

## DIRECT ENERGY CONVERSION BY PROTON-CONDUCTING CERAMIC FUEL CELL SUPPLIED WITH CH<sub>4</sub> AND H<sub>2</sub>O AT 600-800°C

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### *Abstract*

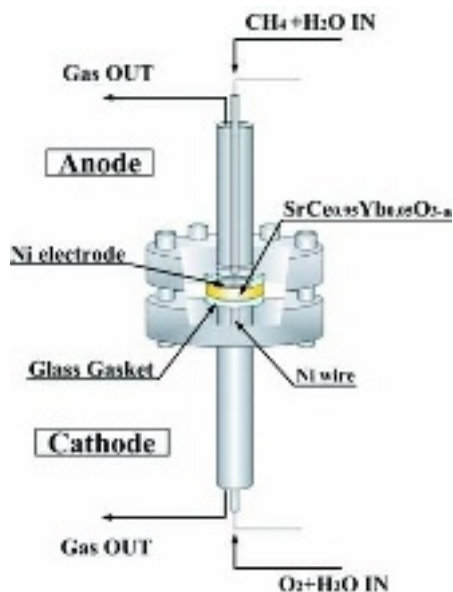
Relations between current density and terminal voltage (I-V curves) of the proton-conducting ceramic of SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-a</sub> were determined for application to a fuel cell working at 600 - 800°C. In a similar way to the introduction of a H<sub>2</sub> + H<sub>2</sub>O mixture, its anode supplied with a CH<sub>4</sub> + H<sub>2</sub>O mixture worked as a fuel cell efficiently without any external CH<sub>4</sub>-to-H<sub>2</sub> reformer. The overall electric resistance for the CH<sub>4</sub> + H<sub>2</sub>O supply was larger than that for the H<sub>2</sub> + H<sub>2</sub>O supply, because of activation over-potential of the anode electrode. Although a small amount of carbon deposited at a narrow interface between the ceramic and the Ni/SiO<sub>2</sub> electrode, it did not affect cell performance. The overall conductivity was smaller than other fuel-cell systems such as PEM-FC. Application to the recovery of tritium from process streams of a fusion reactor was discussed in terms of the protonic conductivity.

Keywords: proton-conducting ceramic, Sr-Ce-Yb oxide, fuel cell, high temperature, CH<sub>4</sub> steam reforming, internal reform.

## 1. Introduction

Some doped perovskite-type oxides exhibit proton conduction at higher temperatures. SrCeO<sub>3</sub>- or CaZrO<sub>3</sub>-based ceramics demonstrated higher proton-conducting performance at 600°C to 1 000°C [1]. Therefore, application to a hydrogen pump, a hydrogen sensor and a hydrogen (or tritium) purifier operated at 600-800°C is expected in the nuclear engineering field, especially in the tritium recovery process of fusion reactors [2-4]. In order to apply proton-conducting ceramics to produce H<sub>2</sub> and electricity for the effective use of nuclear heat as well as to recover tritium from gas streams in fusion reactors, combining steam-reforming reaction of CH<sub>4</sub> on a Ni catalyst with a proton-conducting ceramic fuel cell was studied experimentally. Especially we focused on mass and charge transfer in a SrCeO<sub>3</sub>-based proton-conducting ceramic with internal reformation on a Ni electrode under the supply of CH<sub>4</sub> + H<sub>2</sub>O. The oxide used in the present experiment was SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-a</sub>, and the electrodes were composed of a Ni wire mesh and Ni-SiO<sub>2</sub> paste comprising Ni and SiO<sub>2</sub> fine particles and a vaporable paste. The cell system was described by CH<sub>4</sub>+H<sub>2</sub>O|Ni|SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-a</sub>|Ni|O<sub>2</sub>+H<sub>2</sub>O. Several I-V characteristic curves (i.e., electric current density, I, versus cell potential, V) were determined for different H<sub>2</sub>O/CH<sub>4</sub> concentration ratios in the temperature range of 600 to 800°C. A thermodynamic electromotive force, E<sub>0</sub>, and an overall electric conductivity, σ, were determined from the intercept on the y-axis of an I-V curve and its slope, respectively. These results were compared with our previous results of the direct and alternating current methods [4,5]. They were determined under the condition where a similar cell system composed of SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-a</sub> and porous Ni/SiO<sub>2</sub> electrodes was supplied with a mixture of H<sub>2</sub> + H<sub>2</sub>O in the anode and with a mixture of O<sub>2</sub> + H<sub>2</sub>O in the cathode. We observed surfaces in/on the porous anode electrode using a SEM (Scanning Electron Microscope) and an EDX (Energy Dispersive X-ray spectroscopy), and we investigated whether or not carbon deposited in/on the porous Ni/SiO<sub>2</sub> electrode.

Figure 1. Experimental apparatus of a fuel



## 2. Experimental

Figure 1 shows a schematic illustration of the fuel-cell system used in the present study. Ni-SiO<sub>2</sub> paste (Aremco Corp. Pyro-Duct 598) was painted on both surfaces of a SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-a</sub> ceramic pellet with Ni wire mesh. The surface area and thickness of the ceramic pellet was 1.77 cm<sup>2</sup> and 3.5 mm. The ceramic was calcinated in an electric heater at 1 000°C for several hours.

**Figure 2. SEM photo of NiO/SiO<sub>2</sub> electrode**

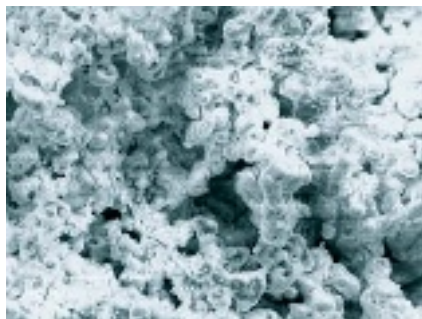
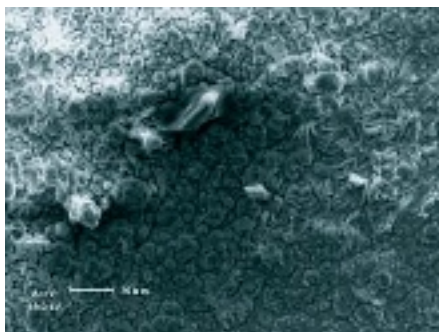


Figure 2 shows a SEM photo of a porous NiO/SiO<sub>2</sub> electrode. The Ni/SiO<sub>2</sub> electrode in the anode was reduced under H<sub>2</sub> atmosphere at 800°C for several hours. Figure 3 shows a SEM photo of the SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-a</sub> electrolyte. A structure of multi-crystal ceramic was observed in this photo.

**Figure 3. SEM photo of SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-a</sub>**



The ceramic pellet with the porous Ni/SiO<sub>2</sub> electrodes was placed in a 316 stainless-steel holder. A glass gasket was used in order to eliminate gas leak through interfaces between the anode and cathode. The anode was supplied with a gas mixture of H<sub>2</sub> + H<sub>2</sub>O or CH<sub>4</sub> + H<sub>2</sub>O. The H<sub>2</sub> concentration was 20%. The CH<sub>4</sub> concentration was 10%. Results for other H<sub>2</sub> or CH<sub>4</sub> concentrations were reported elsewhere [4,5]. H<sub>2</sub>O was added to the CH<sub>4</sub> or H<sub>2</sub> gas flow by a bubbler immersed in a constant temperature bath. The cathode was supplied with a mixture of O<sub>2</sub> and H<sub>2</sub>O. The H<sub>2</sub>O concentration in the cathode was 20%. The concentrations of H<sub>2</sub>, O<sub>2</sub>, CH<sub>4</sub>, CO, CO<sub>2</sub> and other hydrocarbons at the outlets of the anode and cathode were detected by gas chromatography. The H<sub>2</sub>O concentration was measured by a dew-point meter. The gas flow rate was controlled to a constant flow rate by mass-flow regulators. The fuel-cell holder was placed in an electric furnace controlled to a constant temperature. Temperature was measured by C-A thermocouples. I-V curves were determined by a two-terminal direct-current method.

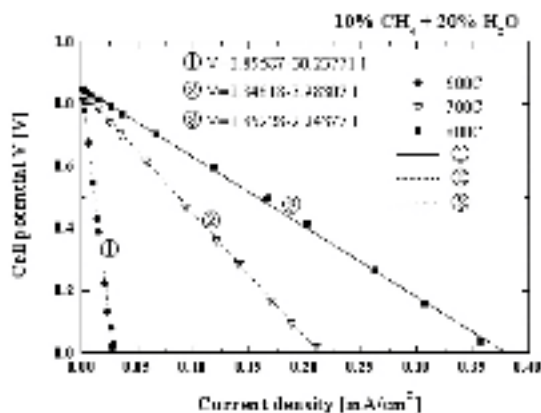
### 3. Results and Discussion

#### (1) I-V curves

Figures 4 and 5 show typical I-V curves for the Sr-Ce-Yb fuel-cell system. The former figure shows results for different temperatures and the latter does those for different input CH<sub>4</sub>/H<sub>2</sub>O ratios. As seen in the two figures, the whole I-V curves were correlated to the following relation:

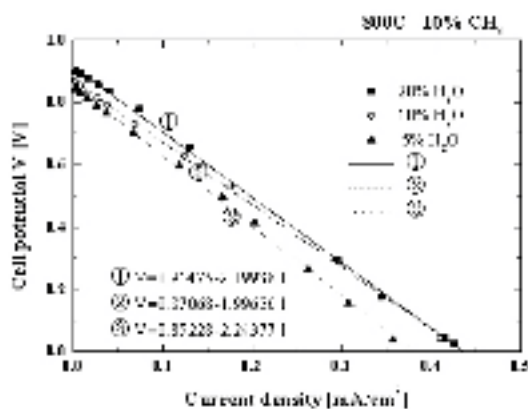
$$V = E_0 - Id/\sigma \quad (1)$$

**Figure 4. I-V curves for CH<sub>4</sub>+H<sub>2</sub>O|Ni|SrCe<sub>0.95</sub>YbO<sub>3- $\delta$ }|NiO|O<sub>2</sub>+H<sub>2</sub>O cell</sub>**



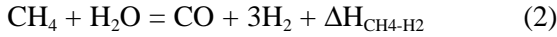
Here,  $V$  and  $E_0$  are cell potentials or terminal voltage at arbitrary current density  $I$  and  $I = 0$ . Usually,  $E_0$  is called a thermodynamic electromotive force, EMF. The notations of  $A$  and  $\delta$  are a surface area and thickness of the ceramic electrolyte, and  $s$  is an electric conductivity. Since measurement was carried out by a direct current method, the  $s$  value corresponded to an overall one including the contributions of anode and cathode polarities as well as the protonic conductivity of ceramic electrolyte. The dependence of  $E_0$  on temperature was very small and almost independent of it. On the other hand, the  $E_0$  values slightly depended on the input CH<sub>4</sub>/H<sub>2</sub>O ratio.

**Figure 5. I-V curves for CH<sub>4</sub>+H<sub>2</sub>O|Ni|SrCe<sub>0.95</sub>YbO<sub>3- $\delta$ }|NiO|O<sub>2</sub>+H<sub>2</sub>O cell</sub>**



## (2) Correlations of $E_0$ and $\sigma$

It was found that the system could work well even without any external  $\text{CH}_4$ -to- $\text{H}_2$  reformer. Figure 6 shows a relation of  $E_0$  and the partial pressure of  $\text{H}_2\text{O}$ ,  $p_{\text{H}_2\text{O}}$ , supplied to the anode. As seen in the figure,  $E_0$  (on the left-hand-side axis) slightly increased with an increase in  $p_{\text{H}_2\text{O}}$ . This is because the steam-reforming reaction proceeded more with elevating temperature. This increase was attributable to a difference in the  $\text{H}_2$  partial pressure,  $p_{\text{H}_2}$ , on the anode electrode. When a  $\text{CH}_4$  and  $\text{H}_2\text{O}$  mixture was introduced into the anode, the following  $\text{CH}_4$  steam-reforming reaction occurred as well as other by-product reactions [6,7].



The  $E_0$  values determined were compared with the following Nernst equation [8].

$$E_0 = -\frac{\Delta G_{\text{H}_2\text{O}}}{2F} - \frac{R_g T}{2F} \ln \left( \frac{p_{\text{H}_2\text{O}, \text{cathode}}}{p_{\text{H}_2, \text{anode}} p_{\text{O}_2, \text{cathode}}^{0.5}} \right) \quad (3)$$

Here,  $\Delta G_{\text{H}_2\text{O}}$  is a Gibbs free-energy change of the reaction of  $\text{H}_2 + (1/2)\text{O}_2 = \text{H}_2\text{O} + \Delta H_{\text{H}_2\text{O}}$ , and its value was given in the reference [9]. The  $p_{\text{H}_2, \text{anode}}$  values determined using Eq. (3) were plotted on the right-hand-side axis of Figure 6. Since the residence time of the  $\text{CH}_4 + \text{H}_2\text{O}$  gas in the anode was around one second, the exhaust gas was not under equilibrium condition. Since the order of reaction was the first on  $p_{\text{H}_2\text{O}}$  [10,11], the partial pressure of  $\text{H}_2$  produced in the anode should be in proportion to  $p_{\text{H}_2\text{O}}$ . The experimental results also showed a proportional relation between  $p_{\text{H}_2\text{O}}$  supplied and  $p_{\text{H}_2}$  produced. Therefore,  $p_{\text{H}_2, \text{anode}}$  was deeply related with the rate of the steam-reforming reaction of Eq. (2). This will be investigated also in the following discussion on  $\sigma$ .

**Figure 6. Relation of EMF and  $p_{\text{H}_2}$  versus  $p_{\text{H}_2\text{O}}$**

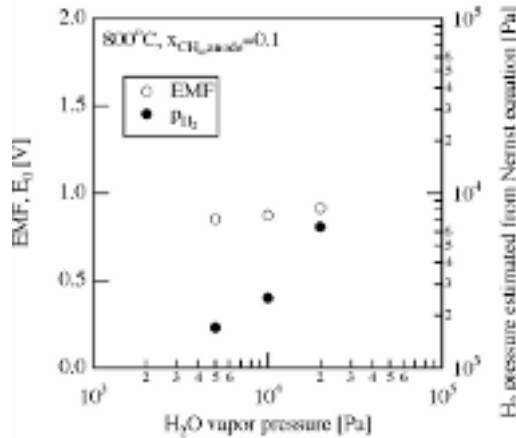
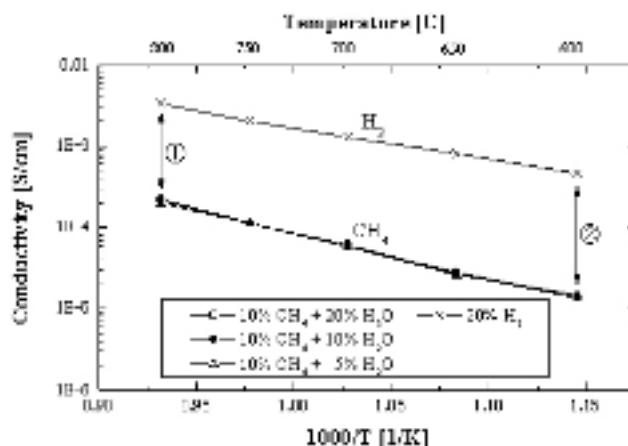


Figure 7 shows a comparison of  $\sigma$  between the  $\text{CH}_4 + \text{H}_2\text{O}$  and  $\text{H}_2 + \text{H}_2\text{O}$  supplies. As seen in the figure,  $\sigma$  for the  $\text{CH}_4 + \text{H}_2\text{O}$  supply was much smaller than that for the  $\text{H}_2 + \text{H}_2\text{O}$  one. The ratio became over one-tenth, and the  $\sigma$  values for the  $\text{CH}_4 + \text{H}_2\text{O}$  supply were independent of  $p_{\text{H}_2\text{O}}$ . Referring to the discussion in our previous paper [4], where complex impedance plots were obtained in the system of a  $\text{SrCe}_{0.95}\text{Yb}_{0.05}\text{O}_{3-a}$  fuel cell supplied with  $\text{H}_2 + \text{H}_2\text{O}$  mixtures, the rate-determining step of the overall charge transfer was  $\text{H}^+$  diffusion in the ceramic electrolyte. Therefore, a decrease in  $\sigma$  for the  $\text{CH}_4 + \text{H}_2\text{O}$  supply was not caused by the  $\text{H}^+$  conduction in the ceramic electrolyte. This is because the cathode was under the same condition between the  $\text{H}_2 + \text{H}_2\text{O}$  and  $\text{CH}_4 + \text{H}_2\text{O}$  supplies. The second contribution

was the oxidation reaction of  $H_2$  in/on the cathode. However, it could not be also explained by the same reason. Therefore, the rate-determining step of the charge transfer for the  $CH_4 + H_2O$  supply should be related to the steam-reforming reaction of  $CH_4$  on the anode electrode.

**Figure 7. Comparison of conductivity between  $CH_4+H_2O$  and  $H_2-H_2O$  supplies**



**Figure 8. Mass and charge transfer on proton-conducting ceramics fuel cell**

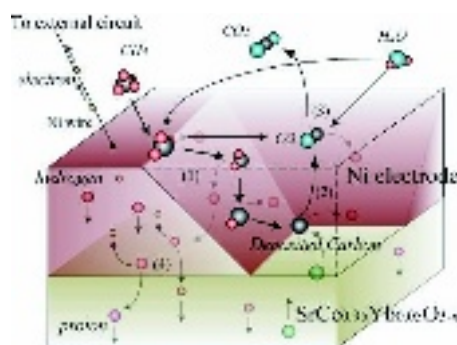


Figure 8 shows a schematic illustration of the anode reaction in the present proton-conducting ceramic fuel-cell system. In the anode, the following reactions may occur simultaneously:

- (i) convection of  $CH_4$  and  $H_2O$  in the anode,
- (ii)  $CH_4$  and  $H_2O$  reaction on/in the porous  $Ni/SiO_2$  electrode,
- (iii)  $CH_4$  and  $H_2O$  diffusing through the porous  $Ni/SiO_2$  electrode,
- (iv) reaction product of  $CO$ ,  $CO_2$  and so on diffusing in a counter-current way,
- (v) decomposition to  $H$  atom,
- (vi)  $H_2$  or  $H$  diffusion in the porous  $Ni/SiO_2$  electrode,
- (vii)  $H^+$  diffusion through the ceramic electrolyte.

The activation energy of  $\sigma$  was 106 kJ/mol for the  $\text{CH}_4 + \text{H}_2\text{O}$  supply and 75.6 kJ/mol for the  $\text{H}_2 + \text{H}_2\text{O}$  supply. Difference between the two energies was 30 kJ/mol. The value was in close agreement with the activation energy of the reaction-rate constant of Eq. (2) [10]. Therefore, the largest contribution to  $\sigma$ , in other words the rate-determining step, was considered the intrinsic rate of Eq. (2). It was clear that the steam-reforming reaction contributed heavily to the overall conductivity.

### (3) Carbon deposition

**Figure 9. Profiles of carbon deposition in/on porous Ni electrode**

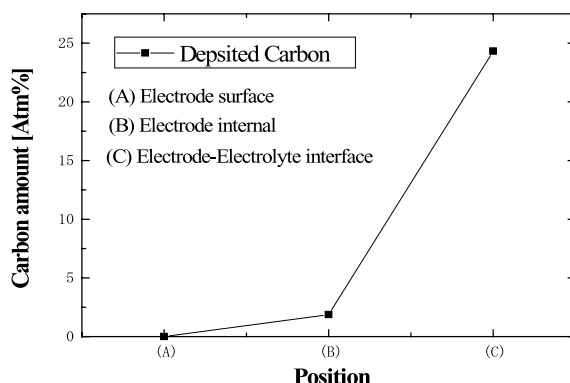


Figure 9 shows the profile of carbon deposition in the porous Ni/SiO<sub>2</sub> electrode determined using SEM-EDX. There was no carbon deposition on its surface. There were also very few carbon depositions in the porous electrode. This was because the following direct decomposition reaction did not occur in/on the porous electrode.



The ratio of the CH<sub>4</sub>-to-H<sub>2</sub> direct decomposition rate to that of the CH<sub>4</sub> steam-reforming reaction one was not found to be different from location to location in the porous electrode. Therefore, it was considered that the supply of H<sub>2</sub>O was sufficient in the present experiment. It was probable that a sufficient amount of CH<sub>4</sub> and H<sub>2</sub>O diffused through the porous electrode and arrived at an electrode-electrolyte interface. The direct decomposition reaction as well as the steam-reforming reaction simultaneously occurred at a narrow interface under the condition of the sufficient supply of electric charge to the cathode electrode. As seen in Figure 9, some carbon deposited at the narrow interface between the electrolyte and electrode, This may be because the steam-reforming reaction was slower than the protonic conductivity of electrolyte and, consequently, the direct decomposition of CH<sub>4</sub> provided electron and H<sup>+</sup> ion to the interface. Since the carbon deposition was localized at the interface. Therefore, there was no effect on the mass and charge transfer in the present cell system.

The Sr-Ce oxides were stable at 600-800°C when H<sub>2</sub>O was added in the streams of anode and cathode. The addition of H<sub>2</sub>O was necessary in order to maintain protonic conduction through the ceramic electrolyte [4]. However, it is known that Sr-Ce oxides decomposed to SrCO<sub>3</sub> and CeO<sub>2</sub> at high CO<sub>2</sub> atmosphere [12]. Therefore, the use of the Sr-Ce oxide is not proper under the condition of CO<sub>2</sub> atmosphere. In the present study, gas composition detected in the exhaust from the anode was CH<sub>4</sub>, H<sub>2</sub>, H<sub>2</sub>O, and CO. No CO<sub>2</sub> was observed throughout the present study. This may be because the reaction temperature was higher than 600°C, and a sufficient amount of H<sub>2</sub>O was supplied to the anode and cathode. There was high possibility of production of CO<sub>2</sub> at lower temperatures of 300-500°C [7]. The use of a compact Ni electrode in place of the porous Ni/SiO<sub>2</sub> electrode may be proper if CO<sub>2</sub> is

generated in the anode. The ceramic electrode with a compact Ni electrode that was electrically deposited will not contact with CO<sub>2</sub> directly. Therefore, there may be no possibility of the decomposition to SrCO<sub>3</sub> and CeO<sub>2</sub>.

#### **(4) Application of Sr-Ce-Yb oxide to high-temperature system**

There are several advantages and disadvantages for the present Sr-Ce-Yb oxide fuel-cell system. The largest advantage is that it can work at 600-800°C, at which the steam-reforming reaction simultaneously occurred. The internal reformation became possible at these temperatures. Therefore, its structure became very simple. Heat to maintain the steam-reforming reaction can be converted to electricity directly because of endothermic reaction of Eq. (2). This thing makes it possible to convert the chemical energy of CH<sub>4</sub> to electricity with a higher efficiency. The Carnot-cycle efficiency can be raised by operation at higher temperatures. Its working temperature is near that of a high-temperature nuclear reactor. Therefore, it may be possible to provide measures to utilise nuclear heat with high efficiency. This thinking is taken into consideration in other countries in different ways. There are applications of some ceramics to high-temperature steam electrolysis with use of nuclear heat, such as Generation IV nuclear energy system [13].

On the other hand, the largest disadvantage is that the protonic resistance of the Sr-Ce-Yb oxide was comparatively larger than that of a polymer-electrolyte-membrane fuel cell (PEM-FC) and was comparable with O<sup>2-</sup> ion conductivity of an yttria-stabilised zirconia (YSZ). Consequently, as seen in Figs. 4 and 5, the current density through the Sr-Ce-Yb oxide fuel cell was order of mA/cm<sup>2</sup> and was much smaller than that of PEM-FC. This is because a thin ceramic is very difficult to manufacture. The protonic conductivity of the Sr-Ce-Yb oxide itself was around one-tenth smaller than that of PEM. Moreover, the conductivity was order of 10<sup>-4</sup> S/cm when a CH<sub>4</sub> and H<sub>2</sub>O mixture was supplied directly to the cell without external reformer. The overall conductivity became around 10<sup>-2</sup>-fold less than that of PEM, because the rate-controlling step was in the steam-reforming reaction.

Judging from the data available in the present study, it is difficult to apply the Sr-Ce-Yb oxide (as it is) to the effective use of nuclear reactor heat, where a large amount heat should be converted to electricity. Considering H<sub>2</sub> production rate, the current density of 1 mA/cm<sup>2</sup> corresponds to the H<sub>2</sub> production rate of 1.2x10<sup>-6</sup> Nm<sup>3</sup>/m<sup>2</sup>s. Therefore, the surface area of 8.6x10<sup>5</sup> m<sup>2</sup> is necessary for the H<sub>2</sub> production rate of 1 Nm<sup>3</sup>/s. This is an unrealistic scale. On the other hand, it is desirable to utilise the proton-conducting ceramic under special or severe conditions, such as hydrogen or tritium pump at higher temperatures. Converting to tritium activity, the current density of 1 mA/cm<sup>2</sup> corresponds to 11 MBq/cm<sup>2</sup>s. Since the tritium generation rate in a blanket to maintain 1 GW commercial fusion power reactor is 0.63 TBq/s, the surface area of the Sr-Ce-Yb oxide necessary to extract tritium from CH<sub>4</sub> – H<sub>2</sub>O or H<sub>2</sub> – H<sub>2</sub>O streams as a tritium pump amounts to 5.7 m<sup>2</sup>. It is expected that the tritium pump will be operated at 600-800°C in a blanket tritium recovery system or a tritium purification system in a fusion fuel cycle. This is a realistic scale.

## **4. Conclusions**

The I-V curves for the fuel-cell system composed of a SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-a</sub> ceramic electrolyte and Ni/SiO<sub>2</sub> porous electrodes were determined under the conditions of CH<sub>4</sub> + H<sub>2</sub>O and H<sub>2</sub> + H<sub>2</sub>O supplies, and the values of E<sub>0</sub> and σ were correlated to a function of temperature and the anode H<sub>2</sub>O partial pressure. The E<sub>0</sub> values were consistent with the Nernst equation. It was found that E<sub>0</sub> was a good indication of p<sub>H2</sub> generated on the anode electrode when the CH<sub>4</sub> and H<sub>2</sub>O mixture was introduced into the anode. The σ values determined included the two contributions of the protonic conductivity of the



Sr-Ce-Yb oxide electrolyte and the activation polarity of the steam-reforming reaction on the Ni/SiO<sub>2</sub> porous electrode. Because of stable operation of the Sr-Ce-Yb oxide as a fuel cell at 600-800°C, it is expected to utilise the ceramic for tritium pump. The profile of carbon depositions were determined by SEM-EDX, and it was found that carbon depositions by the direct decomposition of CH<sub>4</sub> were localised at a narrow interface between the electrode and electrolyte.

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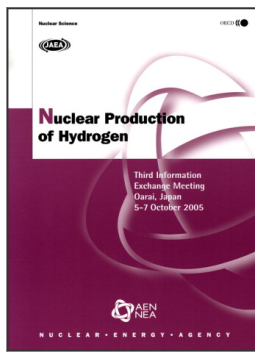
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