Effect of Ni-loading on Sm-doped CeO$_2$ anode for ammonia-fueled solid oxide fuel cell

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Samaria-doped ceria, (SmO$_{1.5}$)$_{0.2}$(CeO$_2$)$_{0.8}$ (SDC), containing nickel (Ni) was prepared as the anode of solid oxide fuel cell fueled with 6% ammonia (NH$_3$). The Ni-free SDC powders prepared by the reverse co-precipitation method exhibited poor catalytic activities for NH$_3$ decomposition in 6% NH$_3$/Ar. Impregnation of Ni onto the SDC powders significantly enhanced its catalytic activity. The catalytic activity was highest at 10 wt% Ni–SDC, but it decreased with an increase in the Ni content. Contrary to expectation, the anodic performances were similar between 10 and 40 wt% of Ni loading and the highest maximum power densities were 98.8 and 96.5 mW·cm$^{-2}$ at 900°C, respectively. Impedance analysis of the anodes revealed that the anodic performance was rate-controlled by the similar process in 4% H$_2$ fuel and that was electrochemical oxidation and diffusion processes.

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1. Introduction

Upgrading the infrastructure for hydrogen fuel is essential for spreading hydrogen power systems, especially fuel cells. The major drawbacks of hydrogen energy are its large energy consumption and its high cost for liquefaction, storage, and transport.$^{1,2}$ To overcome this, several highly portable hydrogen carriers have been proposed.$^3$ At present, methane, which is a primary component of natural gas, is widely used as a hydrogen carrier, especially in stationary fuel cell systems. Hydrogen is usually produced through the steam–methane reforming.$^{4,5}$ However, this process simultaneously yields carbon dioxide as a by-product.

Ammonia (NH$_3$) easily liquefies because of its high boiling point of −33.4°C at 1 atm, its ability to serve as a carbon-free fuel, and its high hydrogen density (17.8 mass %).$^6,7$ NH$_3$ is an important chemical for producing fertilizers and resins, and infrastructure for the storage and transport of NH$_3$ have already been established. Hence NH$_3$ is now considered as a new alternative hydrogen carrier. Till date, NH$_3$-reforming catalysts have been studied by many groups.$^{8-13}$ Ruthenium-based catalysts are very active for NH$_3$ decomposition, and its full conversion can be obtained below 500°C.$^{10}$ Catalysts based on non-noble metals such as Ni exhibited high catalytic activity, and its full conversion can be obtained at around 600°C.$^{12,13}$ Though the Ni-based catalytic activity is inferior to that of the Ru-based ones, the full conversion temperature is still in the working region for solid oxide fuel cells (SOFCs). Hence direct NH$_3$-fed SOFCs are expected to be a next-generation fuel cell system, and many groups have conducted feasibility tests on the cells, especially with Ni-based anodes.$^{14-24}$ Comparative studies of SOFCs fueled by hydrogen and NH$_3$ have revealed that the performance of NH$_3$-fed SOFCs is comparable to that of H$_2$-fed SOFCs above 700°C.$^{17}$ Meng et al.$^{21}$ examined a Ni/Sm-doped ceria (SDC) anode in H$_2$ and NH$_3$, and interfacial resistances between electrodes and electrolytes in both fuels (H$_2$ and NH$_3$) were comparable at 650°C. Yahiro et al.$^{22}$ measured the oxide ionic conductivities of the series of doped ceria and reported that 20 wt%–Sm-doped SDC possessed the highest oxide ionic conductivity. Furthermore, it is accepted that doped ceria exhibits a high electronic conduction associated with a partial reduction of Ce$^{4+}$ to Ce$^{3+}$ in a reducing atmosphere.$^{23,24}$ Therefore, SDC behaves as a mixed ionic electronic conductor in an anode. Akimoto et al. investigated bimetallic Ni–Fe$^{25}$ and Ni–Mo$^{26}$ loaded on SDC as anodes for NH$_3$-fueled SOFCs. The anodic performance of the Ni–SDC anode was modified by Fe and Mo additions. The SDC-based anodes were based on the metal-SDC cermet and contained relatively high metallic concentrations at above 60 wt%.$^{27}$ In Ni–SDC with a high concentration of Ni, the Ni particles are highly aggregated and form an electron conduction path. The SDC cluster phase in a Ni–SDC anode can be solely an electron conduction path, and if the electronic conductivity is sufficient, the amount of Ni loading can be suppressed. Sugihara et al.$^{28}$ investigated
the anodic performance of Ni–SDC with 0–40 wt % Ni in H₂ and reported that the 20 wt % Ni loading exhibited the maximum anodic performance. In this study, we examined the anodic performances of Ni–SDCs with different Ni loading contents using NH₃ fuel. Since NH₃ is a corrosive gas, it might be important to elucidate the anodic behavior in diluted NH₃ fuel. In this study, argon-balanced 6% NH₃ gas was used.

2. Experimental section

2.1 Preparation of Ni–SDC powders

The SDC powders [(SmO₁₅)₀.₂(CeO₂)₀.₈] were prepared by the reverse co-precipitation method with NH₃ using Sm(NO₃)₃·6H₂O (Wako, >99% pure) and Ce(NO₃)₃·6H₂O (Wako, 99.5%) as starting materials. Aqueous solutions containing Sm(NO₃)₃·6H₂O and Ce(NO₃)₃·6H₂O in prescribed ratios were added to aqueous solution of NH₃ (28%) with vigorous stirring. The resultant precipitates were filtered, dried, and calcinated at 900°C in air. The resultant powders were characterized via X-ray diffraction. To obtain Ni–SDC powders, the obtained SDC powders were dispersed in aqueous solution of Ni(NO₃)₃·6H₂O (Wako, 99.5%). This solution was evaporated at 80°C and then heated at 100°C for 4 h. The dried powders of the precursors were calcinated at 900°C for 4 h and then ball-milled for 24 h. The amounts of Ni-loading in the Ni–SDC powders were 10, 40, and 50 wt % in Ni-metal equivalent. Specific surface area of the Ni–SDC powders determined by the Brunauer–Emmett–Teller (BET) analysis was 21.5 m²·g⁻¹.

2.2 Half-cell fabrications

The cell configuration used in this study is shown in Fig. 1. Ceria–scandia–stabilized zirconia, (Sc₂O₃)₀.₃(Fe₂O₃)₀.₇–(Ce₂O₃)₀.₀₂(Zr₂O₃)₀.₈ (ScCSZ) that was purchased from Daiichi Kigenso Kagaku Kogyo was used as an electrolyte. The ScCSZ powder was pressed at 14.7 MPa and sintered at 1500°C for 10 h to yield a dense electrolyte disk of 0.8 cm diameter and 1.0 mm thickness. The cathode and reference electrodes were formed on a surface of the ScCSZ disk using Pt paste followed by calcination at 1000°C for 20 min. The opposite surface of the ScCSZ disk was hand-printed with graphite as a conductive treatment. The anode films were deposited by electrochemical deposition (EPD). To prepare the starting EPD bath, 0.24 g of the anode powders were added to 30 mL of acetylacetone containing 0.016 g of I₂ as a charging agent. The above mixture was sonicated for 20 min to yield a suspension. The ScCSZ substrate and stainless sheet were immersed into the suspension. The distance between them was 10 mm. The anode powders were deposited onto the graphite surface with an applied dc voltage of 30 V and a deposition time of 4.5 min. The deposits were sintered at 1000°C in air for 2 h. The sintered anode was coated with Pt paste as a current collector. All of the electrodes were led with a Pt wire. The apparent areas of the anode and the cathode were 0.13 and 0.20 cm², respectively.

2.3 Measurement of the cell properties

To measure the I–V and I–P curves of the single cell, the sets of potential and current terminals at the anode and cathode were used. Since the thickness of the electrolyte support is as large as 10 mm in the present case, the current density was calculated based on the area of the cathode which is larger than that of the anode. The I–V measurement was carried out by the pseudo 4-terminal configuration as shown in Fig. 1. For anodic impedance analysis were carried out by the three-terminal configuration. In the present cells, the cathode and anode have round shapes with the identical center. They have different diameters of 4.0 and 5.0 mm, respectively. Therefore, the edge of the cathode is overhanging by 0.5 mm from that of the anode. Alder et al. estimated the effect of overhang of the counter electrode across an electrolyte support. According to their estimation, the potential difference between the active and reference electrodes is approximately half the cell potential, which is expected in a symmetric cell. In the present case, the degree of overhang (0.5 mm) is half the electrolyte thickness (1.0 mm). Therefore, we think that the influence of the non-symmetric configuration in the present study on the anodic potential is very small.

The sides of anode and cathode were exposed to 6% dry NH₃/Ar gas and ambient air. The flow rate of the NH₃/Ar gas was 300 ml min⁻¹. The test cells were placed in a tubular furnace with a temperature controller. The I–V characteristics and impedance measurements were conducted in the temperature range between 700 and 900°C using a Solartron 1260 impedance/gain phase analyzer with a 1287A potentiostat/galvanostat.

2.4 Examination of NH₃ decomposition activity of the Ni–SDC catalysts

The catalytic reaction of NH₃ decomposition (1) was carried out in a conventional fixed-bed flow reactor.

\[2\text{NH}_3 = \text{N}_2 + 3\text{H}_2\] (1)

The Ni–SDC catalysts (1.0 g) were placed in a fixed-bed reactor. The reaction gas was 6% NH₃/Ar. The total
flow rate was 50 cm$^3$min$^{-1}$; the space velocity was 80,000 h$^{-1}$. The reaction temperature was increased step-wise from 100 to 600°C, and the reaction was carried out at each temperature until the conversion reached a constant value. The conversion of NH$_3$ was evaluated by measuring the H$_2$ concentration arising from the reaction (1) using gas chromatography (Shimadzu, GC-8AIT) with an Active Carbon column and Ar carrier gas.

3. Results and discussion

3.1 Catalyst examinations

Figure 2 shows XRD patterns of the NiO–SDC powders. All the patterns consisted of the peaks attributed to SDC and NiO. From the full-width at half maximum (FWHM) of the NiO (111) peaks at 2$\theta$ = 37.2°, crystallite sizes of NiO were approximately estimated by the Scherer’s Eq. (1):

$$D = \frac{K\lambda}{B \cos \theta} \quad (2)$$

where, $D$ is a crystallite size, $K$ is Sherrer’s constant (0.9 in the present study), $\lambda$ is wave length of X-ray ($\lambda = 0.154$ nm for Cu-K$_\alpha$), $B$ is FWHM, and $\theta$ is diffraction angle of the peak. The estimation resulted in 20, 29, 30 nm for the powders containing 10, 40 and 50 wt% in terms of Ni element, respectively. The NiO crystallite size tend to increase with increase in the Ni content. In our previous work on the Ni–SDC anode,$^{28}$ crystallite size of Ni was correlated with Ni particles size, that the particle size increased with increase in the crystallite size. Therefore, we concluded that NiO particle size may increase with the increase in the Ni content.

The catalytic activity of NH$_3$ decomposition over the Ni–SDC powder was examined. Figure 3 shows the conversion curves of NH$_3$ decomposition over the Ni-free and Ni-loaded SDC powders. The temperatures for 50 and 100% NH$_3$ conversion, $T_{50}$ and $T_{100}$, are shown in Table 1. For the Ni-free SDC powder, the onset temperature of NH$_3$ decomposition was around 500°C, and temperature of 50% NH$_3$ conversion ($T_{50}$) of all the SDC powders were around 650°C. The Ni-loading remarkably enhanced the catalytic activity, and full conversion was achieved at around 630°C. Among the Ni-loaded catalysts, catalytic activity was highest at 10 wt% Ni–SDC, and it decreased with an increase in the Ni content. The large amount of Ni loading might induce Ni-particle aggregation that would lead to the surface area reduction of Ni.

3.2 $I$–$V$ characteristics

Figure 4(a) shows $I$–$V$ and $I$–$P$ characteristics of single cells with the Ni–SDC anodes at 900°C. The maximum power density (MPD) values of the Ni–SDC anodes are listed in Table 2; they were in the order of 10 wt% (98.8 mV·cm$^{-2}$) > 40 wt% (96.5 mV·cm$^{-2}$) > 50 wt% (67.7 mV·cm$^{-2}$). The 10 wt% Ni–SDC exhibited the similar but slightly higher MPD value than the 40 wt% Ni–SDC. This result is the order of the catalytic activity of NH$_3$ decomposition shown in Fig. 3. The Ni–SDC anodes were also examined in hydrogen fuel (97%H$_2$ + 3%H$_2$O). The obtained MPDs in hydrogen fuel are also listed in Table 2. Consistent with the 6% NH$_3$ case, the 10 wt% Ni–SDC exhibited the similar MPD value with the 40 wt% Ni–SDC and that higher than the 50 wt% Ni–SDC. Therefore, the performance of 10 wt% Ni–SDC anode in 6%NH$_3$ fuel

![Fig. 2. XRD patterns of the NiO–SDC powders with different Ni contents. The weight ratios denoted in the figure are those at Ni element. The XRD peaks marked with S and N are attributed to those of SDC and NiO, respectively.](image)

![Fig. 3. Conversion curves for NH$_3$ decomposition over the SDC and Ni–SDC catalysts with different Ni contents measured under 6% NH$_3$/Ar flow. Space velocity of the NH$_3$ flow is 80,000 h$^{-1}$.](image)

**Table 1.** Characteristic temperatures of ammonia decomposition over the SDC and Ni–SDC catalysts

| Ni content (wt%) | $T_{50}$ | $T_{100}$ |
|-----------------|---------|---------|
| 0               | 636     | —       |
| 10              | 493     | 608     |
| 40              | 518     | 650     |
| 50              | 533     | 674     |

$T_{50}$ and $T_{100}$ are the values of temperature for 50% and 100% NH$_3$ conversions, respectively. The total flow rate was 50 cm$^3$min$^{-1}$; the space velocity is 80,000 h$^{-1}$. 

![Table 1](image)
is probably characterized not only by the good catalytic activity of NH₃ decomposition but also by the substantially good activity in H₂ fuel. To achieve a good anode performance, enough electronic conductivity is required, as well as a good catalytic activity. Although SDC exhibits an electronic conduction in reducing atmosphere, it may not be sufficiently large, that is at most 10⁻¹ S·cm⁻¹ in H₂ at 800°C.31) In this sense, the 10 wt % Ni–SDC anode film is good at the catalytic activity but still lack of electronic conductivity. On the other hand, the 40 wt % Ni–SDC anode has an opposite situation of the 10 wt % Ni–SDC anode. The similar anode performance of the 10 and 40 wt % Ni–SDC anodes is probably due to the result of the balance of such the trade-off. In the case of 50 wt % Ni–SDC, the remarkable reduction of its catalytic activity is a possible reason of the low MPD.

Figure 4(b) shows the temperature dependence of the I–V and I–P characteristics of the cell with 40 wt % Ni–SDC. The cell performance significantly decreased with a decrease in temperature. At 850°C and below, large potential drop was observed at lower current density region, causing a potential drop of 0.2 V. Further research is required to determine the reason for the large potential drop, but the molecular diffusion of H₂ or the surface diffusion of dissociated hydrogen might be possible reasons. From the result of catalytic activity examination for 40 wt % Ni–SDC, the NH₃ molecules fully decomposed above 750°C. Since such a limiting current was not observed in 4% H₂ fuel, it was deduced that the steady-state concentration of H₂ originated from NH₃ fuel was still less than 4% in the actual anode film.

Since Pt paste was used as the current collecting layer, it is concerned that diffusion of Pt into the anode layer affect the anodic property. Figure 5 shows elemental mapping of the cross-section of the cell. The region between the two dotted lines is the 10 wt % Ni–SDC layer, where the intensity of X-ray fluorescence Ni strong. The gap between the Ni–SDC and ScCSZ layers was caused by a peeling-off during cutting along the cross-section. It should be noted that the elemental mappings of K₂Ni (7.471 KeV) and L₂Pt (2.048 KeV) may be slightly overlapped with L₂Ni from Pt (9.441 KeV) and L₂Zr from Zr (2.042 KeV), respectively. Distribution of Pt element on the anode layer is not observed in the Pt elemental mapping. The analysis revealed that the Pt layer deposited on the surface of the anodes as a current collector hardly diffuse into the anode layer. Therefore, we considered that contribution of Pt in the anode layer such as catalytic property and electrical conductivity is negligibly small. To discuss on the influence of the Pt diffusion layer on the anodic property, the symmetrical cell, Pt|ScCSZ|Pt, was examined in 6%NH₃/Ar. Figure 6(a) shows the I–V characteristic of the Pt|ScCSZ|Pt cell.
Compared to the cells with the Ni-SDC/Pt anodes, cell performance was remarkably decreased. The MPD values are shown in Table 2. Although the MPD value in H2 is comparable to those of the Ni-SDC anodes, that in 6% NH3 is no more than 7.2 mW/cm². The Nyquist plot observed under OCV is shown in Fig. 6(b). The estimated parameters are shown in Table 3. The Pt anode has significantly large resistance in the higher frequency region attributable to the electrochemical oxidation process, which means the concentration of decomposed H2 is very low at the TPB. The Pt anode may have a good property under H2 fuel but have a poor activity of NH3 decomposition. Therefore, we conclude that the Pt layer deposited on the Ni-SDC has small contribution of NH3 decompaction. However, the Pt layer may interrupt gas diffusion of NH3 to some degree.

### 3.3 AC impedance analysis

Figure 7 shows the Nyquist plots of the 40 wt% Ni-SDC anode in 6% NH3 and 4% H2 observed at open circuit voltage (OCV). The semicircles of the Nyquist plots in 4% H2 are much smaller than those in 6% NH3. All the Nyquist plots consisted of a couple of depressed semicircles. The curve fittings were plotted by assuming the equivalent circuit as shown in Fig. 8. Since R3 seems to have a small contribution, R2 and R1 were added as a high-frequency component (RHF), and the low-frequency R4 was referred to as RL.F. The R1 component is assigned to a contribution from an ohmic resistance of the solid electrolyte substrate and other ohmic elements. Figure 9 shows the Arrhenius plots of the RHF and RL.F components in 6% NH3 and 4% H2, respectively. Good linearity was obtained for all the resistant components, and activation energies were evaluated as follows: RHF (6% NH3) = 1.02 eV, RHF (4% H2) = 1.03 eV, RL.F (6% NH3) = 0.27 eV, and RL.F (4% H2) = 0.23 eV. The activation energies in 6% NH3 and 4% H2 are very close to each other, which means that the anode reactions are rate-controlled by similar processes in both the atmospheres. In NH3 fuel, electrochemical oxida-
tion of NH$_3$ (3) and H$_2$ (4) are possible to occur. However, the process (3) does not seem to be a rate-determining process under the 6% NH$_3$ fuel. Theoretically, 9% H$_2$ is expected to yield from a fully converted 6% NH$_3$, but in fact the impedances in 6% NH$_3$ is larger than those of 4% H$_2$. This indicates that concentration of H$_2$ produced from the NH$_3$ decomposition on the Ni–SDC anode is still lower than 4%.

$$\begin{align*}
2\text{NH}_3 + 3\text{O}_2^-> \text{N}_2 + 3\text{H}_2\text{O} + 6\text{e}^- & \quad (3) \\
\text{H}_2 + \text{O}_2^- \rightarrow \text{H}_2\text{O} + 2\text{e}^- & \quad (4)
\end{align*}$$

Kishimoto et al. observed the impedance spectra of the Ni–SDC anode in hydrogen atmosphere. They also observed three resistance components in a low water vapor pressure. The highest frequency semicircle is very small also in that case. They assigned the semicircles in the middle and low frequency regions to electrochemical oxidation process with charge transfer and adsorption and desorption of dissociated hydrogen on the Ni surface, respectively. The reported activation energies of the middle and low frequency components are 0.83 and 0.41 eV, respectively. The activation energy of the former is close to that of the R$_{HF}$ component in the present case.

Figure 9 shows the Nyquist plots of the 40 wt% Ni–SDC anodes in 6% NH$_3$ and 4% H$_2$.

![Fig. 9](image-url)  
**Fig. 9.** Temperature dependence of the resistance components, $R_{HF}$ and $R_{LF}$, of the 40 wt% Ni–SDC anodes in 6% NH$_3$ and 4% H$_2$.

The Ni-loaded SDCs were examined as the SOFC anodes fueled with 6% NH$_3$. The Ni-free SDCs exhibited poor catalytic activity for NH$_3$ decomposition. The catalytic activity was significantly enhanced by the 10, 40 and 50 wt% Ni-loading on SDC, and full conversion was observed around 600°C; the maximum activity was obtained at the 10 wt% Ni-loading. The 10 and 40 wt% Ni–SDC anodes gave the similar MPDs of 98.8 and 96.5 mW·cm$^{-2}$ as single-cell performances at 900°C, respectively. The impedance analyses revealed that anode process in 6% NH$_3$ is similar to that in 4% H$_2$. The impedance spectra suggested that the electrochemical oxidation and gas diffusion could be a rate-controlling processes. The catalytic activity of the anode for NH$_3$ decomposition may not be sufficiently large, which affected rate of the diffusion process. Therefore, further enhancement of the catalytic activity seems to be required to obtain the larger MPD value in such the low concentration NH$_3$.

4. Conclusion

The Ni-loaded SDCs were examined as the SOFC anodes fueled with 6% NH$_3$. The Ni-free SDCs exhibited poor catalytic activity for NH$_3$ decomposition. The catalytic activity was significantly enhanced by the 10, 40 and 50 wt% Ni-loading on SDC, and full conversion was observed around 600°C; the maximum activity was obtained at the 10 wt% Ni-loading. The 10 and 40 wt% Ni–SDC anodes gave the similar MPDs of 98.8 and 96.5 mW·cm$^{-2}$ as single-cell performances at 900°C, respectively. The impedance analyses revealed that anode process in 6% NH$_3$ is similar to that in 4% H$_2$. The impedance spectra suggested that the electrochemical oxidation and gas diffusion could be a rate-controlling processes. The catalytic activity of the anode for NH$_3$ decomposition may not be sufficiently large in 6% NH$_3$.

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