



The influence of polymer electrolyte fuel cell cathode degradation on the electrode polarization

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ABSTRACT

The electrode of polymer electrolyte fuel cell (PEFC) consists of the porous catalyst layer and gas diffusion layer (GDL). Quantitative evaluation of the influence of these porous layers' degradation on the cell performance was attempted. The cell was assembled by using the catalyst layer or GDL, which had been corroded *ex situ*, as the cathode and the cell performance was characterized. The oxygen diffusion polarizations of the catalyst layer and that of the GDL were evaluated from the polarization curves. The polarization curves before and after a long-term operation were also analyzed by the same way, and the influences of the degradation of catalyst layer and GDL were evaluated. The increase of the gaseous diffusion loss in the catalyst layer was found to cause the cell performance loss mainly from the analysis of the simulated corrosion test and the long-term operation cell.

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1. Introduction

Fuel cell is a well known as a device which efficiently converts hydrogen into electricity efficiently, and much effort is currently being invested in making such technology practical. So far, many valuable outputs have been obtained for residential fuel cell systems based on polymer electrolyte fuel cell (PEFC) though the large-scale stationary fuel cell demonstration in Japan. The development of PEFC is also being carried out in automobile industry as a potential power supply for vehicles. Despite the growing interest in PEFC, the improvement of cell durability is still necessary before the PEFC can become fully commercialized. Today, the causes which shorten the life of PEFC are assumed to be the chemical degradation of the electrolyte membrane, the dissolution of the platinum catalyst, poisoning by contamination and the corrosion of catalyst supports [1]. The electrodes of a PEFC consist chiefly of carbon which suffers electrochemical oxidation at a high potential. The reverse current mechanism to which the carbon is exposed at a high potential has been reported [2]. Furthermore, the shift

of the electrolyte potential by which the cathode rises to a high potential through the high fuel utilization, low hydrogen concentration and/or high CO concentration, is also reported in the case of phosphoric acid fuel cell [3]. The carbon support for cathode catalyst is also reported to corrode during dynamic potential changes [4,5]. Here, the cathode catalyst support is reported to corrode by the platinum sintering which affects the oxygen diffusion in the electrode. Dynamic potential changes during the start and stop sequences cannot be avoided, suggesting that the possibility for the electrochemical oxidation of the carbon material is very high during the operation of a PEFC.

During the operation of a PEFC, oxygen arrives at catalyst layer by diffusing through the gas diffusion layer (GDL). In minimizing the increase in the oxygen diffusion losses, the decrease in the diffusion rate, which is associated with the degradation of the GDL and catalyst layer, should be considered. There are few reports which examine the occurrence of the diffusion losses and their influence on the cell performance. Hicks co-workers reported the influence of the catalyst support and GDL degradation on the cell performance [6,7]. They assembled a membrane electrode assembly (MEA) using already-corroded component to investigate the influence of such a corroded component on the cell performance. The cell characteristics have shown that the influence of the catalyst support corrosion to be dominant factor on the cell performance decay, and if the graphitized carbon is used for the catalyst support, its physical

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stability increases greatly. This method of assembling MEA from the components, purposely corroded before the assembly, seems as an effective method to examine the influence of the electrode corrosion on the cell performance.

It is preferable to analyze the diffusion losses in the GDL and the catalyst layer separately when their influences on the PEFC performance are evaluated. However, it is difficult to find the reports which mention the methods to separately and quantitatively evaluate the influence of the GDL and catalyst layer diffusion losses on the cell performance using actual cells. In particular, such a separate evaluation of the influence of degraded components after a long-term cell operation cannot be found. In this report, the reactant diffusion losses in the catalyst layer and GDL have been evaluated separately by using the polarization curve. The measurements are taken after the influence of the corroded components on the cell performance has been confirmed to exist. This technique is also applied to analyze the performance losses after a long-term operation of a cell.

2. Experimental

2.1. Simulated electrochemical corrosion

The GDL and catalyst layer were assembled into an MEA, and treated by applying a potential, initiating simulated corrosion. The corrosion treatment was conducted by holding the cathode at 1.5 V relative to the anode, by an external power source (Hokutodenko HZ5000) at 70 °C, while the cathode and anode were exposed to nitrogen and hydrogen flows respectively. The potential was applied for up to 8 cycles with each cycle continuing for 30 s. The potential, therefore, have been applied for 30–240 s in total. This corrosion treatment is believed to simulate the corrosion in actual operation because it is conducted using an actual MEA and under actual operating conditions.

The changes in catalyst surface area, wettability, and pore volume were determined after applying the potential were analyzed. The catalyst surface area is calculated from the amount of hydrogen adsorption by cyclic voltammetry. The voltammetry measurement is performed by introducing hydrogen and nitrogen to the anode and cathode respectively at 30 °C. The potential was swept from 0.08 V to 0.7 V at a rate of 10 mV s⁻¹.

The wettability of catalyst layer was estimated from the ratio of steam and nitrogen adsorption. After degassing for 24 h at 80 °C in a vacuum chamber, the amounts of adsorbed steam and nitrogen were measured by static volumetric method using BELSORP-aqua3 and BELSORP-mini (BEL JAPAN Inc.). The fine pore volume of the catalyst layer was also calculated from amount of nitrogen adsorption.

The wettability of GDL was defined by contact angle. Because the small difference of the contact angle by water is difficult to distinguish, the contact angle response of liquid with smaller surface tension than that of water is measured.

2.2. Simulated GDL degradation by chemical oxidation

During the operation of a PEFC, the catalyst layer, especially on the cathode, is expected to experience electrochemical corrosion because it is exposed to a high potential. On the other hand, as the GDL of a PEFC is in wet environment, where the GDL is in contact with water of extremely high purity and very low electronic conductivity, during the operation, the level of corrosion which the GDL suffers from application of the cathode potential is uncertain.

The decrease in the hydrophobicity of the GDL is thought to be caused by the oxidation of the GDL surface. Here, one of the experiments involved the GDL which was chemically made hydrophilic.

Hicks [6] conducted the chemical oxidation treatment by soaking GDL in hydrogen peroxide. The oxidation treatment of GDL was performed also in this study by the same technique. The GDL was soaked in a 30% hydrogen peroxide solution and placed in an oven for 50 h at 80 °C. After cooling down, the GDL was washed with purified water. The result of the GDL oxidation treatment was also evaluated from the liquid contact angle.

2.3. Cell performance with simulated corroded electrode component

The corroded catalyst layer and the corroded GDL were assembled into MEA with other unused components to investigate the influence of such corroded components on the cell performance. The cell performance test was carried out by measuring the air utilization response and the oxygen gain at 0.2 A cm⁻², and polarization curve. The polarization curve was measured while applying a hydrogen flow with 80% hydrogen utilization to the anode and an air flow with 60% air utilization to the cathode. By using hydrogen on the anode, the anode can be considered as a standard hydrogen electrode.

3. Result

3.1. Influence of carbon support on cell performance

The corrosion of the cathode was confirmed after corrosion treatment. The platinum surface area decreased after applying 1.5 V across the cell. Fig. 1 shows the loss in platinum surface area with respect to the time for which the potential was applied. The platinum surface area has decreased up to around 60% by application of the potential for 240 s.

The corroded cathode catalyst layer was observed to adsorb nitrogen and steam. As shown in Fig. 2, the ratio between the steam adsorption and nitrogen adsorption increased very much by applying the potential, indicating the increase in the hydrophilicity with respect to the time during which the potential was applied. After applying the potential for 240 s, the steam adsorption became larger than nitrogen adsorption. This is expected due to the capillary condensation in catalyst layer which occurs as the finer pores are produced in catalyst layer by the corrosion. The trend in pore volume loss with respect to the duration of the potential application, in Fig. 3, was calculated from the nitrogen adsorptions. The loss in the pore volume loss by the applied potential possibly resulted

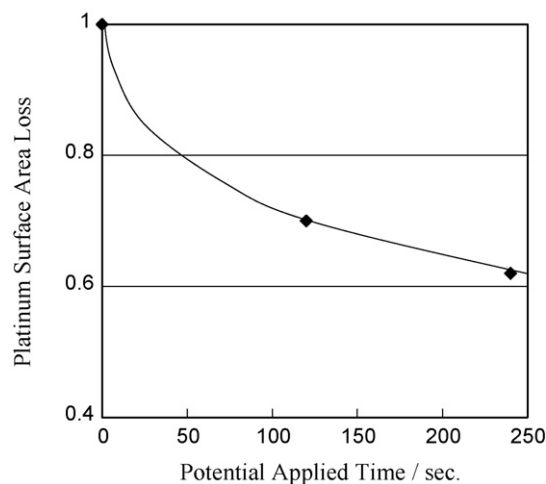


Fig. 1. Loss in a platinum surface area on cathode against duration of potential application. Loss in the surface area is shown as a ratio between the surface area after and before the potential application.

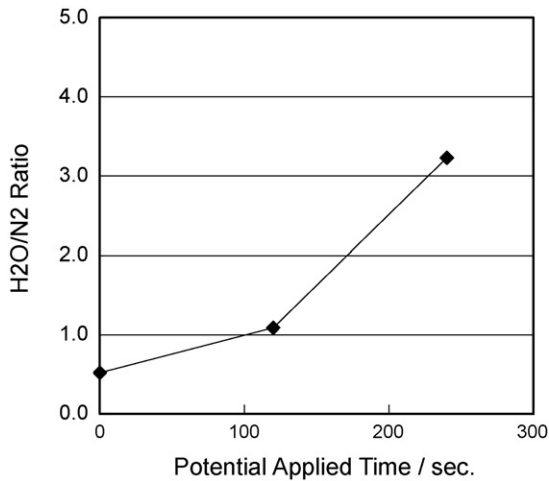


Fig. 2. Ratio of steam adsorption to nitrogen adsorption against duration of potential application.

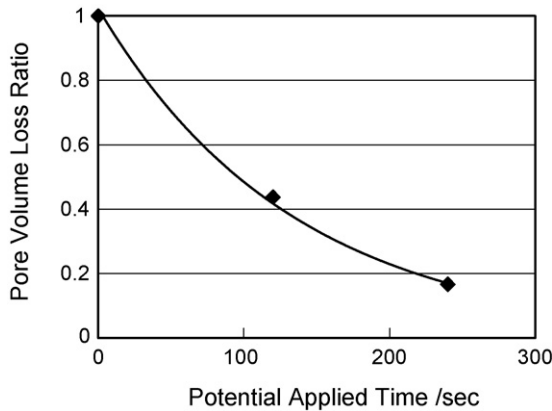


Fig. 3. Ratio of pore volume loss against duration of potential application.

in the form of finer pore than the initial ones, decreasing in the bulk density.

The performance of the MEA, which was rebuilt using the corroded cathode and other unused components, was characterized. The polarization curve is shown in Fig. 4. The cathode, to which the potential was applied for 120 s, has shown a diffusion loss at high electrical loading. But in the case of the cathode, to which the potential was applied for 240 s, not only the diffusion polarization loss but also the active loss were observed from low electrical loading.

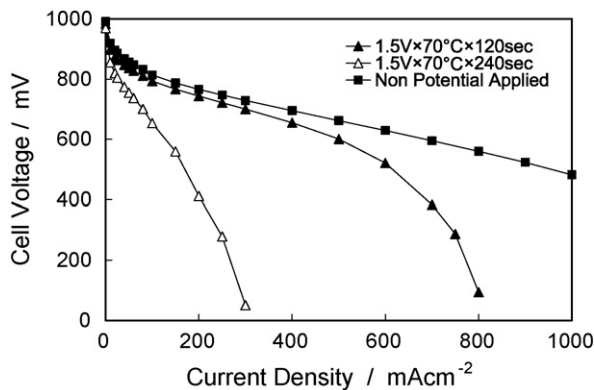


Fig. 4. Polarization curve by MEA with non-potential applied catalyst and corroded cathode catalyst. (Air utilization: 60%, fuel utilization: 80%, full humidified.)

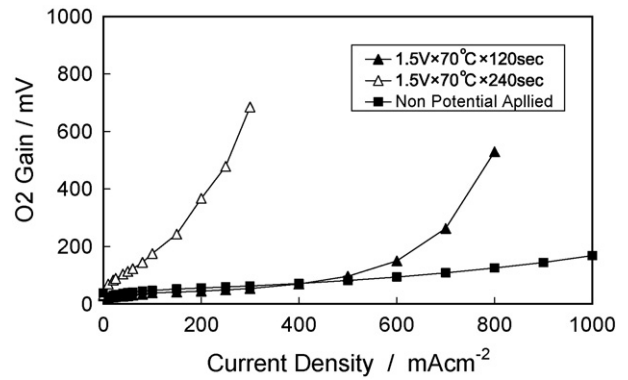


Fig. 5. Oxygen gain at 0.2 A cm⁻² for MEA with no-potential applied cathode and corroded cathode.

To confirm the cause of the performance loss of the MEA, the trend in oxygen gain with respect to the level of the electrical loading is also shown in Fig. 5. The increase in the oxygen gain was synchronized with the voltage loss of the polarization curve in Fig. 4. It was, therefore, possible to consider the main cause of the voltage loss was the diffusion loss.

Fig. 6 indicates the response of the cell voltage to the change in air utilization. The cell voltage of the MEA, to which the potential was applied for 240 s, was much lower than the initial performance. It is impossible to account for the entire voltage loss only by the catalyst activity loss. Here, a very severe diffusion loss was considered to have affected the performance.

3.2. Influence of GDL corrosion on cell performance

The simulated corrosion of catalyst layer was treated electrochemically by using MEA. The corrosion of the GDL which was treated at same time as catalyst layer was examined. The contact angle measurements of the corrosion treated GDL and unused one are shown in Fig. 7. Although the potential was applied to the GDL for 120 s, the contact angle was hardly changed, meaning that it must have been intact. Even if the high potential was applied to the GDL within MEA, the electrochemical oxidation could not be observed. An MEA was rebuilt by using this GDL and its response on the air utilization was characterized. The response on the air utilization at 0.2 A cm⁻² is shown in Fig. 8, together with that of the corroded cathode. The air utilization response of the GDL, to which a high potential is applied, shows the same response as the unused GDL. On the other hand, the air utilization response of the catalyst layer, to which a high potential is applied, shows voltage loss at high

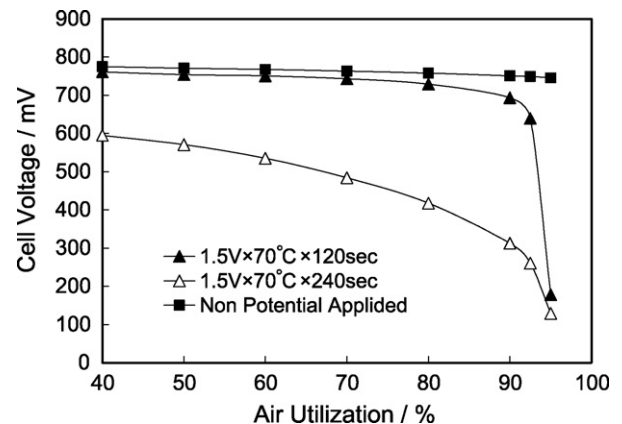


Fig. 6. Cell voltage at 0.2 A cm⁻² against air utilization for MEA with non-potential applied cathode and corroded cathode.

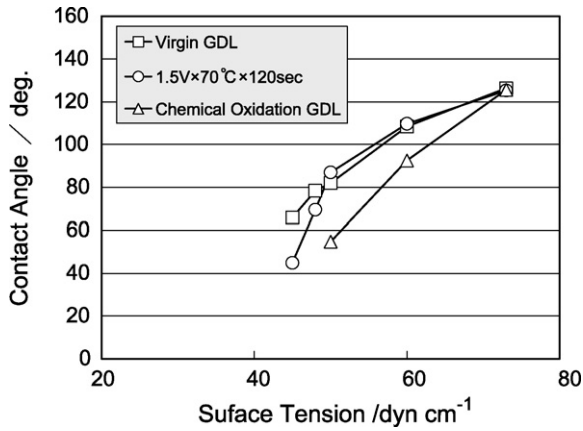


Fig. 7. Contact angle against surface tension of dropped liquid on GDL.

air utilization. The high potential did not affect GDL characteristics in the MEA and the performance loss was hardly observed.

The contact angle of the chemically corroded GDL is also indicated in Fig. 7. Compared to the GDL which has been corroded by the application of the potential for 120 s, the chemically corroded GDL showed lower contact angle. The GDL seems to become hydrophilic by chemical corrosion treatment. An MEA was rebuilt by using chemically oxidized GDL and its response on air utilization was examined. Fig. 9 shows the result. The air utilization response was hardly changed by chemical oxidation. Although hydrophobicity is decreased, the influence on the performance was not observed.

4. Discussion

4.1. Polarization analysis using the polarization curve

A schematic diagram of the cathode cross-section of a PEFC is shown in Fig. 10. In the case of the cathode, the oxygen concentration gradient appears between the gas channel and the membrane when the electrical current is applied across the cell. In particular, the catalyst layer possesses a concentration gradient showing the oxygen consumption towards the membrane. The cell voltage, U , at a current density, i , is defined by subtracting the resistance, diffusion and activation polarization from the theoretical voltage

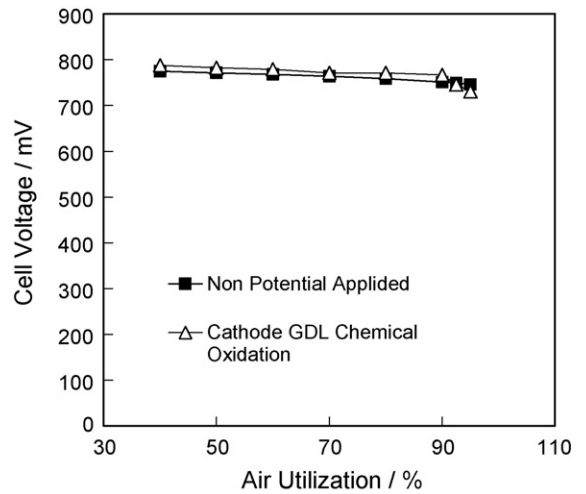


Fig. 9. Cell voltage at 0.2 A cm^{-2} against air utilization for untreated GDL and chemical oxidized GDL.

U_0 .

$$U = U_0 - ir - b \ln \left(\frac{i}{i_0} \right) + b \ln \left\{ \frac{i_L - i}{i_L} \right\} \quad (1)$$

In Eq. (1), i_0 is exchange current density, i_L is limiting current density, b is Tafel slope and r is resistance. When the electrode reaction is analyzed, $b \ln(i/i_0)$ and $b \ln\{(i_L - i)/i_L\}$ represent activation and diffusion polarizations respectively. However, the activation polarization has the exchange current density, i_0 , which is shown by Eq. (2). In the case of the fuel cell cathode, the influence of the oxygen concentration on the activation polarization needs to be considered because there is an oxygen concentration distribution in catalyst layer as shown in Fig. 10.

$$i_0 = nFkC \quad (2)$$

i_0 is determined by reaction rate constant, k , and oxygen concentration, C . The reaction rate constant is dependent on the catalyst activity, and the catalyst activity is determined by multiplication catalyst specific activity and specific surface area. For an alloy catalyst, there is a possibility of the change in its specific activity by the change in its formation, however in the case of fuel cell electrodes, major change is due to the loss in the specific surface area by sintering. The activation loss of catalyst is one of the important

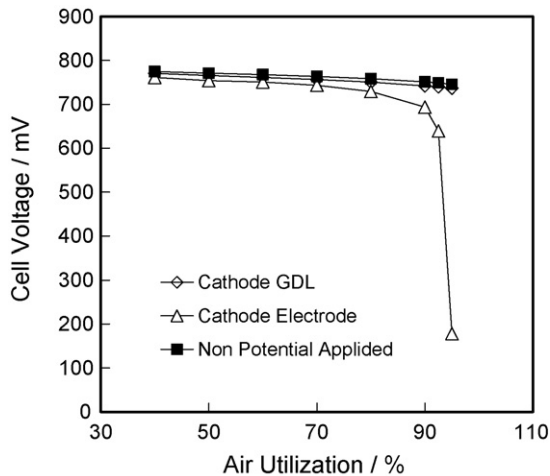


Fig. 8. Cell voltage against air utilization at 0.2 A cm^{-2} for cathode catalyst layer and GDL, to which 1.5 V was applied for 120 s.

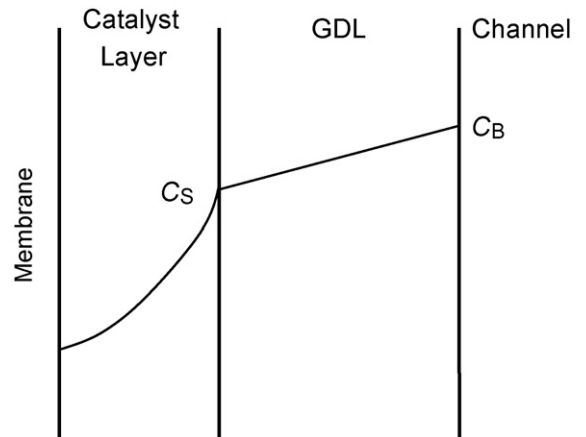


Fig. 10. Oxygen concentration distribution of PEFC cathode cross-section. C_B is oxygen concentration in gas flow channel, and C_S is oxygen concentration in boundary area between GDL and catalyst layer.

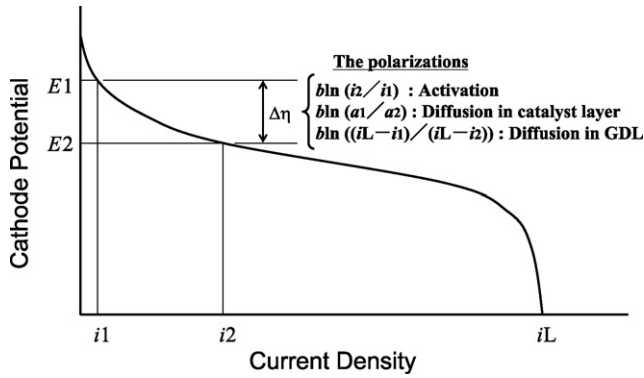


Fig. 11. Polarizations in cathode potential difference between E_1 and E_2 .

evaluation items and the analysis of such a catalyst activation loss should be separated from other items.

The oxygen concentration changes with electrical loading because porous electrodes are used in a fuel cell. The polarization difference $\Delta\eta$ between current density i_1 and i_2 can be described as in Eq. (3) by using exchange current density $i_{0,1}$ and $i_{0,2}$.

$$\begin{aligned} \Delta\eta &= E_1 - E_2 \\ &= b \ln \left\{ \frac{i_{0,1} i_2}{i_{0,2} i_1} \right\} + b \ln \left\{ \frac{i_L - i_1}{i_L - i_2} \right\} \end{aligned} \quad (3)$$

Here, E_1 and E_2 are the cathode potentials that are defined by $E_1 = U_1 + i_1 r$ and $E_2 = U_2 + i_2 r$. And $i_{0,1}$ and $i_{0,2}$ are considered to take different values from the actual exchange current density i_0 because of the oxygen concentrations difference. Eq. (3) becomes Eq. (4) by substituting $i_{0,1} = a_1 i_0$, $i_{0,2} = a_2 i_0$. Here, a_1 represents the ratio between the exchange current density, $i_{0,1}$, and the current density, i_0 , when the load, i_1 , is applied to the MEA. Similarly, a_2 represents the ratio between $i_{0,2}$ and i_0 when the load, i_2 , is applied to the MEA.

$$\begin{aligned} \Delta\eta &= E_1 - E_2 \\ &= b \ln \left(\frac{i_2}{i_1} \right) + b \ln \left(\frac{a_1}{a_2} \right) + b \ln \left\{ \frac{i_L - i_1}{i_L - i_2} \right\} \end{aligned} \quad (4)$$

When oxygen diffusion loss is evaluated from the polarization curve, it is preferable to separate the diffusion losses of GDL, which is considered to be caused by the simple pass of oxygen through the layer, and that of the catalyst layer, which includes consumption of oxygen. The term, $b \ln\{(i_L - i_1)/(i_L - i_2)\}$, represents the diffusion loss caused by oxygen transfer loss from gas channel to the reaction point. Therefore, this term is insufficient as the description of the diffusion loss which also takes the consumption of the reactant into account, and it should only be used to describe the diffusion loss such as that in GDL, which does not include reactant consumption. On the other hand, the diffusion loss which takes the consumption

Table 1

Measured key values for a polarization analysis from polarization curves of MEA with a simulated corroded component.

Cathode catalyst layer	Cathode GDL	$i_{0,9}$ (mA cm ⁻²)	i_L (mA cm ⁻²)	r (Ω cm ²)
1.5 V, 120 s applied	Unused	9.0	8.0×10^2	1.0×10^{-1}
1.5 V, 240 s applied	Unused	5.9	3.0×10^2	1.2×10^{-1}
Unused	1.5 V, 120 s applied	1.9×10	1.0×10^3	1.1×10^{-1}

of the reactant into account can be evaluated by the ratio of a_1 to a_2 , because the exchange current density is related to oxygen concentration. The diffusion loss in the catalyst layer is assumed to be described by $b \ln(a_1/a_2)$. The polarizations in the potential loss $\Delta\eta$ from E_1 to E_2 are summarized in Fig. 11.

When the polarization losses at every electrical loading are evaluated from the polarization curve, it is convenient to provide the base potential that corresponds to E_1 . The base potential should be decided in the current region which was not influenced by reactant diffusion loss, where the current density can suitably be described by $i_1 \ll i_L$. In this report, 0.9 V (i_r free) was selected as the base potential. The potential difference between 0.9 V and E_n at some current density i_n was separated to $b \ln(i_n/i_{0,9})$, $b \ln(a_{0,9}/a_n)$ and $b \ln\{(i_L - i_{0,9})/(i_L - i_n)\}$. Here, $i_{0,9}$ is the current density at 0.9 V.

4.2. Influence of diffusion loss in the cathode catalyst layer

If 3 nm platinum particles on carbon support are being used as the cathode catalyst, several tens of percent of the platinum surface area is generally lost during a long-term operation. The diffusion loss in simulated corroded cathode was analyzed. As shown in Fig. 4, the voltage of MEA which is rebuilt with the simulated corroded cathode, to which a high potential was applied for 120 s, starts to drop from 400 mA cm⁻². The reason for the voltage drop is thought to be the increase in the diffusion loss. But the platinum surface loss is not so large at around 15%. Moreover, in the case of MEA, to which the potential was applied for 240 s, in spite of the loss in the surface area of less than 40%, the voltage loss which is considered to be the diffusion loss is observed from a low current density.

It is clear that the performance difference can hardly be explained by the catalyst surface area loss. The polarization analysis results of these MEAs are shown in Fig. 12. In order to analyze the polarizations, $i_{0,9}$, i_0 and resistance from the polarization curves in Fig. 4. The derived key values are in Table 1. In this report, the polarizations are calculated by using $b = 40$ mV decade⁻¹.

The diffusion loss in catalyst layer was clearly larger than that of GDL. Fig. 2 shows an increase in the wettability which is caused by the corrosion of catalyst support by applying a high potential. It is expected that the increase in the wettability has caused the flooding in the catalyst layer. The diffusion loss in GDL is also indicated in Fig. 12 in spite of using unused GDL. It seems that the diffusion

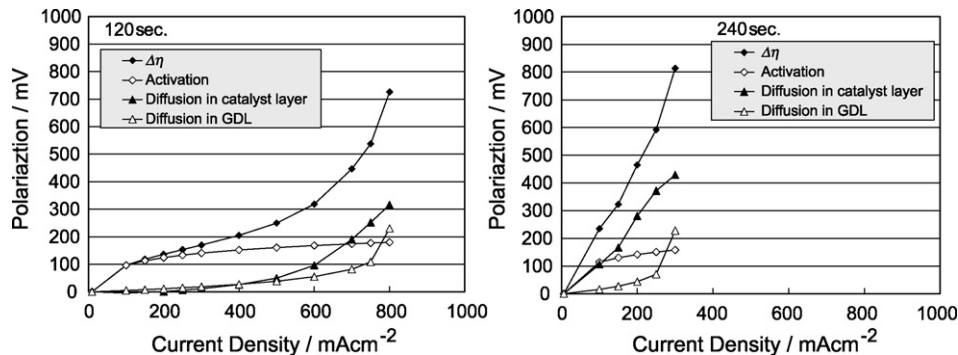


Fig. 12. Polarization analysis on simulated corroded cathode.

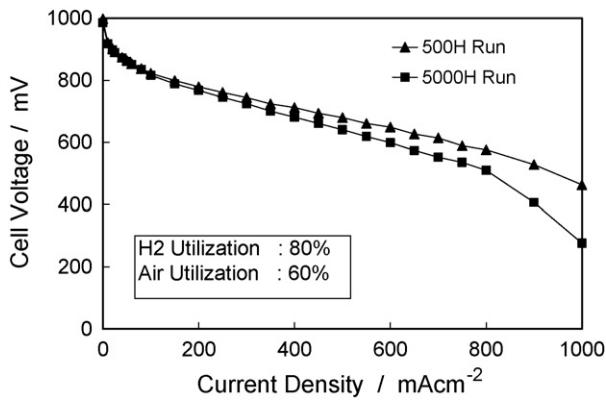


Fig. 13. Polarization curve for a continuously operated cell after 500 h and 5000 h.

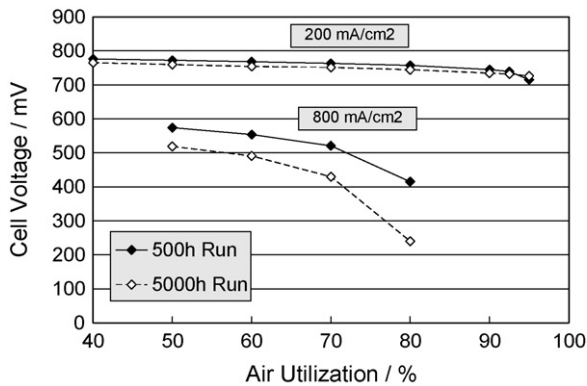


Fig. 14. Cell voltage against air utilization after 500 h and 5000 h.

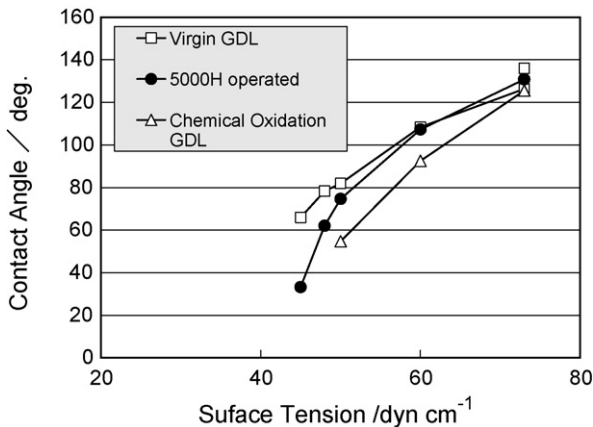


Fig. 15. Contact angle against surface tension of dropped liquid on GDL.

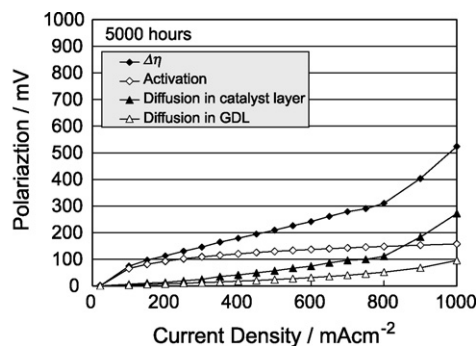
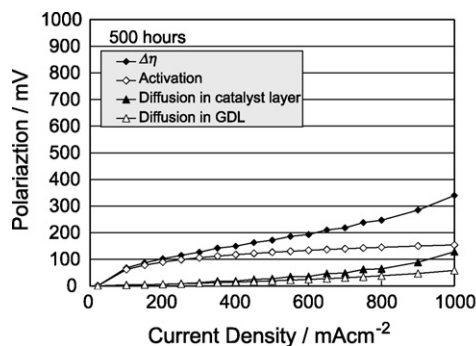


Fig. 16. Polarization analysis at 500 h and 5000 h for a long-term operation cell.

Table 2

Measured key values for polarization analysis from polarization curves of 500 h and 5000 h operated cell.

Operation time (h)	$i_{0.9}$ (mA cm^{-2})	i_L (mA cm^{-2})	r ($\Omega \text{ cm}^2$)
500	2.1×10	1.3×10^3	9.7×10^{-2}
5000	2.0×10	1.1×10^3	1.0×10^{-1}

loss is not associated with GDL, but it should be caused by the low catalyst utilization as the result of flooding in catalyst layer.

4.3. Diffusion loss change in the cathode by a long-term operation

Same analysis was conducted for a long-term operation of the cell. Fig. 13 shows the comparison of polarization curve of the cell after 500 h and 5000 h of operation. After 5000 h of operation, the voltage loss around high current density becomes larger than that after 500 h of operation. The change in air utilization response from 500 h to 5000 h, in Fig. 14, is small although the voltage loss is observed at 800 mA cm^{-2} .

Moreover, the contact angle response on dropped liquid surface tension of GDL was also confirmed. After 5000 h of operation, as shown in Fig. 15, the contact angle response of the GDL showed a mid-value between unused and the chemical oxidation. The change in hydrophobicity of the GDL after 5000 h of operation is expected not to influence the air utilization response. This is because the chemically oxidized GDL has not affected the air utilization response.

In order to examine the performance loss after 5000 h operation, the polarization losses are analyzed from the polarization curve. Fig. 16 shows the comparison of polarization analysis after 500 h and 5000 h in Fig. 13 are presented in Table 2. The activation polarization losses were almost unchanged but the diffusion losses in catalyst layer clearly increased after 5000 h of operation. The diffusion loss in catalyst layer is caused by the flooding and/or poor catalyst utilization. Main cause of the performance loss after 5000 h could be the flooding in catalyst layer because of the small change in activation polarization.

4.4. Influence of GDL oxidation

It seemed that the GDL was hardly corroded during operation and the loss of hydrophobicity did not affect the performance loss. In order to assess the influence of GDL hydrophobicity change on the performance, the polarization analysis was conducted for the MEA with chemically oxidized GDL on the cathode. Fig. 17 shows the result of polarization analysis. As shown in Fig. 15, the contact angle response was shown to be smaller than that after 5000 h of operation, indicating that chemically oxidized GDL was more hydrophilic. However, as shown in Fig. 9, this chemical oxidation

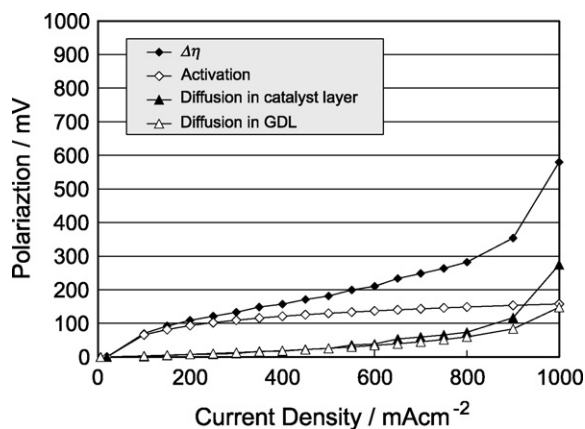


Fig. 17. Polarization analysis from a polarization curve of MEA by using a chemical oxidized GDL on cathode.

did not affect the air utilization response. It would be clear that the oxidized GDL did not affect the performance. By the polarization analysis, the diffusion loss of GDL was almost same as the unused one. It could be said that the degradation of GDL hardly affect the performance loss.

5. Conclusion

The influence of catalyst layer and GDL degradation on cell performance has been examined. In case of PEFC, GDL is a component that is hardly corroded by electrochemical oxidation in MEA. The

component which was corroded by applying high potential was only the catalyst layer and such corrosion affected the diffusion loss of not only catalyst layer but also of the GDL.

The influence of the degradation of the catalyst layer on the cell performance was investigated quantitatively. The performance loss was caused by lower catalyst utilization because of flooding. On the other hand, it was confirmed that there was little influence on the performance though GDL was becoming hydrophilic. It was found that the increase in the GDL diffusion loss which was observed in aging cell was caused by the diffusion loss in catalyst layer.

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