Performance of Ni$_{0.8-x}$Cu$_{0.2}M_x$ (M = Fe and Co) alloy-based cermet anodes for intermediate-temperature solid oxide fuel cells fueled with syngas

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The performance of Ni-based trimetallic alloy cermet anodes, Ni$_{0.8-x}$Cu$_{0.2}M_x$ (M = Fe and Co; x = 0.1, 0.2, and 0.3)/Ce$_{0.85}$Sm$_{0.15}$O$_{1.9}$ (SDC), was investigated for intermediate-temperature (500–700°C) solid oxide fuel cells (IT-SOFCs), using humidified (3% H$_2$O) model syngas with a molar ratio of H$_2$/CO = 3/2 as the fuel. Though doping Fe or Co 10 mol % into the Ni$_{0.8}$Cu$_{0.2}$/SDC anode led the cell performance to the increase, the cell performance decreased as Fe or Co content increased further. On the one hand, doping Fe or Co into the Ni$_{0.8}$Cu$_{0.2}$/SDC anode further effectively inhibited carbon deposition owing to CO on the anode. Our results suggest that the Ni$_{0.8}$Cu$_{0.2}$Fe$_{0.1}$/SDC and Ni$_{0.8}$Cu$_{0.2}$Co$_{0.1}$/SDC anodes are more promising materials than Ni$_{0.8}$Cu$_{0.2}$/SDC anode for syngas fuel in IT-SOFCs.

Key-words : Nickel-based trimetallic alloy cermet anode, Intermediate-temperature solid oxide fuel cell (IT-SOFC), Syngas fuel, Carbon deposition

1. Introduction

Biogas is mainly composed of CH$_4$ and CO$_2$ and is saturated with water vapor.\textsuperscript{1-3} The application of biogas as a fuel for solid oxide fuel cells (SOFCs) has attracted much attention in terms of environmental protection.\textsuperscript{4-6} We are developing intermediate-temperature (500–700°C) SOFCs (IT-SOFCs) fueled with syngas derived from biogas in an external process.\textsuperscript{7} Biogas is transformed to the syngas by CH$_4$ dry reforming with CO$_2$ (1) and CH$_4$ steam reforming with H$_2$O (2), as shown below.

\[ \text{CH}_4 + \text{CO}_2 \rightarrow 2\text{H}_2 + 2\text{CO} \] (1)
\[ \text{CH}_4 + \text{H}_2\text{O} \rightarrow 3\text{H}_2 + \text{CO} \] (2)

In the process of developing IT-SOFCs fueled with syngas, inhibiting carbon deposition on the anode is important because it lowers the power generation performance. Coking from CO in the syngas is due to the reverse water gas reaction (3) and disproportionation reaction (4).

\[ \text{CO} + \text{H}_2 \rightarrow \text{C} + \text{H}_2\text{O} \] (3)
\[ 2\text{CO} \rightarrow \text{C} + \text{CO}_2 \] (4)

Carbon deposition reactions become dominant below 700°C.\textsuperscript{8} We recently investigated the electrochemical performance of Ni$_1-x$Cu$_x$/Ce$_{0.85}$Sm$_{0.15}$O$_{1.9}$ (SDC) cermet anodes in IT-SOFCs at 600 and 700°C using a model syngas fuel humidified (3% H$_2$O) with a molar ratio of H$_2$/CO = 3/2. In this previous study, we found that the Ni$_{0.8}$Cu$_{0.2}$/SDC cermet anode remarkably suppresses carbon deposition and its electrochemical performance is comparable to that of the Ni/SDC cermet anode.\textsuperscript{9} Still, it is necessary to develop improved anode materials that can further inhibit carbon deposition and show high electrochemical performances for practical applications.

Based on the studies of the Ni-based alloy anode catalysts for H$_2$\textsuperscript{10-13} or CH$_4$\textsuperscript{14} fueled SOFCs, here, we added Fe, Co and Zn as a third component to the Ni$_{0.8}$Cu$_{0.2}$ alloy, replacing some Ni with these transition metals to examine its electrochemical performance for practical applications. Our results suggest that adding a small amount of Fe or Co can improve the inhibition of carbon deposition. We also report the power generation performance of IT-SOFCs with Ni$_{0.8-x}$Cu$_{0.2}$Fe$_x$/SDC or Ni$_{0.8-x}$Cu$_{0.2}$Co$_x$/SDC cermet anodes using model syngas fuel with H$_2$/CO = 3/2 at 700 and 600°C.

2. Experimental procedures

Precursors of Ni$_{0.8-x}$Cu$_{0.2}M_x$ (M = Fe and Co; x = 0.0, 0.1, 0.2, and 0.3) alloys, Ni$_{0.8-x}$Cu$_{0.2}$M$_x$O, were prepared by a solid-state reaction, using NiO (99.97%, Japan Pure Chemical, Japan), CuO (99.99%, Kanto Chemical, Japan), Fe$_2$O$_3$ (99.99%, Japan Pure Chemical, Japan), and Co$_2$O$_3$ (99.99%, Kanto Chemical, Japan) as starting materials. NiO, CuO, and Fe$_2$O$_3$ (or Co$_2$O$_3$) in desired molar ratios were mixed and ground in ethanol for 24 h using a ball mill, and then calcined at 900°C for 12 h in air. Mixture of the calcined Ni$_{0.8-x}$Cu$_{0.2}$M$_x$O and SDC (AGC Seimi Chemical, Japan) powders in a mass ratio of Ni$_{0.8-x}$Cu$_{0.2}$M$_x$O/SDC = 70/30 for cermet anode was ground in ethanol for 24 h using a ball mill and then dried at 50°C overnight.

Electrolyte-supported single test-cells were fabricated with the SDC powder, the prepared Ni$_{0.8-x}$Cu$_{0.2}$M$_x$O/SDC powder and commercial La$_{0.6}$Sr$_{0.4}$Co$_{0.2}$Fe$_{0.8}$O$_{3}$ (LSCF; Hosokawa Powder Technology Research Institute, Japan) cathode powder, according to the previous paper.\textsuperscript{9} The SDC electrolyte pellet uniaxially pressed at 10 MPa and sintered at 1400°C for 10 h was approximate 490 µm thick. An anode paste of the prepared powder and polyethylene glycol (Hampton Research, USA) was screen-printed on the SDC electrolyte pellet followed by heating at 1000°C for 2 h. A cathode paste of LSCF powder and polyethylene glycol was also screen-printed onto the opposite side of
the anode-coated SDC electrolyte pellet followed by heating at 1000°C for 2 h. The electrode areas were estimated to be 28.3 mm². The test-cell, which a Pt reference electrode was attached to, was set in a device for measurements of cell performance followed by heating in situ at 700°C under H₂ atmosphere to reduce Ni₀.₈-Cu₀.₂-M₀ₓ to trimetall Ni₀.₈-Cu₀.₂-M₀ₓ alloy for forming cermet anode. Commercial soft-glass rings were used for gas seal. The cell performances were measured at 700 and 600°C using humidified (3% H₂O) model syngas and dry air fed as fuel and oxidant, respectively, at a controlled gas flow rate of 50 cm³/min. Considering the time dependence of cell performance was examined under a load of 0.1 A/cm² using humidiﬁed syngas at 700°C for 24 h.

Pt mesh current collectors were availed for both electrodes. Current–voltage curves and electrochemical impedance spectra were measured using an electrochemical analyzer (BioLogic, single-channel potentiostat SP-150, France). The current-interruption method was separately employed to estimate ohmic and overpotential losses in the test-cell in the similar way to the previous paper. The time dependence of cell performance was examined under a load of 0.1 A/cm² using humidiﬁed syngas at 700°C for 24 h.

The anode materials were characterized using powder X-ray diffraction (XRD; Rigaku RINT2100/PC, Japan) with monochromated Cu Kα radiation and scanning electron microscopy (SEM; Jeol JSM-6380, Japan). The chemical composition and distribution images of Ni₀.₈-Cu₀.₂-M₀ₓ alloy particles in the anodes were examined using energy-dispersive X-ray spectroscopy (EDS; Jeol EX-54175JMU, Japan) attached to SEM. The chemical composition and distribution images of Ni₀.₈-Cu₀.₂-M₀ₓ alloy, because the atomic radius of Fe is larger than that of Ni. The other XRD peaks were due to SDC. Therefore, the Ni₀.₈-Cu₀.₂ alloy shifted to lower angles as Fe content increased, in Fig. 1. The XRD peaks at 2θ ≈ 44.4 and 51.6° due to the Ni₀.₈-Cu₀.₂ alloy shifted to lower angles as Fe content increased, suggesting the formation of a Ni₀.₈-Cu₀.₂-Feₓ alloy, because the atomic radius of Fe is larger than that of Ni. The other XRD peaks were due to SDC. Therefore, the Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes were conﬁrmed to be a mixture of Ni₀.₈-Cu₀.₂-Feₓ and SDC.

The power density curves of the test-cells with Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes at 700 and 600°C are shown in Fig. 2. The maximum power density of the test-cell with Ni₀.₈-Cu₀.₂/SDC anode was about 148 mW/cm² at 700°C. This value is 126% of the previously reported maximum power density for a Ni₀.₈-Cu₀.₂/SDC anode test-cell. The diﬀerence is attributable to the decrease in the ohmic loss of the electrolyte pellet because the present electrolyte pellet is thinner than the previous one that was about 660 μm thick. At 700°C, there was no signiﬁcant difference between power densities of the test-cells with Ni₀.₈-Cu₀.₂/SDC and Ni₀.₅-Cu₀.₂Fe₀.₃/SDC anodes, but the power densities decreased as Fe content increased further. In other words, the order of the maximum power densities at 700°C was Ni₀.₈-Cu₀.₂/SDC > Ni₀.₅-Cu₀.₂Fe₀.₃/SDC > Ni₀.₆-Cu₀.₂Fe₀.₂/SDC > Ni₀.₅-Cu₀.₂F₀.₃/SDC. The diﬀerences among the cell performances were explored by the current-interruption technique, i.e., by splitting the current into ohmic loss and overpotential loss. The overpotential losses for the test-cells slightly increased at x = 0.2 and 0.3, while no remarkable diﬀerences in the ohmic losses were observed. The increase in the overpotential loss, namely the decrease in triple-phase boundaries, resulted in a decrease in the power density.

The power densities at 600°C showed a similar trend to those observed at 700°C, but their values were about 40% of those at 700°C. The order of the maximum power densities at 600°C was Ni₀.₅-Cu₀.₂/SDC > Ni₀.₆-Cu₀.₂Fe₀.₂/SDC > Ni₀.₅-Cu₀.₂Fe₀.₃/SDC > Ni₀.₆-Cu₀.₂F₀.₃/SDC. The current-interruption method revealed that the notable increase in the overpotential loss at x = 0.3 caused a decrease in the power density.

Figure 3 shows the time dependence of voltages of the test-cells with Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes under a 0.1 A/cm² load at 700°C. The voltage of the test-cell with Ni₀.₅-Cu₀.₂/SDC anode decreased slightly for 24 h. On the other hand, the voltages of the test-cell with Fe-doped anodes were relatively stable. The order of the cell voltages after 24 h was Ni₀.₅-Cu₀.₂F₀.₃/SDC > Ni₀.₆-Cu₀.₂Fe₀.₂/SDC > Ni₀.₅-Cu₀.₂Fe₀.₃/SDC > Ni₀.₆-Cu₀.₂F₀.₃/SDC > Ni₀.₈-Cu₀.₂F₀.₃/SDC. These results indicate that the cell performance with Ni₀.₇-Cu₀.₂Fe₀.₁/SDC anode hardly changed during electrochemical tests and can last 24 h.

3. Results and discussion

3.1 Ni₀.₈-Cu₀.₂Feₓ/SDC cermet anode

XRD patterns of the prepared anode materials are shown in Fig. 1. The XRD peaks at 2θ ≈ 44.4 and 51.6° due to the Ni₀.₈-Cu₀.₂ alloy shifted to lower angles as Fe content increased, suggesting the formation of a Ni₀.₈-Cu₀.₂-Feₓ alloy, because the atomic radius of Fe is larger than that of Ni. The other XRD peaks were due to SDC. Therefore, the Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes were conﬁrmed to be a mixture of Ni₀.₈-Cu₀.₂-Feₓ and SDC.

The power density curves of the test-cells with Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes at 700 and 600°C are shown in Fig. 2. The maximum power density of the test-cell with Ni₀.₅-Cu₀.₂/SDC anode was about 148 mW/cm² at 700°C. This value is 126% of the previously reported maximum power density for a Ni₀.₈-Cu₀.₂/SDC anode test-cell. The diﬀerence is attributable to the decrease in the ohmic loss of the electrolyte pellet because the present electrolyte pellet is thinner than the previous one that was about 660 μm thick. At 700°C, there was no signiﬁcant difference between power densities of the test-cells with Ni₀.₈-Cu₀.₂/SDC and Ni₀.₅-Cu₀.₂Fe₀.₃/SDC anodes, but the power densities decreased as Fe content increased further. In other words, the order of the maximum power densities at 700°C was Ni₀.₈-Cu₀.₂/SDC > Ni₀.₅-Cu₀.₂Fe₀.₃/SDC > Ni₀.₆-Cu₀.₂Fe₀.₂/SDC > Ni₀.₅-Cu₀.₂F₀.₃/SDC. The diﬀerences among the cell performances were explored by the current-interruption technique, i.e., by splitting the current into ohmic loss and overpotential loss. The overpotential losses for the test-cells slightly increased at x = 0.2 and 0.3, while no remarkable diﬀerences in the ohmic losses were observed. The increase in the overpotential loss, namely the decrease in triple-phase boundaries, resulted in a decrease in the power density.

The power densities at 600°C showed a similar trend to those observed at 700°C, but their values were about 40% of those at 700°C. The order of the maximum power densities at 600°C was Ni₀.₅-Cu₀.₂/SDC > Ni₀.₆-Cu₀.₂Fe₀.₂/SDC > Ni₀.₅-Cu₀.₂Fe₀.₃/SDC > Ni₀.₆-Cu₀.₂F₀.₃/SDC > Ni₀.₈-Cu₀.₂F₀.₃/SDC. The current-interruption method revealed that the notable increase in the overpotential loss at x = 0.3 caused a decrease in the power density.

Figure 3 shows the time dependence of voltages of the test-cells with Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes under a 0.1 A/cm² load at 700°C. The voltage of the test-cell with Ni₀.₅-Cu₀.₂/SDC anode decreased slightly for 24 h. On the other hand, the voltages of the test-cell with Fe-doped anodes were relatively stable. The order of the cell voltages after 24 h was Ni₀.₅-Cu₀.₂F₀.₃/SDC > Ni₀.₆-Cu₀.₂Fe₀.₂/SDC > Ni₀.₅-Cu₀.₂Fe₀.₃/SDC > Ni₀.₆-Cu₀.₂F₀.₃/SDC > Ni₀.₈-Cu₀.₂F₀.₃/SDC. These results indicate that the cell performance with Ni₀.₇-Cu₀.₂Fe₀.₁/SDC anode hardly changed during electrochemical tests and can last 24 h.

Fig. 1. X-ray diffraction patterns of the Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes after reduction treatment at 700°C.

Fig. 2. Power density versus current density at (a) 700°C and (b) 600°C for the Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes.

Fig. 3. Time dependences of cell voltages for the Ni₀.₈-Cu₀.₂-Feₓ/SDC anodes under 0.1 A/cm² load at 700°C.
Carbon deposition is mainly generated by reactions (3) and (4) when syngas fuel is used. Figure 4 shows the amounts of carbon deposited on the anodes during the 24 h electrochemical test, estimated by SEM-EDS. The amount of carbon deposited on the present Ni$_{0.8}$Cu$_{0.2}$/SDC anode was smaller than the previously reported value; it decreased from 13.6 to 7.6 mass%. This reduced carbon deposition can be attributed to the improvement of the power density owing to decrease in the ohmic loss. Doping Fe into the Ni$_{0.8}$Cu$_{0.2}$/SDC anode further inhibited carbon deposition and the amount of deposited carbon decreased to about 4 mass%. The effect of doping Fe to Ni/GDC cermet anodes on carbon deposition have been elucidated as follows: Fe addition (i) restrains C–C bond formation and/or reduces the thermodynamic driving force for carbon nucleation and (ii) promotes the formation of disordered amorphous carbon which can be easily removed as CO by the water gas reaction, i.e., the reverse of reaction (3).

XRD patterns revealed that the structures of anode materials were maintained after the 24 h electrochemical tests. Figure 5 shows SEM micrographs of the test-cell with the Ni$_{0.7}$Cu$_{0.2}$Fe$_{0.1}$/SDC anode before and after the 24 h electrochemical test. As seen in the images, the Ni$_{0.7}$Cu$_{0.2}$Fe$_{0.1}$/SDC anode was in close contact with the electrolyte. A comparison of the SEM micrographs before and after the 24 h electrochemical test demonstrates that the sinterability did not change significantly. Moreover, a comparison of the analytical results by SEM-EDS showed that the chemical composition and particle distribution of the Ni$_{0.7}$Cu$_{0.2}$Fe$_{0.1}$/SDC anode hardly changed, suggesting minimal change in overpotential loss and that the test-cell output was maintained, as shown in Fig. 3. Overall, we found that the Ni$_{0.7}$Cu$_{0.2}$Fe$_{0.1}$/SDC anode was the best Ni$_{0.8}$Cu$_{0.2}$/Fe$_{0.1}$/SDC anode for syngas fuel with H$_2$/CO = 3/2.

### 3.2 Ni$_{0.8-x}$Cu$_{0.2}$Co$_x$/SDC anode

XRD patterns of the prepared anode materials are shown in Fig. 6. The Ni$_{0.8-x}$Cu$_{0.2}$ alloy peaks at 2θ ≈ 44.4 and 51.6° slightly shifted to lower angles as the Co content increased. This suggests that Co was also occluded in the Ni$_{0.8}$Cu$_{0.2}$ alloy like Fe, forming the Ni$_{0.8-x}$Cu$_{0.2}$Co$_x$ alloy because the atomic radius of Co is slightly larger than that of Ni.$^{16}$ The other XRD peaks were due to SDC. Therefore, the Ni$_{0.8-x}$Cu$_{0.2}$Co$_x$/SDC anodes were confirmed to be a mixture of Ni$_{0.8-x}$Cu$_{0.2}$Co$_x$ and SDC.

The power density curves of the test-cells with the Ni$_{0.8-x}$Cu$_{0.2}$Co$_x$/SDC anodes at 700 and 600°C are shown in Fig. 7. At 700°C, the power density of the Ni$_{0.8}$Cu$_{0.2}$Co$_{0.1}$/SDC anode test-cell was superior to that of the Ni$_{0.8}$Cu$_{0.2}$/SDC anode test-cell, but the power density decreased as the Co content increased further. The order of the maximum power densities at 700°C was Ni$_{0.8}$Cu$_{0.2}$Co$_{0.1}$/SDC > Ni$_{0.8}$Cu$_{0.2}$/SDC ≈ Ni$_{0.8}$Cu$_{0.2}$Co$_{0.2}$/SDC > Ni$_{0.8}$Cu$_{0.2}$Co$_{0.3}$/SDC. The current interruption method revealed that there were no remarkable differences in the ohmic and overpotential losses of the test-cells with different anodes except for the Ni$_{0.8}$Cu$_{0.2}$Co$_{0.3}$/SDC anode. The power densities at 600°C decreased to about 40% of those at 700°C. The order of the maximum power densities at 600°C was Ni$_{0.8}$Cu$_{0.2}$Co$_{0.1}$/SDC > Ni$_{0.8}$Cu$_{0.2}$Co$_{0.2}$/SDC > Ni$_{0.8}$Cu$_{0.2}$/SDC > Ni$_{0.8}$Cu$_{0.2}$Co$_{0.3}$/SDC.

Figure 8 shows the time dependence of voltages of the test-cells under a 0.1 A/cm$^2$ load at 700°C. The voltages of the test-cell with the Co-doped anodes were relatively stable for 24 h. The order of the cell voltages after 24 h was Ni$_{0.8}$Cu$_{0.2}$Co$_{0.1}$/SDC > Ni$_{0.8}$Cu$_{0.2}$Co$_{0.2}$/SDC > Ni$_{0.8}$Cu$_{0.2}$/SDC > Ni$_{0.8}$Cu$_{0.2}$Co$_{0.3}$/SDC. These indicate that the cell performance of the Ni$_{0.8}$Cu$_{0.2}$Co$_{0.1}$/SDC anode test-cell hardly changed during the 24 h electrochemical test.

Figure 9 shows the amounts of carbon deposited on the anodes, estimated using SEM-EDS, during the 24 h electrochemical test. Doping Co into the Ni$_{0.8}$Cu$_{0.2}$/SDC anode also inhibited carbon deposition, and the amount of deposited carbon decreased to about 4 mass%. The role of Co in the Ni$_{0.8-x}$Cu$_{0.2}$Co$_x$/SDC cermet anode is probably similar to that of Fe in the
4. Conclusion

The effect of Fe or Co addition to Ni$_{0.8}$Cu$_{0.2}$/SDC cermet anode on the performance of fuel test-cells was examined using humidified (3% H$_2$O) syngas fuel with H$_2$/CO = 3/2 at 700 and 600°C. Doping Fe or Co 0.1 mol% into the Ni$_{0.8}$Cu$_{0.2}$/SDC anode brought the increase in the cell performance, and effectively restrained carbon deposition owing to CO on the anode, exhibiting the reduced amounts of deposited carbon. As the Fe and Co contents increased further, the cell performance decreased due to the increase in the overpotential loss in the anode. In the electrochemical test for 24 h, the test-cells revealed the cell performance in the order of Ni$_{0.8}$Cu$_{0.2}$Fe$_{0.3}$/SDC > Ni$_{0.8}$Cu$_{0.2}$/SDC > Ni$_{0.8}$Cu$_{0.2}$/Ni$_{0.7}$Cu$_{0.2}$/Fe$_{0.3}$/SDC > Ni$_{0.8}$Cu$_{0.2}$/Ni$_{0.7}$Cu$_{0.2}$/Co$_{0.2}$/SDC > Ni$_{0.8}$Cu$_{0.2}$/Ni$_{0.7}$Cu$_{0.2}$/Co$_{0.2}$/Fe$_{0.3}$/SDC > Ni$_{0.8}$Cu$_{0.2}$/Ni$_{0.7}$Cu$_{0.2}$/Co$_{0.2}$/Fe$_{0.3}$/SDC for the Co-doped anodes. This study suggests that the Ni$_{0.5}$Cu$_{0.2}$/Fe$_{0.3}$/SDC and Ni$_{0.7}$Cu$_{0.2}$/Fe$_{0.3}$/SDC cermet anodes are promising candidates for practical applications.

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