Suppression of Chromium Diffusion to an SOFC Cathode from an Alloy Separator by a Cathode Second Layer

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Received December 8, 1995 ; Accepted February 13, 1996

Chromium oxide vapor species that influence cathode degradation were studied and the effect of a cathode second layer that suppresses chromium diffusion to the cathode was investigated in an SOFC using an alloy separator. $P_{CrO_2(OH)_2}$ has the highest value at high P_{H_2O} condition and P_{CrO_3} becomes highest at low P_{H_2O} condition ($\sim 10^{-4}$ atm). The cathode degradation rate seemed to be influenced by the total vapor pressure of all gaseous species of chromium. The P_{H_2O} dependence of the cell life indicated that cathode degradation can be reduced by decreasing P_{H_2O} so far as that $P_{CrO_2(OH)_2}$ is equal to P_{CrO_3} . Chromium diffusion to the cathode was suppressed by adopting a cathode second layer that contains La_2O_3 . This effect seemed to be caused by the reaction of La_2O_3 with the chromium vapor which decreased the chromium vapor pressure in the cathode. The life of the cell with the cathode second layer was extended by decreasing the cathode polarization and the chromium vapor pressure from the alloy. Almost all La_2O_3 in the cathode second layer seemed to react with the chromium vapor and contribute to the increase in cell life.

1 INTRODUCTION

Planar-type solid oxide fuel cells (SOFCs) have received a great deal of attention because of their simple manufacturing process and the promise of a higher power density than that available from corresponding tubular designs.

For the separator material, a heat-resistant alloy such as Inconel 600 must offer good performance in terms of electrical and heat conductivity, gas-tightness, mechanical strength, and ease of manufacturing. Furthermore, its material cost is much lower than $LaCrO_3$ -based ceramics. It has been pointed out, however, that chromium diffuses from the alloy to the cathode at the operating condition and degrades the characteristics of the electrode.

The authors have studied the degradation phenomena induced by chromium in the cathode and clarified the following phenomena¹⁾:

- 1) Chromium vaporizes from the surface oxide layer of the alloy separator.
- 2) Chromium is deposited at the cathode/electrolyte interface under the action of the discharge current and increases the cathode polarization.
- 3) Chromium deposition at the cathode/electrolyte interface is caused by the decrease in oxygen activity at the interface ($\Delta \log a_o$). $\Delta \log a_o$ is calculated from:

$$\Delta \log a_o = - (2 \eta F / RT) \log e \quad (1)$$
 where η is the cathode polarization that is caused by the discharge current.

Chromium was thought to diffuse to the cathode in the gas state. In ref.2, equilibrium vapor pressures calculated by thermodynamic data for various chromium oxide gaseous species were reported. Among those species, the vapor pressure of $CrO_2(OH)_2(g)$ ($P_{CrO_2(OH)_2}$) has the highest value at high $H_2O(g)$ pressure (P_{H_2O}) condition³⁾. $P_{CrO_2(OH)_2}$ decreases with decreasing P_{H_2O} as shown in Fig. 1. $CrO_2(OH)_2(g)$ and $CrO_3(g)$ are

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Key Words: Solid oxide fuel cells, Cathodes,
Degradation, Chromium

Fig. 1 Calculated P_{CrO_3} and $P_{CrO_2(OH)_2}$.²⁾

produced by the following reactions:

Therefore, the chromium diffusion to the cathode is expected to be decreased by decreasing P_{H_2O} in the cathode gas. In section 3.1, the influence of P_{H_2O} in the cathode gas on chromium vaporization and cathode degradation is discussed.

In order to suppress chromium diffusion to the cathode, the authors have devised a cathode second layer that contains La_2O_3 . Long-range cell performance was improved by adopting the cathode second layer. This effect seemed to be caused by a reaction of La_2O_3 with the chromium gas to decrease the chromium vapor pressure. For practical use, surface treatment for the alloy is necessary, but the cathode second layer is also necessary because it is probable that the chromium diffusion from the alloy separator cannot be suppressed perfectly. In section 3.2, a quantitative evaluation of the relationship between chromium diffusion and the effect of the cathode second layer is described.

2 EXPERIMENTAL

The test cells were constructed as follows. The electrolyte was made from PSZ (3 mol% Y_2O_3 + 97 mol% ZrO_2) and had a thickness of 200 μm . Anode material comprised of NiO 1 μm , 56 wt.% + YSZ (8 mol% Y_2O_3 + 92 mol% ZrO_2) 0.5 μm , 44 wt.% was painted on the surface of the electrolyte. It was then sintered at 1573 K for 2 h. $La_{0.9}Sr_{0.1}MnO_3$ 1 μm , 80 wt.% + YSZ 0.5 μm , 20 wt.% was used for the cathode material. It was painted on the other side and sintered at 1373 K for 4 h. The

Fig. 2 Schematic of the design of the test cell.

electrode had a diameter of 8 mm. The reference electrode was on the cathode side. The current collector was a platinum mesh.

The cathode polarization η was measured excluding ohmic drop by current-interruption method. In the following experiments, the cathode degradation was evaluated by the cell voltage because the cell performance degradation was induced only by chromium deposition at the cathode/electrolyte interface¹⁾

In section 3.1, the influence of P_{H_2O} in the cathode gas on the volatility of chromium and the cathode degradation was investigated. The thickness of the cathode was 100 μm in the test cells. In order to evaluate the influence of chromium that diffuses from the alloy to the cathode, Cr_2O_3 tablets were put on the cathode as shown in Fig. 2. The diameter of the Cr_2O_3 tablets was 10 mm and they were press-formed and sintered at 1673 K. Test cells were operated at a constant current density j of 0.3 A/cm² at 1073 K or 1173 K with air that contains $H_2O(g)$ (cathode) and $H_2+3\%H_2O$ (anode) gas. P_{H_2O} in the cathode gas was controlled by cooling and bubbling the gas at a prescribed temperature.

In section 3.2, the effect of a cathode second layer was evaluated. $La_{0.9}Sr_{0.1}MnO_3$ 1 μm , 90 wt.% + La_2O_3 0.5 μm , 10 wt.% was used for the cathode second layer material. It was painted on the cathode and was not sintered. The current was collected by the platinum paste and the current collector. The thickness of the cathode was 50 μm and that of the cathode second layer was 80 μm . Test cells were operated at 1073~1173 K with

various current densities to change the vapor pressure of the chromium and the decrease in oxygen activity at the cathode/electrolyte interface. A CoCr_2O_4 tablet was also used to change the chromium vapor pressure. The $P_{\text{H}_2\text{O}}$ in the cathode gas was kept constant at 1.43×10^{-3} atm.

3 RESULTS AND DISCUSSION

3.1 Influence of H_2O vapor pressure on chromium volatility and cathode degradation

Figure 3 shows the change in cell voltage with time at 1073 K for $P_{\text{H}_2\text{O}}$ in the cathode gas. Cell performance degradation was caused by chromium deposition at the cathode/electrolyte interface and it was severer than at higher operating temperatures¹⁾. This is mainly due to higher cathode polarization η of the lanthanum manganite cathode at lower temperatures. That is, the chromium deposition at the cathode/electrolyte interface caused by $\Delta \log a_o$ (eq. (1)) becomes severer because $\Delta \log a_o$ decreases with decreasing operating

Fig. 3 Cell voltage (V) at 1073 K for $P_{\text{H}_2\text{O}}$ in the cathode gas. (■): $P_{\text{H}_2\text{O}} \sim 1 \times 10^{-5}$ atm; (□): $P_{\text{H}_2\text{O}} \sim 1 \times 10^{-4}$ atm; (◇): $P_{\text{H}_2\text{O}} = 1.43 \times 10^{-3}$ atm; (◆): $P_{\text{H}_2\text{O}} = 5.55 \times 10^{-2}$ atm; (○): $P_{\text{H}_2\text{O}} = 3.08 \times 10^{-1}$ atm.

Fig. 4 Calculated chromium vapor pressures and experimental cell degradation rates at 1073 K for $P_{\text{H}_2\text{O}}$ in the cathode gas.

temperature. The cell performance degraded faster with increasing $P_{\text{H}_2\text{O}}$. The relationship between the calculated P_{CrO_3} , $P_{\text{CrO}_2(\text{OH})_2}$ ²⁾ at 1073 K and $P_{\text{H}_2\text{O}}$ is shown in Fig. 4. $\text{CrO}_3(\text{g})$ or $\text{CrO}_2(\text{OH})_2(\text{g})$ has the highest vapor pressure of all gaseous species containing chromium. In Fig. 4, cell degradation rates were also plotted. The cell degradation rates were defined as the reciprocal of the cell life t_L , where t_L was defined as the time until the cell voltage decreased to -0.3 V in Fig. 3. Figure 4 indicates that the gaseous species that induces cathode degradation is mainly $\text{CrO}_2(\text{OH})_2(\text{g})$ in this region. $P_{\text{H}_2\text{O}}$ in atmospheric air is generally in the order of $3 \times 10^{-3} \sim 4 \times 10^{-2}$ atm. (Temperature, 273 K, relative humidity, 50 % and 308 K, 80 % are assumed.) Therefore, the cell performance degradation can be reduced by decreasing $P_{\text{H}_2\text{O}}$ when using atmospheric air as the cathode gas because $P_{\text{CrO}_2(\text{OH})_2}$ decreases with decreasing $P_{\text{H}_2\text{O}}$.

Figure 5 shows the relationship between the calculated P_{CrO_3} , $P_{\text{CrO}_2(\text{OH})_2}$, the cell degradation rates at 1173 K and $P_{\text{H}_2\text{O}}$. In this case, the $P_{\text{H}_2\text{O}}$ at which $P_{\text{CrO}_2(\text{OH})_2}$ equals P_{CrO_3} ($\equiv P_{\text{H}_2\text{O}}^*$) is a little higher than at 1073 K. The cell degradation rates were found to be independent on the $P_{\text{H}_2\text{O}}$ at low $P_{\text{H}_2\text{O}}$ region. The $P_{\text{H}_2\text{O}}$ dependence of the cell degradation rates indicates that cathode degradation was induced mainly by $\text{CrO}_3(\text{g})$ in low $P_{\text{H}_2\text{O}}$ region and mainly by $\text{CrO}_2(\text{OH})_2(\text{g})$ in high $P_{\text{H}_2\text{O}}$ region.

In consideration of Fig. 4 and Fig. 5, it was suggested that the cathode degradation rate is influenced by the total vapor pressure of all gaseous species of chromium, where the total vapor pressure is nearly equal to

$$P_{(\text{Cr})} \equiv P_{\text{CrO}_2(\text{OH})_2} + P_{\text{CrO}_3}. \quad (4)$$

$P_{(\text{Cr})}$ decreases by decreasing $P_{\text{H}_2\text{O}}$ so far as $P_{\text{H}_2\text{O}}^*$. Therefore, cathode degradation induced by chromium

Fig. 5 Calculated chromium vapor pressures and experimental cell degradation rates at 1173 K for $P_{\text{H}_2\text{O}}$ in the cathode gas.

Fig. 6 Calculated chromium vapor pressures at 1273 K for P_{H_2O} in the cathode gas.

Fig. 7 H_2O vapor pressure at which $P_{CrO_2(OH)_2}$ equals P_{CrO_3} .

can be reduced by decreasing P_{H_2O} so far as $P_{H_2O}^*$

Figure 6 shows the relationship between the calculated P_{CrO_3} , $P_{CrO_2(OH)_2}$ at 1273 K and P_{H_2O} . The $P_{H_2O}^*$ at 1273 K is higher than at 1073, 1173 K. Considering the total chromium vapor pressure, the cathode degradation can be reduced by decreasing P_{H_2O} so far as $P_{H_2O}^*$ at any temperature. The temperature dependence of $P_{H_2O}^*$ is shown in Fig. 7.

On the basis of the above data and ref. 1, Fig. 8 and Fig. 9 are obtained. Fig. 8 shows the relationship between the ratio of the intensity of chromium at the cathode/electrolyte interface measured by EPMA to the $P_{(Cr)}$ and the $\Delta \log a_o$. $\Delta \log a_o$ in Fig. 8 is calculated by eq. (1) using initial η which is not influenced by chromium deposition although $\Delta \log a_o$ changes with time by the cathode degradation. The $P_{(Cr)}$ is nearly equal to the highest pressure of all chromium vapor species. Fig. 8 indicates the following phenomena. Chromium diffusion occurs mainly in the state of $CrO_2(OH)_2(g)$ in high P_{H_2O} region and $CrO_3(g)$ in low P_{H_2O} region. The gas diffuses into the cathode and chromium is deposited at the cathode/electrolyte interface due to $\Delta \log a_o$ which is caused by the discharge current.

Fig. 8 Relationship between the ratio of the intensity of chromium (I_{Cr} / kcps ($\text{kcps} = 10^3 \text{ counts} \cdot \text{s}^{-1}$)) at the cathode/electrolyte interface to the calculated $P_{(Cr)}$ and the decrease in oxygen activity at the interface from the gas phase. (○): 1373 K, $P_{O_2} = 0.21$ atm; (◇): 1273 K, $P_{O_2} = 0.21$ atm; (□): 1173 K, $P_{O_2} = 1.00$ atm; (○) (◇) (□): 1173 K, $P_{O_2} = 0.21$ atm; (△): 1173 K, $P_{O_2} = 0.05$ atm; (●): 1123 K, $P_{O_2} = 0.21$ atm.

Fig. 9 Chromium vaporization and displacement process.

Fig. 10 Image of the effect of the cathode second layer.

3.2 Evaluation of the effect of a cathode second layer

In order to reduce the cathode degradation from chromium, a cathode second layer containing La_2O_3 was formed on the cathode. La_2O_3 is expected to react with the chromium to form $LaCrO_3$ and decrease the

Table 1 Experimental conditions in section 3.2.

T / K	Oxide	$P_{\text{H}_2\text{O}}$ / atm	$P_{\text{CrO}_2(\text{OH})_2}$ / atm	P_{CrO_3} / atm	j / $\text{A} \cdot \text{cm}^2$	η / V	$\Delta \log a_o$
1073	Cr_2O_3	1.43×10^{-3}	2.94×10^{-8}	1.79×10^{-9}	0.120	0.120	-1.127
1073	Cr_2O_3	1.43×10^{-3}	2.94×10^{-8}	1.79×10^{-9}	0.180	0.175	-1.644
1073	Cr_2O_3	1.43×10^{-3}	2.94×10^{-8}	1.79×10^{-9}	0.200	0.200	-1.879
1073	Cr_2O_3	1.43×10^{-3}	2.94×10^{-8}	1.79×10^{-9}	0.240	0.240	-2.254
1073	Cr_2O_3	1.43×10^{-3}	2.94×10^{-8}	1.79×10^{-9}	0.345	0.310	-2.912
1073	CoCr_2O_4	1.43×10^{-3}	9.41×10^{-9}	5.74×10^{-10}	0.120	0.120	-1.127
1073	Cr_2O_3	1.43×10^{-3}	2.94×10^{-8}	1.79×10^{-9}	0.120	0.120	-1.127
1123	Cr_2O_3	1.43×10^{-3}	4.14×10^{-8}	5.96×10^{-9}	0.240	0.125	-1.127
1173	Cr_2O_3	1.43×10^{-3}	5.69×10^{-8}	1.79×10^{-8}	0.300	0.130	-1.127

Fig. 11 Cell voltages for given $\Delta \log a_o$ at 1073 K. $\Delta \log a_o$ was changed by the cathode polarization η . (■): $j = 0.120 \text{ A/cm}^2$; (---): $j = 0.120 \text{ A/cm}^2$, without the cathode second layer; (◇): $j = 0.180 \text{ A/cm}^2$; (○): $j = 0.200 \text{ A/cm}^2$; (●): $j = 0.240 \text{ A/cm}^2$; (△): $j = 0.345 \text{ A/cm}^2$.

chromium vapor pressure in the cathode (Fig. 10). It was not examined whether the hygroscopic property of La_2O_3 has an influence on the stability of the cathode second layer at room temperature. It has been confirmed, however, that cell performance was improved with increasing La_2O_3 content in the cathode second layer⁴⁾.

To evaluate this effect quantitatively, the following two experiments were performed. One is to evaluate the influence of $\Delta \log a_o$ on the cell life and the other is to evaluate the influence of chromium vapor pressure. The experimental conditions are shown in table 1. $\Delta \log a_o$ in this section is calculated by eq. (1) using initial η which is not influenced by chromium deposition although $\Delta \log a_o$ changes with time by the cathode degradation.

Figure 11 shows the change in cell voltage with time for the current density at 1073 K. The cell performance

Fig. 12 Relationship between the cell life t_L and $\Delta \log a_o$. Each cell performance was plotted in Fig. 11.

without the cathode second layer at 0.120 A/cm^2 was also presented. It was much worse than with the cathode second layer. The cell performance with the cathode second layer became worse at higher current density due to larger cathode polarization. Figure 12 shows the relationship between the cell life t_L and the $\Delta \log a_o$, where t_L was defined as the time until the cell voltage decreased to -0.3 V in Fig. 11. The cell life became shorter with decreasing oxygen activity at the interface. This result indicates that the chromium flux to the interface is influenced by the decrease in oxygen activity at the interface even with the adoption of the cathode second layer.

Figure 13 shows the change in cell voltage with time for the chromium vapor pressures from the tablet. In this figure, cell performances without the cathode second layer were also presented. Cell performance without the cathode second layer was extremely worse than with the cathode second layer. Cell performance with the cathode

Fig. 13 Cell voltage with ((a)) and without ((b)) the cathode second layer for given $P_{CrO_2(OH)_2}$. Each cell was operated at the same $\Delta \log a_o$ condition. $P_{CrO_2(OH)_2}$ was changed by the operating temperature and the chromium oxide tablet. ($\circ$): $T = 1173$ K, Cr_2O_3 tablet; ($\blacksquare$): $T = 1123$ K, Cr_2O_3 tablet; ($\diamond$): $T = 1073$ K, Cr_2O_3 tablet; ($\boxtimes$): $T = 1073$ K, $CoCr_2O_4$ tablet.

Fig. 14 Relationship between the cell life (t_L) and the calculated $P_{(Cr)}$ with ((a)) and without ((b)) the cathode second layer.

second layer became more stable with decreasing chromium vapor pressure.

Figure 14 shows the relationship between the cell life t_L and the $P_{(Cr)}$, where t_L was defined as the time until the cell voltage decreased to 0 V in Fig. 13. Cell life with the cathode second layer appeared to be in inverse proportion to $P_{(Cr)}$. By comparison with the cell life without the cathode second layer, cell life was influenced almost by the effect of the cathode second layer. The cell life can be determined by the time until the effect of the cathode second layer comes to an end. It seems to be equal to the time until the reaction of La_2O_3 with the chromium vapor saturates.

An estimation of the effect of the cathode second layer was made as follows.

Γ ($g \cdot cm^{-2} \cdot h^{-1}$): Chromium flux from the surface of the separator to the cathode/electrolyte interface. The amount of the chromium is evaluated as the weight of Cr_2O_3 .

$C_{La_2O_3}$ ($g \cdot cm^{-2}$): La_2O_3 content in the cathode second layer.

$C_{Cr_2O_3} \equiv C_{La_2O_3} \times 152.0 / 325.8$ ($g \cdot cm^{-2}$): Amount of Cr_2O_3 which can react with La_2O_3 in the cathode second layer by the reaction;

$\Delta T(h)$: Increase in cell life that is extended by the cathode second layer.

On the assumptions that 100 % of La_2O_3 in the cathode second layer can react with the chromium vapor and that cathode degradation starts after the reaction of La_2O_3 and the chromium vapor is saturated,

$$\Delta T = C_{Cr_2O_3} / \Gamma \quad (6)$$

is obtained.

Γ is a function of the total chromium vapor pressure (nearly equals $P_{(Cr)}$) and $\Delta \log a_o$. In ref. 1, SEM and EPMA images at the cathode/electrolyte interface were obtained after a 600 h cell test for the case with untreated inconel 600 at 1273 K, $0.3 A/cm^2$ ($\eta = 0.060$ V). In this case, $P_{(Cr)} = 2.282 \times 10^{-7}$ atm, $\Delta \log a_o = -0.475$ and $\Gamma = 4.1 \times 10^{-6} g \cdot cm^{-2} \cdot h^{-1}$ were obtained. The Γ value was calculated from the thickness of Cr_2O_3 deposition at the interface and the amount of chromium which had reacted with the lanthanum manganite. These data give;

$$\Gamma (P_{(Cr)} = 2.282 \times 10^{-7} \text{ atm}, \Delta \log a_o = -0.475) = 4.1 \times 10^{-6} \quad (7)$$

Γ for various $P_{(Cr)}$ and $\Delta \log a_o$ can be calculated by using the above value on the assumption that it is proportional to $P_{(Cr)}$ and it changes with $\Delta \log a_o$ according to the relationship shown in Fig. 8.

$C_{La_2O_3}$ is calculated from the density of La_2O_3 , $d_{La_2O_3} = 6.5 g \cdot cm^{-3}$ and $La_{0.5}Sr_{0.5}MnO_3$, $d_{LSM} = 5.8 g \cdot cm^{-3}$. In this section, the thickness and the porosity of the cathode second layer are $80 \mu m$ and 60 %. They give $C_{La_2O_3} = 1.88 \times 10^{-3} g \cdot cm^{-2}$ and $C_{Cr_2O_3} = 8.75 \times 10^{-4} g \cdot cm^{-2}$.

Fig. 15 shows the relationship between the experimental ΔT in Fig. 14 and the calculated ΔT from eq. (6). The calculated ΔT agreed well with the experimental data. This result indicates that almost 100 % of La_2O_3 in the cathode second layer reacted with the chromium vapor and had contributed to the increase in cell life.

A rough estimation of cell life adopting the cathode

Fig. 15 Relationship between the experimental increase in cell life (ΔT_{exp}) and the calculated increase in cell life (ΔT_{cal}) that were extended by the cathode second layer.

Fig. 16 Estimated ΔT for $P(\text{Cr})$ and η . In this figure, the thickness of the cathode second layer was assumed to be $800 \mu\text{m}$. (i): Calculated P_{CrO_3} from Cr_2O_3 at 1173 K; (ii): Calculated P_{CrO_3} from CoCr_2O_4 at 1173 K; (iii): Calculated P_{CrO_3} from Cr_2O_3 at 1073 K; (a): $\eta = 0.120 \text{ V}$ (at 1173 K); (b): $\eta = 0.100 \text{ V}$ (at 1173 K); (c): $\eta = 0.080 \text{ V}$ (at 1173 K); (d): $\eta = 0.060 \text{ V}$ (at 1173 K).

second layer was made on the basis of Fig. 12 and 14. The result is shown in Fig. 16. In this figure, the thickness of the cathode second layer was assumed to be $800 \mu\text{m}$. The cell life was estimated with the parameter of the cathode polarization at 1173 K. $P_{\text{H}_2\text{O}}$ in the cathode gas was assumed to be less than 10^{-4} atm , and P_{CrO_3} from Cr_2O_3 at 1073, 1173 K and P_{CrO_3} from CoCr_2O_4 at 1173 K were presented. P_{CrO_3} from Cr_2O_3 means the chromium vapor pressure from the non-treated alloy separator and P_{CrO_3} from CoCr_2O_4 means the level to which chromium vapor pressure can easily be decreased by a simple

surface treatment for the alloy. For example, cell life 40000 h can be achieved by reducing chromium vapor pressure about 1/3 times as low as non-treated alloy at the condition of 1173 K, $\eta = 0.120 \text{ V}$. It can also be achieved by reducing η less than 0.060 V at the condition of 1173 K without reducing chromium vapor pressure from the alloy (without any surface treatment for the alloy).

4 CONCLUSIONS

Chromium oxide vapor specie that influences cathode degradation is mainly $\text{CrO}_2(\text{OH})_2$ at high $P_{\text{H}_2\text{O}}$ condition and CrO_3 at low $P_{\text{H}_2\text{O}}$ ($\sim 10^{-4} \text{ atm}$) condition. Cathode degradation can be reduced by decreasing $P_{\text{H}_2\text{O}}$ so far as that $P_{\text{CrO}_2(\text{OH})_2}$ is equal to P_{CrO_3} .

Chromium diffusion to the cathode was suppressed by adopting a cathode second layer. This effect seemed to be caused by the reaction of La_2O_3 with the chromium vapor to decrease the chromium vapor pressure in the cathode. The cell life with the cathode second layer was extended by decreasing the cathode polarization and the chromium vapor pressure from the alloy. Almost all La_2O_3 in the cathode second layer seemed to react with the chromium vapor and contribute to the increase in cell life.

This work was performed as an R&D program of the New Energy and Industrial Technology Development Organization (NEDO) under the New Sunshine Project of the Agency of Industrial Science and Technology, MITI.

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