

Life Prediction Technology of Fuel Cell Polymer Electrolyte Membrane under Mechanical Degradation

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ABSTRACT

A method was developed for predicting electrolyte membrane life under mechanical degradation, by quantifying the mechanical stress generated on the fuel cell polymer electrolyte membrane and quantitatively understanding the impact on mechanical degradation. In an environment with repeated drying and wetting, mechanical degradation of the electrolyte membrane progresses at the cracks inside the catalyst layers that are bonded to the electrolyte membrane. This study focused on changes in the electrolyte membrane and catalyst layer properties in the temperature and humidity environment in which fuel cells operate as a factor that increases stress. The electrolyte membrane and catalyst layer properties were measured at the temperatures and humidity under which fuel cells operate, and the measured property values were reflected to a stress analysis model that uses the finite element method in an effort to increase the stress prediction accuracy. The cumulative fatigue damage law was applied to the relationship between the generated stress and stress frequency and the material limit line to quantify the impact of mechanical degradation on life as the life consumption rate, and it was confirmed that the life consumption rate is correlated with the thickness of the electrolyte membrane after durability tests. This enabled prediction of electrolyte membrane life according to mechanical stress. Durability tests that accelerate mechanical degradation were performed using a full-scale-size membrane electrode assembly, and the accuracy of the developed life prediction technology was verified. A high correlation was obtained, which confirmed that the developed life prediction technology can be applied to an on-board-size membrane electrode assembly.

1. Introduction

Fuel cells are power-generating devices that produce electricity by the electrochemical reaction between hydrogen and oxygen. The actual reactions are the oxidation reaction of hydrogen at the anode shown in reaction equation (1), and the oxygen reduction reaction at the cathode shown in reaction equation (2).



The electrochemical reactions in a fuel cell are as follows. First, hydrogen supplied to the anode is split into protons (H^+) and electrons on the catalyst, and the H^+ pass through the polymer electrolyte membrane (PEM) and move toward the cathode. The oxygen supplied to the cathode bonds with the electrons that pass around the outside from the anode and the H^+ that pass through the PEM to produce water. At this time the oxygen reduction reaction occurs on the cathode catalyst and produces electromotive force.

Figure 1 shows a schematic image of a fuel cell. The fuel cell structure consists of a membrane electrode assembly (MEA) that has a PEM sandwiched between catalyst layers (CL) and gas diffusion layers (GDL), and separators that form gas and coolant channels.

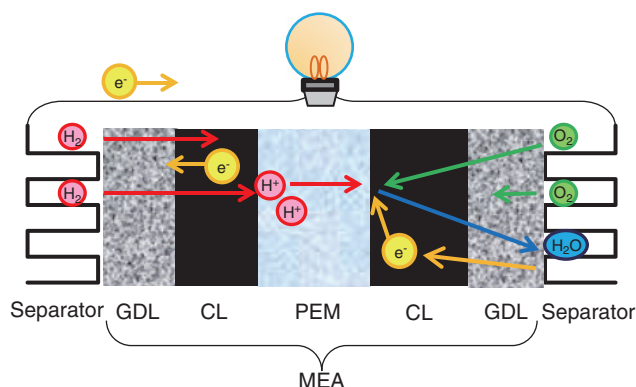


Fig. 1 Schematic image of fuel cell

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Fuel cell stacks for automotive applications call for a smaller size and higher power than those for home applications. In particular, reducing the PEM thickness enhances H^+ conductivity that increases the power, so a thin PEM with a thickness on the order of several tens of μm is generally used. In addition, development is under way to achieve even thinner membranes. However, securing durable reliability for PEM with thinner membranes is an issue. The fuel cell generating environment includes chemical degradation factors specific to polymers such as heat, generated water, and impurity ions^{(1), (2)}, and there are also various mechanical fatigue degradation factors as a result of repeated stress due to pressure differences, heating-cooling cycles, and dry-wet cycles. Among these factors, mechanical degradation of PEM is known to progress in environments where PEM experience excessive and repeated shrinking due to drying and swelling from wetting (dry-wet environment)⁽²⁾⁻⁽⁶⁾. This paper focused on mechanical degradation of PEM. The amount of water produced during fuel cell generation varies according to the power generation load, which means that the PEM water content also varies according to the power generation load. The PEM thickness and in-plane dimensions vary according to the water content, and much water is produced at a high power generation load, so in this condition the PEM water content increases and the PEM swells. In contrast, the PEM water content decreases and the PEM shrinks in the low power generation load condition or when power generation is stopped. Figure 2 shows a slice image of the electrode surface imaged using X-ray computed tomography. Figure 3 shows a cross-sectional image of electrode cracks. Minute electrode cracks on the order of several μm to several tens of μm in size that extend to the PEM surface form in the CL during fabrication. Figure 4 shows a cross-

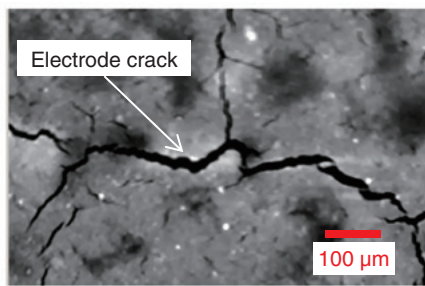


Fig. 2 Image of electrode surface using X-ray computed tomography

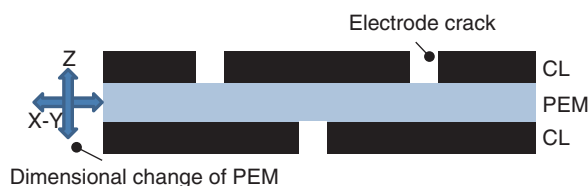


Fig. 3 Cross-sectional image of electrode crack

sectional image of PEM deformation at an electrode crack in a wet environment, and Fig. 5 shows a cross-sectional image of PEM deformation at an electrode crack in a dry environment. Mechanical degradation of PEM at electrode cracks has been reported to progress at a faster rate under this type of dry-wet environment^{(2), (3), (5)}.

As part of continuing efforts to reduce degradation due to these phenomena, various methods have been reported for addressing the issue, such as methods of controlling PEM water content fluctuation, catalyst layer fabrication technology that reduces electrode cracks during fabrication, and enhancement of PEM fatigue durability⁽³⁾. However, it is unlikely that sufficient power generation performance and durable reliability can be achieved by using only a single method. Optimization of each aspect of technology to control PEM water content, catalyst layer fabrication technology, and material technology is called for. For this reason, it is important to understand PEM deformation behavior in a dry-wet environment, clarify the resulting stress applied to the PEM, and understand the impact of this stress on PEM life. However, no known life prediction technology has been established for understanding the impact of mechanical degradation on PEM life.

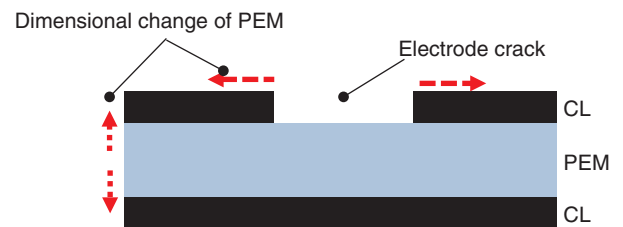


Fig. 4 Cross-sectional image of electrode crack at wet condition

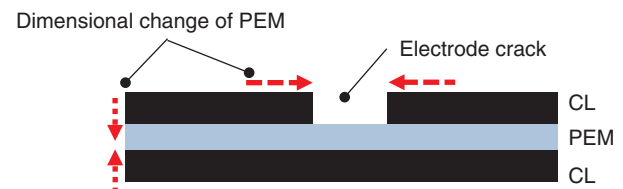


Fig. 5 Cross-sectional image of electrode crack at dry condition

2. Goal

PEM degradation at electrode cracks progresses in a dry-wet environment. Therefore, mechanical degradation of PEM is thought to progress as a result of stress concentration due to the influence of the change in the PEM water content during power generation, the resulting change in PEM dimensions, and the difference in the Young's modulus of the CL and PEM. A technique that uses the impedance

method to understand the change in the PEM water content during power generation has been proposed^{(7), (8)}. However, the PEM is sandwiched by both electrodes as shown in Fig. 1, and it is challenging to directly understand the amount of PEM dimensional change when the water content fluctuates. In addition, the dimensional change of this part is on the order of several percent, so there are issues with using a strain gauge or other means to directly measure the stress generated on the PEM in an environment with water present during power generation. Therefore, the stress generated on the PEM is estimated from a stress analysis model using the finite element method (FEM)⁽⁴⁾.

When constructing life prediction technology for PEM under mechanical degradation, the key factors are to clarify the generated stress and the impact of that stress on PEM life. Therefore, the goal of this study was to clarify these factors and to establish life prediction technology for PEM under mechanical degradation.

3. Establishment of Life Prediction Technology

It was thought that mechanical degradation of PEM at electrode cracks could be predicted from the generated stress and the impact of that stress on PEM life in the same manner as for typical resin materials. It was also considered that the stress generated in each water content environment could be derived using FEM from the difference in the Young's modulus of the CL and PEM and the PEM dimensional change rate in the environment in question, and that the impact on fatigue life could be estimated from the relationship between the material limit line and the generated stress and frequency in that environment. In addition, when stress is repeatedly applied to PEM, plastic deformation and cracking occur and the PEM thickness decreases⁽⁹⁾. Therefore, an attempt was made to predict PEM life under mechanical degradation by determining the relationship between the life and the membrane thickness.

3.1. Determination of PEM and CL Properties for Reflection to Stress Analysis

3.1.1. Determination of PEM and CL Young's modulus

The Young's modulus of the PEM and CL depend on the temperature and humidity, so the Stress-Strain (SS) curve under each environment was measured in a constant temperature and humidity chamber, and the Young's modulus was calculated from the SS curve. Table 1 shows the environmental conditions used for measurement. The shape of the sample used for measurement was a 10 mm × 50 mm rectangle, the distance between chucks was set to 20 mm, and the tensile speed was set to 50 mm/min. As it is not easy to measure the CL Young's modulus E_{CL} alone, this was calculated from the PEM Young's modulus E_{PEM} , the Young's modulus E_{CCM} of the catalyst-coated membrane (CCM) consisting of the CL coated on the PEM, and the CL volume fraction V_{CL} as shown in Eq. (1).

Table 1 Measurement conditions of Young's modulus

	PEM and CL (CCM)	
	Temperature (°C)	RH (%)
SS curve (Young's modulus)	-35	Dry
	23	50
	70	Dry
		95
	90	Dry
		95

Relative humidity: RH

Table 2 Measurement conditions of dimensional change rate

	Direction	PEM		CCM
Dimensional change rate	X-Y (in-plane)	23°C RH50%, 70°C RH95%, 70°C dry, -35°C		
	Z (thickness)	23°C	Wet to dry	
		70°C	Wet to dry	

$$E_{CL} = \frac{[E_{CCM} - (1 - V_{CL})E_{PEM}]}{V_{CL}} \quad (1)$$

3.1.2. Measurement of PEM dimensional change rate

The PEM water content fluctuates and the dimensions change according to the humidity, so the dimensional change rate was measured in a constant temperature and humidity chamber at each temperature and humidity condition shown in Table 2. In addition, the shape of the sample used to measure the dimensional change rate in the in-plane (X-Y) direction was a 10 mm × 50 mm rectangle. The distance between chucks was set to 20 mm, a displacement sensor was attached to the lower part of the measurement sample, and the dimensional change rate in the in-plane direction was measured in each environment. The shape of the sample used to measure the dimensional change rate in the thickness (Z) direction was a 3 mm × 3 mm square, and the dimensional change rate in the thickness direction was also measured in each environment.

3.2. Stress Analysis

The fuel cell internal environment, shape and other factors have various conditions, and direct visualization of the PEM deformation behavior under these conditions takes time. Accurate evaluation of life calls for quantification of stress, and stress analysis using FEM is an effective means of its achievement.

The cross-sectional structure of a fuel cell consisting of the separators, GDL, CL including crack structures, and the PEM was modeled, and stress analysis was performed in the condition with in-plane strain applied. NASTRAN was used as the FEM software, and the material property definitions were treated as material non-linear and anisotropic materials

in order to more accurately reproduce the PEM material properties. In addition, heating and pressurization processes are used when fabricating the MEA, so the interfaces between the GDL and CL and between the CL and PEM are defined as bonded interfaces. Figure 6 shows the model used to perform FEM analysis in this study. FEM analysis was performed in two steps, with input of a clamping load to the MEA via the separators as Step 1, and input of PEM and CL swelling and shrinking as Step 2. The results were used to calculate the stress produced on the PEM at electrode cracks when the PEM swells and shrinks.

3.3. Determination of Material Limit Line

The durability to fatigue resulting from PEM swelling and shrinking drops under high-temperature and high-humidity environments in the same manner as for typical resin materials. In addition, typical PEM have a softening point temperature around 80°C, and the dependence on temperature is assumed to be higher than the dependence on humidity, so the material limit line was determined at each temperature with the humidity fixed to RH 95% in a constant temperature and humidity chamber. Table 3 shows the environmental conditions used for measurement.

The shape of the sample used to determine the Stress-Number (S-N) diagram was a 10 mm × 50 mm rectangle, the distance between chucks was set to 20 mm, and the repetition rate was set to 20 mm/min.

3.4. Life Prediction of PEM under Mechanical Degradation

3.4.1. Calculation method of life consumption rate using cumulative fatigue damage law

The cumulative fatigue damage law is an empirical law related to material fatigue that predicts the life until fatigue breakdown when subjected to fluctuating stress. Figure 7 shows the S-N diagram and the stress frequency. The number of cycles to breakdown of repeated stress P_i on the material limit line is defined as N_i , and the repeated stress P_i frequency is defined as n_i . When the degree of fatigue damage is defined as ΔD_i , this can be expressed as shown in Eq. (2).

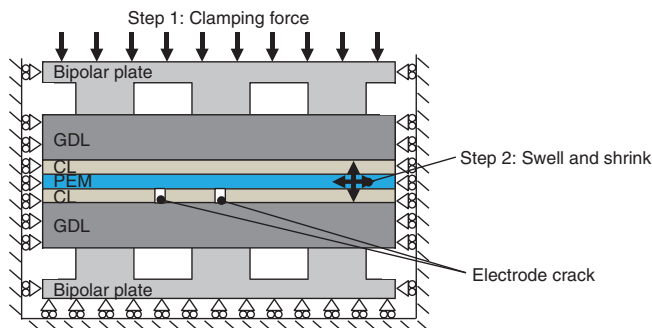


Fig. 6 FEM analysis model for dry-wet cycle

Table 3 Measurement conditions of S-N diagram

	Temperature (°C)	RH (%)
S-N diagram	70	95
	80	
	90	

$$\Delta D_i = n_i / N_i \quad (2)$$

In addition, when the cumulative degree of fatigue stress D is expressed as the cumulative value of each fatigue damage value in accordance with the frequency of various stress amplitudes, this can be arranged as shown in Eq. (3).

$$D = \sum \Delta D_i = \sum (n_i / N_i) \quad (3)$$

The value obtained by expressing the cumulative degree of fatigue stress as a percentage is defined as the life consumption rate (Lcr), and is shown in Eq. (4). The impact on life can be understood by expressing the relationship between the material limit line and each stress frequency using Lcr .

$$Lcr [\%] = \sum (n_i / N_i) \times 100 \quad (4)$$

The PEM properties and material limit line change according to the temperature conditions, so it is necessary to calculate Lcr from the material limit line and the generated stress value in accordance with the environment. Lcr was calculated using the cumulative fatigue damage law from the relationship between the generated stress value obtained by stress analysis using FEM as described in section 3.2. and the PEM material limit line described in section 3.3.

3.4.2. Creation of Lcr - membrane thickness conversion formula

The generated stress depends on the difference in the water content of the electrolyte membrane under a dry-wet environment, so a dry-wet cycle durability test was

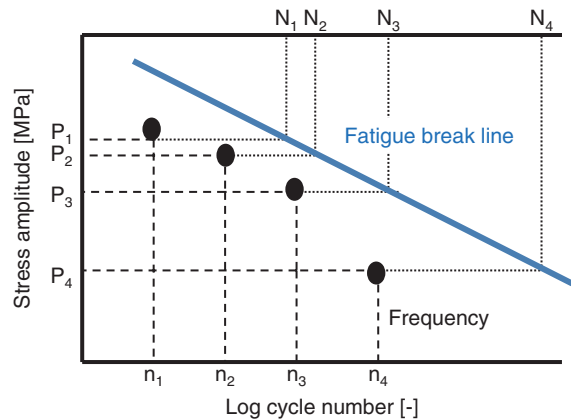


Fig. 7 Cumulative fatigue damage law

Table 4 Conditions of RH cycle test

	Test condition
Temperature (°C)	90
RH (%)	30, 70, 120
Gas supply	Gas: N ₂ (Both anode and cathode) Pressure supply: Normal pressure Flow rate: 2 L/min (Both anode and cathode)

performed. The dry-wet cycle durability conditions set the low-humidity side as the dry condition, and the test was performed with three high-humidity-side conditions as shown in Table 4. An on-board-size MEA has a power-generating area on the order of several hundred cm², and an in-plane humidity distribution occurs during the dry-wet cycle. Therefore, the test was performed using a 40 mm × 60 mm-size MEA to reduce the effect on the in-plane humidity distribution.

The PEM thickness at electrode cracks after the dry-wet cycle durability test was analyzed to determine the correlation between the L_{cr} described in term 3.4.1. and the membrane thickness, and a correlation line was created for deriving the membrane thickness from the L_{cr} .

3.5. Verification Using On-board-size MEA

3.5.1. Verification of prediction accuracy using on-board-size MEA

The prediction accuracy was verified using an on-board-size MEA with a power-generating area on the order of several hundred cm². Figure 8 shows the durability test conditions used for verification. The durability conditions were set to accelerate mechanical degradation of the PEM by applying dry and wet conditions and pressure differences

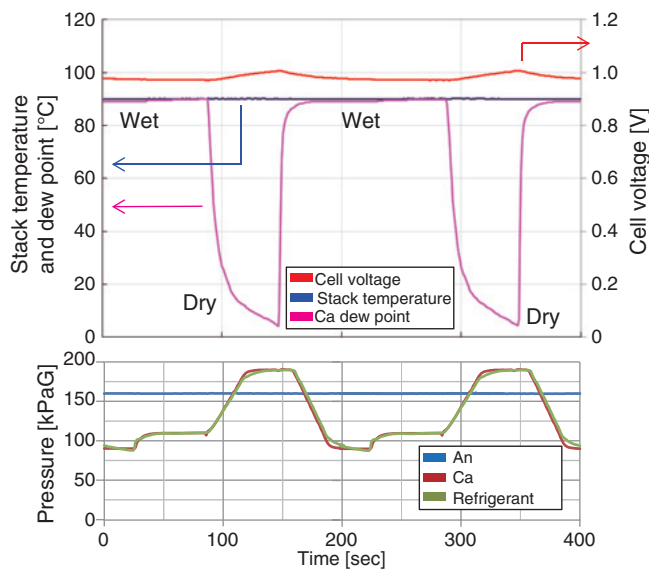


Fig. 8 Profile of accelerated stress test for mechanical degradation of PEM

under a high-temperature environment. The temperature was set to 90°C, which is a condition that reduces PEM fatigue resistance under mechanical degradation. In addition, the dry-wet conditions set the wet side to RH 100% and the dry side to RH 1% to produce a large water content difference. Regarding the pressure difference conditions, the anode pressure was kept constant, and a pressure difference was applied by varying the cathode pressure. An in-plane humidity distribution occurs as the MEA area increases, but the in-plane condition was understood by measuring the in-plane humidity distribution and by using technology to predict the in-plane water content distribution^{(7), (8), (10)}. Mechanical degradation of the PEM progresses in locations where an in-plane water content difference occurs in the power-generating area, so the PEM life prediction accuracy was verified at electrode cracks in the location where the greatest water content difference occurred.

4. Verification Results

4.1. PEM and CL Property Results for Reflection to Stress Analysis

4.1.1. PEM and CL Young's modulus results

Figure 9 shows the temperature dependence of the Young's modulus of the CL and PEM calculated using Eq. (1). These results show that each Young's modulus decreases as the temperature rises. In addition, the Young's modulus decreases additionally under high RH conditions. The CL was confirmed to have a higher Young's modulus than the PEM in each temperature and humidity region.

4.1.2. PEM dimensional change rate results

Figure 10 shows the measurement results of the PEM and CCM dimensional change rates in the in-plane direction. Compared to the dimensions at room temperature, the CCM swells at the high-humidity side and shrinks at the low-humidity side. In addition, these results also show that coating with a CCM reduced the dimensional change in the in-plane direction compared to that of only the PEM. This indicates that the CL has a higher Young's modulus than

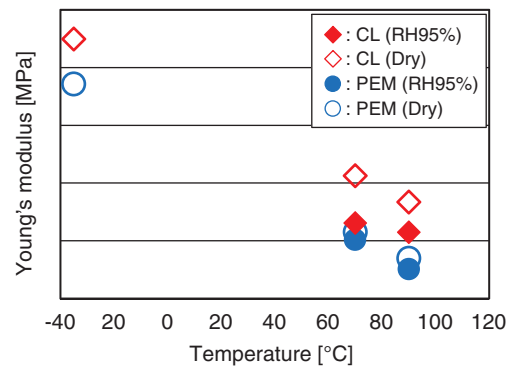


Fig. 9 Results of Young's modulus at various conditions

the PEM, which effectively reduces the PEM dimensional change in the in-plane direction.

Figure 11 shows the measurement results of the PEM dimensional change rate in the thickness direction. This shows that the PEM dimensional change amount in the thickness direction increases together with the temperature and RH.

4.2. Results of Stress Analysis Using FEM

The PEM and CCM properties in each temperature and humidity environment described in terms 4.1.1. and 4.1.2. were reflected to the FEM model shown in Fig. 6, and the PEM deformation behavior in each environment was analyzed. Figure 12 shows the results of using FEM to analyze the deformation behavior when the PEM swells at an electrode crack. The tendency of the PEM to swell as the water content increases was described in term 4.1.2., and it was found that when an electrode crack exists, the PEM deforms in a manner so as to enter the crack. Figure

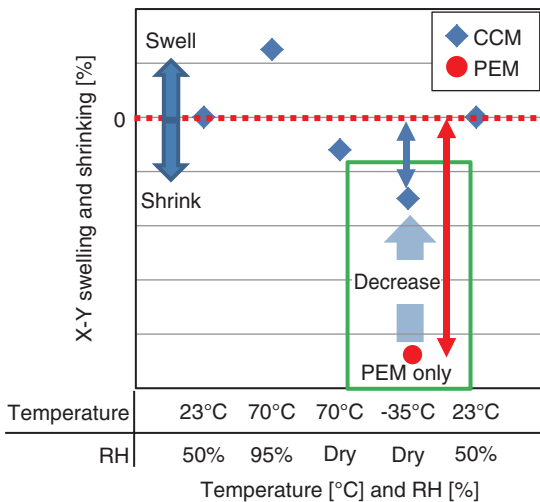


Fig. 10 Results of X-Y dimensional change rate of CCM and PEM

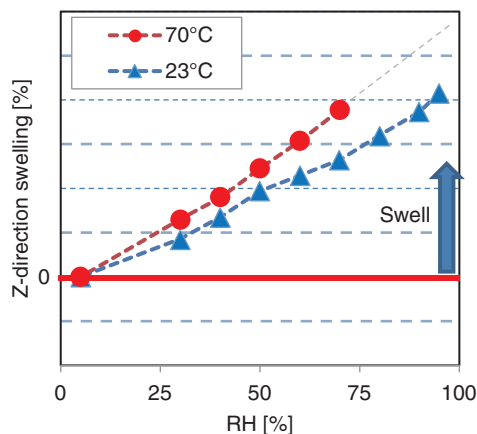


Fig. 11 Results of Z dimensional change rate of PEM

13 shows an enlarged view of the red frame in Fig. 12, indicating that tensile stress concentrates at the edges of the electrode. This can be explained as a result of the CL having a higher Young's modulus than the PEM, as described in term 4.1.1. These results show that stress concentration at the edges of the electrode in a dry-wet environment results in high stress and is a factor promoting mechanical degradation of PEM at electrode cracks. Water is produced in accordance with the load during fuel cell power generation, and the PEM repeatedly swells and shrinks according to the amount of water produced. Therefore, it is thought that high stress is repeatedly input to the PEM around the edges of the electrode at electrode cracks, which promotes mechanical fatigue degradation.

4.3. Material Limit Line Results

Figure 14 shows the PEM S-N diagram results. These results confirm that the stress fatigue durability of the PEM drops as the temperature rises. It was also confirmed that breakdown due to cumulative fatigue occurred even at high fatigue cycle counts in excess of

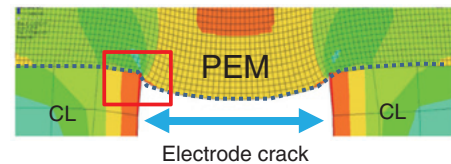


Fig. 12 Stress distribution in PEM simulation at PEM swelling

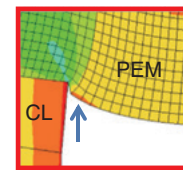


Fig. 13 Enlarged view of red frame in Fig. 12

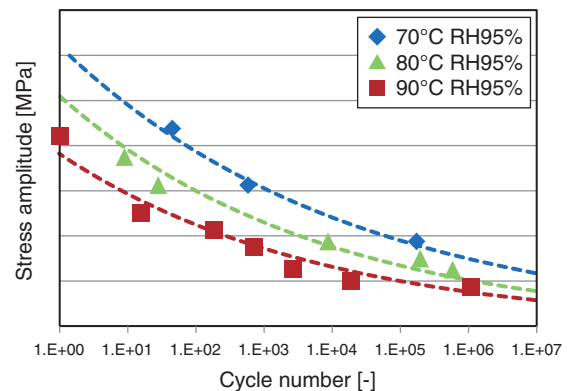


Fig. 14 Results of S-N diagram at various temperature conditions

10^6 times on the PEM fatigue curve. Therefore, when calculating the Lcr shown in Eq. (4), the fact that stress fatigue is input even at low stress was reflected, and the material limit line was calculated from the cumulative fatigue including low stress input.

4.4. Lcr Calculation Using Cumulative Fatigue Damage Law

4.4.1. Generated stress prediction by stress analysis using FEM

The stress analysis using FEM described in section 4.2. was performed to calculate the generated stress from the Young's modulus and dimensional change rate described in section 4.1. and the actual electrode crack width.

Figure 15 shows the relationship between the PEM dimensional change rate in the thickness direction at electrode cracks and the generated stress at each electrode crack width. These results show that the stress generated on the PEM at electrode cracks increases together with both the PEM swelling rate in the thickness direction and the crack width.

4.4.2. Lcr calculation

Lcr was calculated from the material limit line and generated stress values using the cumulative fatigue damage law as described in section 3.4. In addition, the calculations at each condition used the environmental conditions of the dry-wet cycle test at different RH described in term 3.4.2. Figure 16 shows the calculation results. These results show that Lcr increases together with the dry-wet difference, and the PEM is predicted to break at electrode cracks with a width of approximately 30 μm or more under durability test conditions of repeated RH 120% to dry cycle. However, the results also show that Lcr decreases as the dry-wet difference becomes smaller, and that fatigue degradation has little impact on the PEM under durability test conditions of repeated RH 30% to dry cycle.

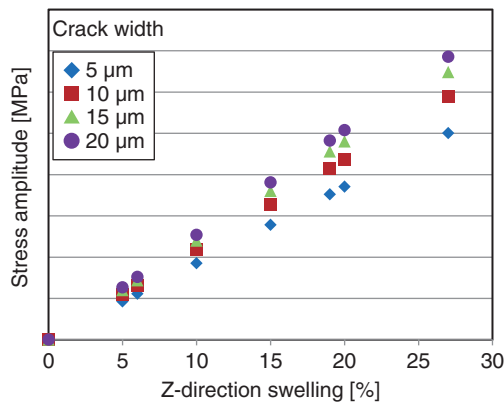


Fig. 15 Calculation results of stress amplitude at various conditions (FEM)

4.4.3. Results of membrane thickness analysis after dry-wet cycle test at different RH

Figure 17 shows the measurement results of the PEM thickness at electrode cracks after the dry-wet cycle test at different RH. The rate of decrease in the membrane thickness becomes more pronounced as the crack width increases, and the PEM was confirmed to break at an electrode crack width of approximately 30 μm . In addition, reducing the membrane thickness was confirmed to have an increased impact on membrane degradation at electrode cracks under conditions with a large dry-wet difference in the dry to wet direction, and this tendency matches the prediction obtained by stress analysis using FEM. This indicates that the dominant factor influencing the phenomenon of mechanical degradation of PEM is the increase in the stress generated at electrode cracks due to PEM swelling variation.

4.4.4. Lcr - membrane thickness conversion formula

The Lcr results relative to crack width calculated under each durability condition described in term 4.4.2. and the analysis results described in term 4.4.3. were used to determine the relationship between Lcr and the rate of

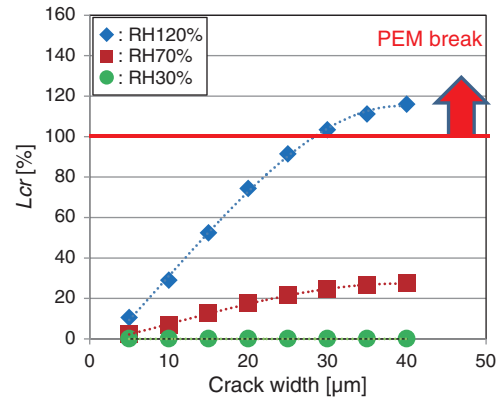


Fig. 16 Calculation results of Lcr in RH cycle test

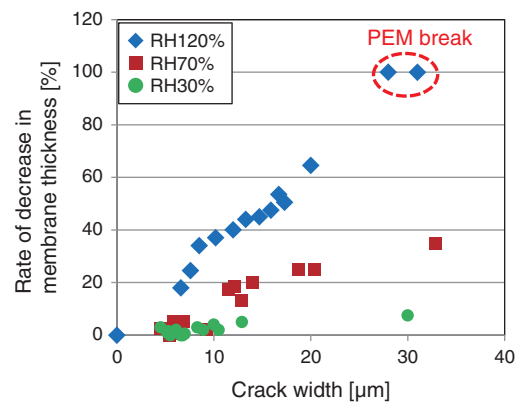


Fig. 17 Analysis results of membrane thickness in RH cycle test

decrease in the membrane thickness.

Figure 18 shows these results, indicating that the rate of decrease in the membrane thickness increases together with Lcr . This indicates that high stress is generated on the PEM at electrode cracks, which promotes mechanical degradation of the PEM, and that the Lcr determined from the relationship between the material limit line and the generated stress influences the rate of decrease in the membrane thickness. In addition, Lcr and the rate of decrease in the membrane thickness exhibited a high correlation even under durability conditions with different dry-wet differences. Based on these results, the membrane thickness, which decreases under the durability conditions, can be predicted by replacing the mechanical degradation stress with Lcr , even under different durability conditions. Potential methods for reducing mechanical degradation of PEM at electrode cracks include methods of controlling PEM water content fluctuation, enhancement of CL manufacturing technology, and increasing PEM fatigue durability. Use of the developed life prediction technology makes it possible to quantify the PEM thickness life-enhancing effects of each countermeasure method, which in turn enables investigation of the optimum countermeasure methods that can secure both sufficient power generation performance and durable reliability.

4.5. Results of Verification of Prediction Accuracy Using On-board-size MEA

Figure 19 shows the measurement results of membrane thickness after a mechanical degradation durability test using an on-board-size MEA. These results confirmed that mechanical degradation progresses in accordance with the electrode crack width, even in an on-board-size MEA. In addition, the results of analyzing the rate of decrease in the membrane thickness matched the trend in the predicted rate of decrease in the membrane thickness calculated using the developed life prediction technology. This confirmed that the developed life prediction technology can also be applied to an on-board-size MEA.

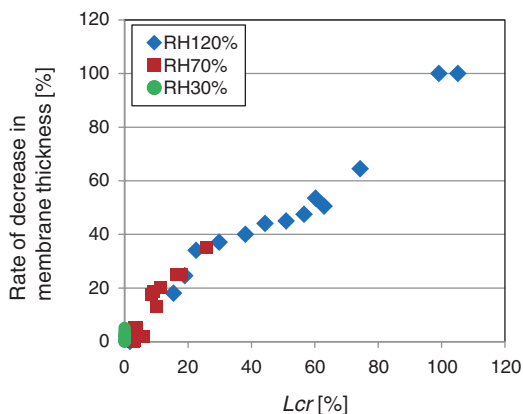


Fig. 18 Relationship between Lcr and rate of decrease in membrane thickness

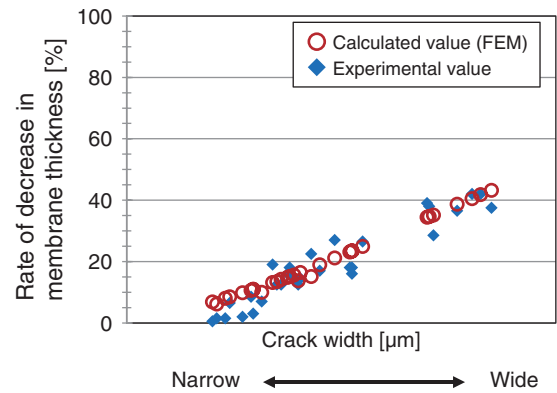


Fig. 19 Analysis results of membrane thickness in accelerated stress test using on-board-size MEA

5. Conclusion

Life prediction technology was established for PEM under mechanical degradation.

- (1) The PEM and CL property results showed that the CL has a higher Young's modulus than the PEM, which helps to reduce dimensional change in the in-plane direction. However, FEM analysis also showed that when electrode cracks are present, this is a factor increasing the stress in accordance with the electrode crack width.
- (2) The stress generated on the PEM in each temperature and humidity environment can be estimated by applying the material property values at each temperature and humidity to the FEM model, and this estimated stress is correlated with the results of dry-wet cycle tests at different RH.
- (3) The impact of mechanical degradation on life can be quantified by using the cumulative fatigue damage law to define the PEM Lcr . In addition, it was confirmed that the membrane thickness after mechanical degradation durability can be predicted for an on-board-size MEA.

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