A small and high-performance solid oxide fuel cell (SOFC) system without any external equipment for fuel reforming has a potential as a power source for mobile machine like automobiles and robots. To realize the small SOFC system, the direct use of liquid hydrocarbon fuel, which has high energy density and is easy to storage and supply, is desirable. In order to use the existing fuel supply infrastructure, the SOFC should be operated by gasoline. For instance, gasoline sold in Japan contains H2S up to 10 ppm. Thus, the SOFC must be able to be stably operated at 10 ppm H2S-containing gasoline. A porous cermet of Ni and yttria-stabilized zirconia (YSZ), which has widely been studied as an anode electrode for the internal reforming operation SOFC, is one of promising anode electrode materials; however, performance degradation of anode electrode due to sulfur poisoning occurs by trace amounts of the impurity sulfur. To date, extensive efforts have been taken to gain a profound understanding of the sulfur poisoning by both experimental and theoretical approaches. Based on the knowledge, promising ways to suppress the sulfur poisoning and recover the degraded performance have discovered as was summarized and reviewed in some papers. Previous studies revealed that the sulfur poisoning of the Ni-YSZ anode is a two-step process. The first process is physical- and/or chemical-adsorption of sulfur onto the surface of Ni particles to block the active sites for electrochemical oxidation of fuel, and causes a rapid increase in anode overpotential. The subsequent second process is microstructure change and the formation of sulfur compounds such as NiS and NiS2, and generates a gradual degradation that lasts for hundred hours or even longer. The sulfur poisoning rate of both processes is dependent on the partial pressure of the sulfur species in the fuel and temperature, and the poisoning is stopped and recovered by introducing a fuel that does not contain sulfur. However, the recovery by the introduction of pure gases (e.g., H2, N2) without sulfur species is unrealistic because it is difficult to equip the pure gases in a limited space of the mobile machines. To overcome the problems, advanced anode materials have been proposed. SOFC was successfully operated by using Cu-ceria based anodes. Mixed ion electron conducting ceramics anode is another candidate such as BaZr0.1Ce0.7Y0.2O3−δ. However, the lack catalytic activity for fuel oxidation of these anode electrodes limits the cell powers. Therefore, a suppressing method of sulfur-poisoning for the Ni-YSZ anode electrode is much expected.

In this study, the effect of controlling the potential of the anode electrode in the stable region to suppress the generation of nickel sulfide is revealed. The possibility of performance recovery of the sulfated anode electrode by shifting the anode electrode potential to the stable region, in which nickel exists as metal, is also investigated.

**Equilibrium Electrode Potential by Thermodynamic Calculation**

Dong et al. and Chen et al. revealed that Ni3S2 is a sulfur composite, which is formed under the conditions of relatively lower partial pressure of the sulfur species and the cell temperature. Phase equilibrium of the Ni-O-S systems also predicts that Ni3S2 is likely formed. Peterson et al. reported that H2S is thermally-decomposed into elemental sulfur and hydrogen. Therefore, for simplification, only three kinds of equilibrium reactions indicated by equations 1, 2, and 3 are assumed to be occurred on the anode electrode during the power generation in a H2 fuel containing H2S impurity.

\[
\begin{align*}
\text{H}_2 + \text{O}_2^- & = \text{H}_2\text{O} + 2e^- \tag{1} \\
\text{H}_2\text{S} & = \text{H}_2 + 1/2\text{S}_2 \tag{2} \\
1/2\text{S}_2 + 3/2\text{Ni} & = 1/2\text{Ni}_2\text{S}_2 \tag{3}
\end{align*}
\]

The standard electromotive forces of each reaction for the various cell temperatures were theoretically estimated by using the Gibbs energy of the reactions calculated by the thermodynamic database MALT2. Then, the equilibrium electrode potentials were indicated against a reference electrode exposed to O2 of 1 atm. For the potential calculation, the atmospheres of cathode and anode were supposed to O2 and 10 ppm H2S/S% H2, respectively.

Figure 1 shows the estimated equilibrium electrode potential for the reaction 3 and the stable-electrode-potential regions of metallic-nickel and Ni3S2 against the cell temperature. In this roughly estimate process, for simplify, the generation reactions of the sulfur oxide and other nickel sulfides, NiS, Ni3S4, and Ni3S2, were ignored. Thus the estimated value includes a certain degree of inaccuracy. However, the estimation indicates that the required anode potential to stabilize nickel particles as metal is a feasible range in the actual SOFC.

**Experimental**

*Test cell preparation.—* Surface-polished yttria stabilized zirconia (YSZ) pellets of 17 mm in diameter and 2 mm in thickness were used for the electrolyte. Pt cathode electrodes and Pt reference electrodes were prepared on the surface and side of the pellets, respectively, by firing a pure-Pt paste at 1300°C for 1 h. An NiO paste was prepared.
by dispersing the NiO powder particles into an organic vehicle consisting with ethyl-cellulose, surfactant and organic solvent. Ni porous electrodes (10 mm diameter) or Ni model electrodes of grid-like pattern (widths of line and space : 1 mm and 1 mm) (Figure 2) were formed on the other side of the pellets through a screen-printing, a firing at 1300 °C for 1 h and a reduction at 950 °C in H2. Powders of NiO and YSZ (NiO/YSZ = 75/25 (wt)) were thoroughly mixed in an appropriate amount of water using a planetary ball mill (200 rpm, 12 h), dried in an oven at 80 °C, and made into the NiO-YSZ paste using the organic vehicle. The NiO-YSZ paste was screen-printed in diameter of 10 mm and fired at 1300 °C for 1 h. The resulted NiO-YSZ electrode was reduced to the Ni-YSZ cermet electrode at 950 °C in H2.

Characterization.— Experimental studies were performed by using the Ni porous electrode, the Ni model electrode and the Ni-YSZ cermet electrode and a mixed gas of 9.31 ppm-H2S/H2 including 3% steam as fuel. Microstructure change of the electrode was analyzed by scanning electron microscopy (SEM). Sulfur on the surface of the anode was detected by energy-dispersive X-ray spectroscopy (EDX). Crystal phase change of the anode electrode was analyzed by grazing incidence X-ray diffraction analysis (GIXRD) with a grazing incidence angle of 1° using Cu Kα radiation with D8 DISCOVER, Bruker AXS, Japan (40 kV, 40 mA) at 20 °C. Electrode performances were investigated by electrochemical analysis using an electrochemical work station (SP150, Biologic SAS). The anode electrode potential was shifted against the reference electrode exposed to O2 gas of 1 atm. The anode overpotential change by a change in the generation current was measured by a three-probe linear sweep voltammetry. The limiting current, which is the maximum current estimated by ignoring the cathode overpotential and electrolyte resistance, was compared to evaluate the performance change of the anode electrode. Anode electrode resistance was measured by a three-probe alternating current impedance method over the frequency range from 1 MHz to 100 mHz with an oscillation voltage of 10 mV on open-circuit potential. Overview of the anode electrode and test specifications are summarized in Table 1.

Results and Discussion 
Characterization of the Ni-YSZ anode electrode in the H2S-containing fuel.—High temperature poisoning.— The Ni porous

Table I. Overview of anode electrode and test specifications.

| Figures | anode electrode | atmosphere | temperature (°C), temperature change rate (°C/min) | potential application (vs reference electrode in O2) |
|---------|-----------------|------------|-----------------------------------------------|-----------------------------------------------|
| Figure 3 | Ni-model (grid) | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 950°C | initial, −0.5, −1.5, −1.6, −1.7, −1.8, −1.9, −2.0, −2.1 V (10000 sec) |
| Figure 4 | Ni-model (grid) | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 700, 950°C | initial, −1.9, −2.5, −2.6 V |
| Figure 5 | Ni-model (grid) | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 950°C | initial, −1.9 V |
| Figure 6 | Ni-model (grid) | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 950°C, 1°C/min | initial, −1.9, −2.5, −2.6 V |
| Figure 7 | Ni-model (grid) | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 400°C | initial, −1.9 V |
| Figure 8 | Ni-model (grid) | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 400°C, quench | initial, −1.9 V |
| Figure 9 | Ni-model (grid) | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 700°C | initial, −1.9, −2.5, −2.6 V |
| Figure 10 | Ni-model (grid) | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 700°C | initial, −1.9, −2.5, −2.6 V |
| Figure 11 | Ni-porous (circle), poisoned at 400°C | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 700°C | initial, −1.9, −2.5, −2.6 V |
| Figure 12 | Ni-porous (circle), poisoned at 400°C | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 700°C | initial, −1.9, −2.5, −2.6 V |
| Figure 13 | Ni-porous (circle), poisoned at 400°C | 3%-H2O/H2, 9.31ppm-H2S/3%-H2O/H2 | 700°C | initial, −1.9, −2.5, −2.6 V |
| Figure 14 | Ni-model (grid) | 9.31ppm-H2S/3%-H2O/H2 | 950°C, 1°C/min | −1.9 V |

Figure 1. Phase equilibrium of the Ni-S system. Atmospheres: anode; 1 atm-10 ppm-H2S/3%-H2O/H2, cathode; 1 atm-O2, reference; 1 atm-O2.

Figure 2. Schematic image of the test cell with Ni model anode electrode.
Figure 3. Anode overpotentials for various current values measured at just before and immediately after the fuel gas change. Fuel: 3%-humidified pure H₂ and 9.31 ppm-H₂S/H₂. Cell temperature: 950 °C.

Figure 4. Time dependency of the recovery rate of the limiting current by anode-potential shift. The recovery rate is the ratio of recovered-limiting current and initial-limiting current. □; Ni-model electrode at 950 °C, ●; Ni-model electrode at 700 °C, Δ; Ni-YSZ cermet electrode at 950 °C.

Figure 5. EDX mapping image (a) and EDX spectra (b) of the Ni model electrode quenched in N₂ purge gas after the power generation in the 3%-humidified 9.31 ppm-H₂S/H₂ fuel at 900 °C for 40 h.
Figure 6. Electrochemical impedance spectroscopy (a) Nyquist plot, (b) Bode plot of an anode electrode at open-circuit potential in the 3%-humidified atmospheres of pure H₂ and 9.31 ppm-H₂S/H₂. Cell temperature: 950°C.

used here is dense and simple shape. Hence, the impedance in the small current region including the OCV state is assigned to the charge transfer reaction. When the current is sufficiently small, the anode polarization resistance is related to the anode exchange current density. In the case of small exchange current density, the overpotential in the sufficiently small current region became larger by the introduction of H₂S into the fuel gas. It is well known that the number of the reaction site near the triple-phase boundary (TPB) of Ni, YSZ and gas phase governs the exchange current density, which means that the observed larger overpotential of the anode in the H₂S-containing fuel in the small current region including the OCV state is attributed to the diminished number of the reaction site by the adsorption of sulfur.

Consequently, the adsorbed sulfur reduced the limiting current in the high temperature region.

Low temperature poisoning. — The Ni model electrode was slowly cooled from 950°C to the room temperature in the 9.31 ppm-H₂S/H₂ fuel by a rate of 1°C/min, and then EDX mapping analysis of its cross section was performed (Figure 7). Sulfur existed on the surface of nickel particle and the interface of Ni particle and YSZ electrolyte. Since nickel atoms also existed at the same position, sulfur on the electrode ought to be present as nickel sulfide formed at the low temperature region during the slow cooling. In order to reveal the effect of the poisoning and identify the sulfide generated at the low temperature, the Ni electrode was poisoned at 400°C. The fresh Ni model electrode was cooled slowly in pure H₂ gas from 950°C to 400°C by a rate of 1°C/min, and the fuel gas was changed to 9.31 ppm-H₂S/H₂ at 400°C. The anode overpotentials for various currents at 400°C were measured before and after the fuel gas change (Figure 8). The overpotential was increased and the resultant limiting current was decreased in a short period of time for the H₂S-containing fuel introduction. Then, although the power generation with constant voltage of −0.5 V against the reference electrode potential at 400°C for 10 h by the H₂S-containing fuel was performed, significant performance degradation was not confirmed. This time dependence infers that the sulfur poisoning is happen quickly even at the low temperature around 400°C and the subsequent progress is slow. After the power generation at 400°C for 5 h in the H₂S-containing fuel, the anode electrode was quenched in N₂ purge gas and its surface was analyzed by SEM and EDX mapping (Figure 9). Many of the nickel particles became sulfide and the shape of the particle was rounded. Figure 10 is a profile of
Figure 9. SEM and EDX mapping images of the surface of the Ni anode electrode quenched in N2 purge gas after the power generation at 400 °C by constant voltage mode (−0.5 V vs reference electrode potential in O2) for 5 h in the 3%-humidified H2S-containing fuel.

CIXRD analysis of the electrode surface. The rounded particle was Ni3S2. That is, the formation of Ni3S2 is a rapid reaction which has happened in the short period of time for the temperature decreasing process. Interestingly, some of the nickel particles in the Figure 9 were metallic nickel even after 5 h of sulfur poisoning. The authors, at the moment, cannot clearly explain this phenomenon.

Performance recovery in the H2S-containing fuel of the sulfur-poisoned Ni anode electrode by sulfide generation.—The fresh Ni anode was cooled in the humidified pure H2 from 950 °C to 400 °C, and the Ni anode potential was held at −0.3 V against the reference electrode potential in the 9.31 ppm-H2S/H2 fuel including 3% steam at 400 °C for 5 h to be sulfur-poisoned. The anode electrode was heated again to 700 °C, and the various negative voltages from −0.5 V to −2.6 V against the reference electrode potential were applied to the anode electrode for 10000 s at each voltage in order to lower the anode electrode potential. Then, for each the applied voltage, the anode overpotential for various current was measured (Figure 11a). In Figure 11a, the initial limiting current at 700 °C in the humidified H2, which was measured on the way of the cooling from 950 °C to 400 °C, was also shown. Figure 11b depicts a relation between the limiting current and the applied negative voltage (anode potential). When the applied voltage was lower than −1.5 V (vs reference electrode potential in O2), the limiting current was increased. When the voltage was lowered to −1.8 V, the limiting current increased sharply with the applied voltage. The recovered performance reached approximately 90% or more of the initial performance. Considering that the performance degraded approximately 10% by the adsorption of sulfur shown in Figures 3 and 4, the anode-electrode shifting recovers the performance of the nickel anode electrode, which is poisoned by the formation of sulfide at lower temperature for the start-stop process of SOFC. Some of the adsorbed sulfur might have been removed by oxidation with the oxygen, which was formed by the electrolysis of steam under the negative potential of the anode electrode, at least temporarily.

The recovery-starting potential of the limiting current was lower than the value (approx. −1.3 V vs reference electrode potential in O2) shown in Figure 1. The deviation of the experimental value of the reduction potential from the estimated value obtained by the thermodynamic calculation is mainly due to simplification of the reaction. In actual experiment, the reaction of sulfur and the oxygen generated by electrolysis of steam (e.g., S + O2 = SO2) must have occurred also at least. However, in the thermodynamic calculation for Figure 1, the oxidative reaction of sulfur was ignored to simplify. The overvoltage, which is caused by the affected by position of the reference-electrode, might have also increased the deviation. Because the reference electrode was attached on the side of the pellet (not on the same plane to the anode electrode), some of the applied voltage might have been divided to the overvoltage by the electrolyte resistance.

Figure 10. Grazing incidence X-ray diffraction profile of the electrode surface of the Ni anode electrode quenched in N2 purge gas after the power generation at 400 °C by constant voltage mode (−0.5 V vs reference electrode potential in O2) for 5 h in the 3%-humidified H2S-containing fuel collected by Cu Kα radiation (40 kV, 40 mA) at 20 °C. Grazing incidence angle: 1°.
Figure 12. Limiting current changes at a relation with the application time at the various negative anode potentials. Cell temperature: 700°C. Atmosphere: 9.31 ppm-H2S/3%-H2O/H2. Anode electrode: Ni electrode including nickel sulfide (Ni3S2) generated at 400°C. Anode electrode potential (vs reference electrode in O2): ●, −1.9 V; □, −2.5 V; Δ, −2.6 V.

Figure 12 shows the limiting current change at 700°C in the H2S-containing fuel as a function of the application time at the various negative anode potentials. The data were obtained on the way to reach the steady value by the potential application for 18000 s (shown in Figures 11a and 11b). While an anode potential was maintained in each value, temporarily, potential application was stopped and the measurement was executed. The limiting current recovered to the almost same value after 75 min independently of the applied voltage, i.e., the required application time is almost same when the negative anode potential voltage is large enough and the sulfur-poisoning time is same of 18000 s each other. The influence of sulfur-poisoning time on the recovery time is shown in Figure 13. The recovery time was short (10 min) when the sulfur-poisoning time was limited to 1 h, although the long time of 75 min is needed for the case of long time poisoning (5 h). It is supposed that the long time exposure to the H2S containing fuel has increased the thickness of nickel sulfide layer and then the long recovery time was necessary to reduce the inner layer of the thick nickel sulfide. Therefore, in order to reduce the recovery time, the potential shift of the anode electrode should be frequently performed before the growing the thickness of nickel sulfide layer.

Figure 13. The influence of sulfur-poisoning time on the recovery time. Cell temperature: 700°C. Atmosphere: 9.31 ppm-H2S/3%-H2O/H2. Anode electrode potential (vs reference electrode in O2): −1.9 V. Anode electrode: Ni electrode including nickel sulfide (Ni3S2) generated at 400°C. Sulfur poisoning time: ○, 1 h; ●, 5 h.

Figure 14. (a) SEM and EDX images of surface and cross section of the anode electrode. (b) EDX Spectra. Ni model electrode: cooled from 950°C by the rate of 1°C/min in 9.31 ppm-H2S/3%-H2O/H2 with applying the negative voltage. Applied anode electrode potential (vs reference electrode in O2): −1.9 V.

Suppression of the sulfur poisoning by sulfide generation during the low temperature region.— The Ni model anode electrode of the SOFC using the H2S-containing fuel is poisoned by sulfide formation in the low temperature range for start-stop operation. In order to suppress the sulfur poisoning, the effect of potential control of anode electrode during the start-stop process was investigated. The fresh Ni model electrode was exposed to the H2S-containing fuel at 950°C, and subsequently cooled by the rate of 1°C/min under the condition of negative voltage of −1.9 V against the reference electrode potential. Figure 14 shows SEM and EDX analysis results of surface and cross section of the anode electrode. Sulfur did not existed in the anode
Conclusions

The explanation of sulfur poisoning of Ni-Y stabilized zirconia anode electrode has been investigated by using a 9.31 ppm-H2S/H2 fuel, and was verified as follows.

1) At high temperature around 900°C, the performance of Ni-based anode is reduced by deceasing the triple-phase boundary as the reaction site of electrochemical oxidation due to physical adsorption and/or chemical adsorption of sulfur.

2) In low temperature range, the Ni particle reacts with sulfur to form Ni3S2 to degrade its performance in a short time period after exposure to the H2S-containing fuel.

3) A simple assumed thermodynamic calculation estimates that the controlling anode electrode potential enables the suppression of the sulfur-poisoning.

4) The nickel sulfide particle in the anode electrode is reduced to metallic nickel by shifting the anode electrode potential to large negative value. By the control of anode potential, its degraded anode performance owing to the formation of nickel sulfide (Ni3S2) at low temperature is recovered even under the sulfur-containing atmosphere. For instance, the anode potential at 700°C in the 9.31 ppm-H2S/3%-H2O/H2 fuel should be -1.9 V or lower (vs reference electrode in O2) to reduce the nickel sulfide (Ni3S2) to metallic nickel for the recovery of the performance. In order to reduce the recovery time, the potential shift of the anode electrode should be frequently performed before the growing the thickness of nickel sulfide layer.

5) The nickel particle in the anode electrode maintains to be metallic nickel even in the sulfur-containing fuel at low temperature, when the anode electrode potential is controlled to the negative value. Therefore, this sulfur poisoning by the formation of nickel sulfide in the low temperature range for the start-stop operation of SOFC is prevented by the electrode potential control.

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