \( \gamma(\tau) \) is the DRT function, \( \omega \) is the angular frequency and \( j \) is the imaginary unit. If the real and imaginary parts of the impedance spectra are satisfied with the Kramers-Kronig relation, it is sufficient to consider only the imaginary part of the impedance.

\[
Z_{\text{pol}}(\omega) = -R_{\text{pol}} \int_0^\infty \frac{\omega \tau}{1 + (\omega \tau)^2} \gamma(\tau) d\tau
\]

(3)

Since the impedance spectra are measured logarithmically, the frequency variables have to be substituted as follows.

\[
Z_{\text{pol}}(\omega) = \frac{R_{\text{pol}}}{2} \int_{-\infty}^\infty \text{sech}(y \cdot g(y - x)) dy = \frac{R_{\text{pol}}}{2} \text{sech} x \cdot g(x)
\]

where \( y = \ln(\omega \tau), \ g(y - x) = \gamma(\tau) \tau \)

(4)

The distribution function \( g(x) \) in doubly Fourier transformed space was calculated using the software program FTIKREG to solve an ill-posed inverse problem by Tikhonov’s regularization method. Before DRT analysis, the inductance components were removed from the impedance spectra by the same method.

3. Results and discussion

First, the mechanical and electrical properties were compared between Ni(O) and NiO-GDC microtubular substrates. Figure 2 shows the radial crushing strength of the anode and metal substrates in air at room temperature before and after reduction treatment. The strength of NiO-GDC porous composite was \( 71 \pm 2 \) MPa. After the reduction treatment of Ni-GDC cermet, the strength slightly increased by 99\%.

Next, the electrochemical properties were compared between the anode-supported cell with YSZ electrolyte (ASC) and the metal-supported cell with GDC electrolyte (MSC). Figure 4 shows the current density–voltage and power density characteristics at 550°C. The maximum power densities were 22 and 29 mW/cm\(^2\) for ASC in 5\%H\(_2\) and 20\%H\(_2\), respectively. On the other hand, the OCV of more than 1 V was obtained in 20\%H\(_2\) for MSC with BCY blocking layer between GDC electrolyte and Ni-GDC anode. The power density was 82 W/cm\(^2\) in 20\%H\(_2\) for MSC, which was approximately 3 times higher than that of the Ni-GDC cermet. The electron conductive percolation in metallic Ni substrate was better than that in Ni-GDC cermet. In particular, metallic substrates have advantages of high mechanical strength and electrical conductivity over cermet for vehicle vibration.

Next, the electrochemical properties were compared between the anode-supported cell with YSZ electrolyte (ASC) and the metal-supported cell with GDC electrolyte (MSC). Figure 4 shows the current density–voltage and power density characteristics at 550°C. The maximum power densities were 22 and 29 mW/cm\(^2\) for ASC in 5\%H\(_2\) and 20\%H\(_2\), respectively. On the other hand, the OCV of more than 1 V was obtained in 20\%H\(_2\) for MSC with BCY blocking layer between GDC electrolyte and Ni-GDC anode. The power density was 82 W/cm\(^2\) in 20\%H\(_2\) for MSC, which was approximately 3 times higher than that of ASC. The lowering the operating temperature contributes to rapid start up and shut down for portable power sources. However, the dependence of hydrogen partial pressure (p\(_{H2}\)) in anode on the performance was large for MSC. The maximum power generation decreased to 47 mW/cm\(^2\) in 5\%H\(_2\) for MSC.
caused by anodic polarization. The peaks of analysis is shown in independent on magnitude higher than that of YSZ. The ohmic resistance was ing layer, because the ionic conductivity of GDC is an order of smaller than that of ASC in spite of the insertion of BCY block-

and MSC, respectively. The ohmic resistance of MSC was cathodic polarization. The ascribed to hydrogen oxidation process.40) The values and conduction process in the LSCF bulk and adsorption of the impedance arcs, were dependent on polarization resistances, which were derived from the diameters of the Nyquist plots of AC impedance spectra for the anode-supported cell with YSZ electrolyte (ASC) and the metal-supported cell with GDC electrolyte (MSC) at 550°C.

Figure 5 shows the Nyquist plots of AC impedance spectra for ASC and MSC at 550°C. The ohmic resistances, which were derived from the intersections of the impedance arcs at high frequency with the real axis, were 2.6 and 0.96 Ω cm² for ASC and MSC, respectively. The ohmic resistance of ASC was smaller than that of ASC in spite of the insertion of BCY blocking layer, because the ionic conductivity of GDC is an order of magnitude higher than that of YSZ. The ohmic resistance was independent on pH2 for both samples. On the other hand, the polarization resistances, which were derived from the diameters of the impedance arcs, were dependent on pH2 for both samples. The result of DRT analysis is shown in Fig. 6. The DRT peaks of P1\textsuperscript{a} (~1kHz) and P2\textsuperscript{a} (~20Hz) were unchanged with pH2, which were caused by cathodic polarization. The P1\textsuperscript{a} and P2\textsuperscript{a} were ascribed to the ionic conduction process in the LSCF bulk and adsorption/desorption process on the LSCF surface, respectively.41) The DRT peaks of P1\textsuperscript{a}, P2\textsuperscript{a}, P3\textsuperscript{a}, P4\textsuperscript{a}, and P6\textsuperscript{a} were changed with pH2, which were caused by anodic polarization. The peaks of P1\textsuperscript{a} (~1Hz) and P4\textsuperscript{a} (~0.2Hz) appeared only for ASC. At the interface between YSZ electrolyte and Ni-GDC anode, the solid solution of YSZ-GDC, which had large pseudo-capacitance due to the oxygen nonstoichiometric variation process,40) was formed. The pH2 dependence of P3\textsuperscript{a} (~5–10Hz),80) which was ascribed to the gas diffusion process in the anodes, was almost the same between ASC and MSC. The P3\textsuperscript{a} (~3kHz) and P2\textsuperscript{a} (~100Hz) were ascribed to hydrogen oxidation process.40) The values and pH2 dependence of P2\textsuperscript{a} for MSC were larger than that for ASC. The DRT spectra for the anode-supported cell with YSZ electrolyte (ASC) and the metal-supported cell with GDC electrolyte (MSC) at 550°C.

4. Conclusion

In this work, the metal-supported microtubular SOFCs with BCY blocking layer between GDC electrolyte and Ni-GDC anode on the Ni metallic substrate were investigated, and their electrochemical properties were compared with the conventional anode-supported SOFCs with YSZ electrolyte. The mechanical strength at room temperature and electrical conductivity at 550°C of metallic Ni substrate were approximately 3 times higher than those of Ni-GDC cermet. The performance of MSC was dependent on pH2, because the polarization resistance for hydrogen oxidation process increased due to insufficient thickness of the anode. Nevertheless, the maximum power density of MSC with GDC electrolyte was approximately 3 times higher than that of ASC with YSZ electrolyte in 20%H2 at 550°C. It is concluded that the MSCs with GDC electrolyte is expected to lower the operating temperature of SOFCs.

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