Improvement of Cell Performance in Low-Pt-Loading PEFC Cathode Catalyst Layers Prepared by the Electrospray Method

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The reduction of Pt loading in cathode catalysts is an important subject for developing polymer electrolyte fuel cells. In order to solve this problem without sacrificing performance, we prepared a uniform catalyst-coated membrane (CCM) by an electrospray (ES) method with a low Pt loading, 0.05 mg Pt cm
\(^{-2}\), which is one-tenth of the typical cathode Pt loading. The ES process has several advantages for the fabrication of thin catalyst layers (CLs), such as smaller droplets and accurate position control, compared with the pulse-swirl-spray (PSS) method. Highly uniform CLs were evaluated by use of a standard evaluation cell (area 29.2 cm\(^2\)). The ES method makes it possible to control precisely the coating area and reduce the losses of the catalyst ink. CLs cross sections prepared by focused ion beam milling indicated that the total occupation fraction of the pores in the CLs prepared by the ES method was about twice as large as that for the PSS method. The ES method improved the ionomer coverage, led to increased electrochemically active surface area, and constructed more porous CLs. The highly porous CLs prepared by the ES method contributed to the improvement of the cell performance.

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Polymer electrolyte fuel cells (PEFCs) are expected to be important alternative power generation systems for co-generation and fuel cell vehicles (FCV). The catalytic activity of the cathode is an important issue to address in order to improve the performance of PEFCs. Many researchers have focused on the development of new Pt alloy catalysts and have paid attention to the control of particle size and Pt-Pt particle distance. Our group confirmed that the effectiveness of Pt in catalyst layers (CLs) was only on the order of 7.5% under practical operating conditions (air, 65°C, 0.1 MPa), and proposed that the enhancement of the effectiveness would improve the mass activity of CLs by reducing the Pt loading amount in the CL to as low as 0.05 mg cm
\(^{-2}\).

The CLs are usually fabricated from a catalyst ink, which includes the Pt catalyst, an ionicomer binder and a dispersant solvent. The ink is typically coated onto the polymer electrolyte membrane by use of a method such as spray coating or one involving a doctor blade. Newer methods such as those involving inkjets, electrospinning, and electrospray (ES) have been developed to improve the precision of the ink coating on the membrane. In particular, the ES method is attractive due to its ability to stably eject sub-micrometer-sized droplets of the highly viscous catalyst ink. This method relies on the existence of a Taylor cone, which is a conically shaped ink meniscus that forms at the tip of a glass capillary when a high voltage is applied. The droplets ejected from the Taylor cone are electrically charged and thus remain as separate, uniformly small droplets due to electrostatic repulsion. The superiority of the ES method lies in its ability to control precisely the coating area and reduce the losses of the catalyst ink. The ES method makes it possible to control precisely the coating area and reduce the losses of the catalyst ink. CLs cross sections prepared by focused ion beam milling indicated that the total occupation fraction of the pores in the CLs prepared by the ES method was about twice as large as that for the PSS method. The ES method improved the ionomer coverage, led to increased electrochemically active surface area, and constructed more porous CLs. The highly porous CLs prepared by the ES method contributed to the improvement of the cell performance.

Experimental

Preparation and evaluation of catalyst ink.—Pt catalyst supported on Ketjenblack (Pt/CB, TEC10E50E, Pt loading percentage 46 wt%, Tanaka Kikinzoku Kogyo K. K.) and a Pt catalyst supported on graphitized carbon black (Pt/CGB, TEC10EA30E, Pt loading percentage 29 wt%, Tanaka Kikinzoku Kogyo K. K.) were applied as the anode and cathode, respectively. Each of the commercial catalysts was mixed with Nafion binder (DE521, E. I. Du Pont de Nemours & Co., Inc.) and solvent (2-propanol:high purified water = 1:1 (vol. ratio) by use of a planetary ball mill (270 rpm, 30 min, P-6, Fritsch GmbH) and were pulverized by use of a wet pulverizing/dispersing device (150 MPa, Star Burst Mini, Sugino Machine, Ltd.) The identical ink composition was used for both the ES method and the pulse-swirl-spray coating (PSS) method. The ink was developed specifically for the PSS method. We optimized other conditions for the ES based on the ink composition of the PSS. The particle size distribution in the catalyst ink was analyzed by use of a particle analyzer (FPAR-1000, Otsuka Electronics Co., Ltd.). The cumulative pore volume in the CB itself was also estimated by the mercury intrusion method (Auto-Pore IV9520, Micromeritics Instruments Co.).

ES method.—A schematic drawing of the ES method is shown in Fig. 1a. The device consists of a capillary nozzle, usually made from a thin, hypodermic needle, and an extractor electrode plate with a single perforation. Usually, the capillary nozzle is connected to a high-voltage supply, while the perforated electrode plate and electrically conductive substrate are grounded. The capillary nozzle (inner diameter 210 μm) is equipped with an ink bottle containing the catalyst ink. The capillary nozzle is positioned above the substrate (surface temperature 55°C). An electrolyte membrane is placed on the substrate. A high voltage supply (610E, Trek Japan Co., Ltd.) is connected between an electrode, which is fitted into the capillary nozzle (positive), and the substrate (negative). The nozzle, equipped with the ink bottle, is positioned above the substrate at a vertical
The cathode CLs were confirmed to be 0.048 mg cm$^{-2}$ (PSS) and 0.056 mg cm$^{-2}$ (ES), and that of the anode CLs was 0.50 ± 0.02 g cm$^{-2}$. The high Pt loading at the anode was prepared so that there would be negligible anode polarization under all of the experimental conditions. The droplets on the membrane of both methods were observed by use of a digital microscope (VHX-1000, Keyence Co.). Scanning electron microscopic images (SEM) of the catalyst layers were obtained in the SEM mode with a scanning transmission electron microscope (STEM, HD-2700, Hitachi High-Technologies Co.). Cross-sectional samples were prepared by use of a focused ion beam milling system (FIB, FB-2200, Hitachi High-Technologies Co.). The average thicknesses of the CLs were estimated from the values measured at 120 positions on the CL. The occupation fractions of the pores in the CLs, based on the high and low magnification of binary SEM images, were estimated with an image analysis system (LUZEX-AP, Nireco Co.). The distributions of the ionomer coated on the Pt and carbon support particle surfaces were observed by use of TEM (HT7700S, Hitachi High-Technologies Co.), with which it is possible to observe the sample with low acceleration voltage (80 kV); this microscope was also equipped with a special high resolution objective lens. This equipment makes it possible to observe soft organic ionomers and inorganic carbon materials at high resolution and high contrast with minimal beam damage.

**Evaluation of Fuel cell performances.**—Cyclic voltammograms (CVs) of each of the cathode CLs were measured with a potentiostat (HZ-5000, Hokuto Denko Co.) during H$_2$ flow to the anode (100 mL min$^{-1}$) and a static N$_2$ atmosphere at the cathode, both at ambient pressure. Prior to the sweep, the cathodic potential was maintained at 0.075 V for 3.0 s. Then, the potential was swept from 0.075 V to 1.000 V at 20 mV s$^{-1}$ and reversed back to 0.075 V. The electrochemically active surface areas (ECAs) of each of the Pt catalysts in the CLs were evaluated from the hydrogen adsorption charge of CVs based on a standard value of charge per unit area (Q$_H$ of 0.21 mC cm$^{-2}$) at 30% relative humidity (RH), 53% RH, 80% RH, 100% RH at 80°C.

The current-voltage (I-V) characteristics were evaluated under steady-state operation, after 5 min at each point. We measured the polarization curves in the current control mode. This mode is more common for the evaluation of cell performance of large size MEAs. The cell potential was controlled galvanostatically by use of an electronic load (PLZ-664WA, Kikusui Electronics Co., Ltd.) and was measured as a function of current density by use of a cell performance evaluation-system (FCE-1, Panasonic Production Technology Co., Ltd.). The cell resistance (at 1 kHz) was measured under load by use of a digital ac milliohmeter (Model 3566, Tsuruga Electric Co., Ltd.). H$_2$ was flowed to the anode and O$_2$/air to the cathode, and the flow rates of all gases were controlled by use of mass flow controllers. The utilization of reactant gases was 70% for H$_2$ and 40% for O$_2$/air. These gases were humidified at 30%, 53%, 80% and 100% RH at 80°C under ambient pressure (0.1 MPa).

**Results and Discussion**

**Construction of CLs.**—We investigated the shape of the Taylor cone as a function of applied voltage and distance between nozzle and substrate. The typical condition of the tip of the nozzle is shown in the images in Fig. 2a. In the low applied voltage region, under 3.4 kV, with a distance between nozzle and substrate (d) of 3.0 cm, the tip of the ink emanating from the nozzle had a rounded, droplet shape, without constructing a Taylor cone (designated by “x,” Fig. 2a, panel A). This condition is referred to as the “drip mode,” in which each droplet drops onto the membrane without atomization. In the intermediate region of applied voltage, between 3.3 kV and 4.3 kV, with d = 3.0 cm, the “Taylor cone” appeared on the tip of the nozzle (designated by “o,” Fig. 2a, panel B). This condition is referred to as the “cone-jet mode,” in which each droplet was dispersed by electrostatic repulsion to form submicrometer atomized droplets, which were subsequently deposited uniformly on the membrane. At applied voltages greater than 4.2 kV with d = 3.0 cm, the Taylor cone became unstable, with wobbling of the droplet surface (designated by...
The areas coated by PSS and ES methods are shown in Figs. 3a and 3b. The average thickness of the ES-cathode-CL (16.2 ± 0.7 μm) was approximately 3 times larger than that of the PSS-cathode-CL (5.5 ± 4.9 μm), in spite of having the same Pt loading amount. The surface roughness for both methods was nearly the same. We also observed that the large pores, those over 300 nm in diameter, which were estimated from the equivalent circles by the image analysis apparatus, were dispersed throughout both CLs. The occupation fractions of the pores for each CL are summarized in Table I. In the low magnification SEM images (Figs. 4a, 4b), the pore fractions for both PSS-cathode-CL and ES-cathode-CL were nearly the same, 0.22 ± 0.024 and 0.23 ± 0.021, respectively. The high magnification SEM images of both CLs are shown in Figs. 4c and 4d, and the corresponding pore parameters, which were estimated from the equivalent circles by the image analysis apparatus, are also listed in Table I. The total occupation fraction of the pores based on the high-magnification images for the ES-cathode-CL was 0.40 ± 0.044, which was about twice as large as that for the PSS-cathode-CL (0.23 ± 0.024). The pore distribution of the CB obtained by use of mercury porosimetry is shown in Fig. 5. The critical change of the cumulative pore volume at about 20 nm indicates the boundary between the primary and secondary pores of the CL. The primary pores are formed between the aggregates of CB (secondary particles of CB) [18,27]. The secondary pores are formed between the Pt/CB agglomerates, which are groups of aggregates. We also evaluated the primary and secondary pore fractions for both CLs prepared by the PSS and ES methods.

Microstructure of CLs prepared by PSS and ES methods.—Low magnification SEM images of the CLs obtained by the PSS (PSS-cathode-CL) and ES (ES-cathode-CL) methods are shown in Figs. 4a and 4b. The average thickness of the PSS-cathode-CL (16.2 ± 0.7 μm) was approximately 3 times larger than that of the PSS-cathode-CL (5.5 ± 4.9 μm), in spite of having the same Pt loading amount. The surface roughness for both methods was nearly the same. We also observed that the large pores, those over 300 nm in diameter, which were estimated from the equivalent circles by the image analysis apparatus, were dispersed throughout both CLs. The occupation fractions of the pores for each CL are summarized in Table I. In the low magnification SEM images (Figs. 4a, 4b), the pore fractions for both PSS-cathode-CL and ES-cathode-CL were nearly the same, 0.22 ± 0.024 and 0.23 ± 0.021, respectively. The high magnification SEM images of both CLs are shown in Figs. 4c and 4d, and the corresponding pore parameters, which were estimated from the equivalent circles by the image analysis apparatus, are also listed in Table I. The total occupation fraction of the pores based on the high-magnification images for the ES-cathode-CL was 0.40 ± 0.044, which was about twice as large as that for the PSS-cathode-CL (0.23 ± 0.024). The pore distribution of the CB obtained by use of mercury porosimetry is shown in Fig. 5. The critical change of the cumulative pore volume at about 20 nm indicates the boundary between the primary and secondary pores of the CL. The primary pores are formed between the aggregates of CB (secondary particles of CB) [18,27]. The secondary pores are formed between the Pt/CB agglomerates, which are groups of aggregates. We also evaluated the primary and secondary pore fractions for both CLs prepared by the PSS and ES methods.
types of CLs, prepared using the PSS and ES methods, according to the boundary at 20 nm, in Table I. The fraction of small pores (<20 nm in diameter) in the high magnification images of the ES-cathode-CL was 0.072 ± 0.0069, which was more than twice as large as that for the PSS-cathode-CL (0.031 ± 0.0042). The fraction of large pores (>20 nm in diameter) in the high magnification images of the ES-cathode-CL was 0.33 ± 0.036, which was more than 1.5 times larger than that for the PSS-cathode-CL (0.20 ± 0.022). These results indicate that a more highly porous CL is able to be fabricated by the ES method than by the PSS method, because the pore volume, which is formed from the primary and the secondary pores, was increased due to the existence of the small ink droplets formed by the electrostatic repulsion. Therefore, the thickness of the CL obtained with the ES method was much larger than that for the CL obtained with the PSS method.

Performance of the cathodes fabricated by the PSS and ES methods.—The CVs for the ES-cathode-cell cathode and PSS-cathode-cell cathode (80°C, 100% RH) exhibited the usual hydrogen absorption/desorption peaks and Pt oxidation/reduction peaks (Fig. 6a); these peaks were rather similar for the two cathodes. The ECA values, which were determined from the hydrogen adsorption peak for the ES-cathode-cell at 80°C and 30–100% RH, increased 20–10% with increasing humidity and were larger than those for the PSS-cathode-cell (Fig. 6b). The ECA value at 100% RH at 80°C was 0.33 ± 0.0042. These results indicate that the ECA values indicated that the ES method was also effective in improving the ionomer coverage. TEM images shown in Figs. 7a, 7b indicate that the ionomer coverage on catalyst particles in a CL prepared using the ES method was clearly larger than that for a CL prepared using the PSS method. The ionomer thickness in the ES CL was approximately 5 nm, and the ionomer was spread uniformly on most of the graphitized carbon surfaces. In contrast, the ionomer in the PSS CL was unevenly distributed. With the ES method, the ink droplets were quite small, in the micrometer range, and thus the drying process during CL fabrication might be faster than that for the PSS method (Figs. 3c, 3d). Such an acceleration of the drying might have an effect on the ionomer coverage.

Fig. 8 shows IR-free polarization curves for the ES-cathode-cell and PSS-cathode-cell at 80°C at 80% RH under O2/air cathode conditions. The Tafel slopes for the ES-cathode-cell and PSS-cathode-cell were 84.3 mV decade⁻¹ and 89.5 mV decade⁻¹, respectively, indicating that nearly the same kinetic behavior was occurring for the oxygen reduction reaction (ORR) in both cathode CLs. The cell voltage of the ES-cathode-cell was higher than that of the PSS-cathode-Cell for current densities below 0.5 A cm⁻². The dependence of the mass activity (MA) on humidity is shown for both cells for the 30–100% RH range in air in Fig. 9. The MA values for the ES-cathode-cell at 0.85 V at each humidity were more than twice as large as those for the PSS-cathode-cell. The improvements of the MA values for the ES-cathode-cell were significantly higher than those for the ECA values (20–10%). These results suggest that the effectiveness of Pt¹⁰ was improved by the use of the ES method, in addition to the improvement of the Pt utilization.

Fig. 10 shows the current density and ohmic resistance of both cells at 0.70 V under the various RH conditions. The superiority of the performance of the ES-cathode-cell compared to that of the PSS cell decreased with decreasing RH. In order to compare the cell performance to previous cell performance,²⁰⁻²² Table II summarizes the current per unit cathode Pt at 0.70 V and maximum power for selected evaluation conditions. These data indicate that the performance of our ES cathode is superior to those in previous reports, because our evaluation conditions included air with zero backpressure. The
Figure 6. (a) Cathode cyclic voltamograms of ES\textsubscript{cathode-cell} and PSS\textsubscript{cathode-cell} at 80°C, 100% RH, and (b) Humidity dependence of the ECA of ES\textsubscript{cathode-cell} and PSS\textsubscript{cathode-cell} at 80°C.

Figure 7. Morphologies of the ionomer covered on carbon-supported Pt catalysts in the PSS electrode (a) and ES electrode (b). The dashed line indicates the carbon surface, and the solid line indicates the ionomer surface.

Figure 8. IR\textsubscript{-free} polarization curves for the ES\textsubscript{cathode-cell} and PSS\textsubscript{cathode-cell} at 80°C, 80% RH, air/H\textsubscript{2}, ambient pressure.

Figure 9. Humidity dependence of mass activity at 0.85 V (IR\textsubscript{-free}), 80°C, air/H\textsubscript{2}, ambient pressure.

Figure 10. Humidity dependence of the current density and the ohmic resistance at 0.7 V (IR\textsubscript{-free}), 80°C, air/H\textsubscript{2}, ambient pressure.

difference between the ohmic resistances of both cells increased with decreasing RH. The ionomer contents of both cells were nearly the same. The ionomer coverage was apparently improved by the use of the ES method. The average ionomer thickness on the Pt/CB surface using the ES method is estimated to be smaller than that using the PSS method. Therefore, we consider that the performance superiority of the ES\textsubscript{cathode-cell} should decrease with decreasing RH, because the ionic resistance of the ES\textsubscript{cathode-cell} increased more rapidly with decreasing RH than that for the PSS\textsubscript{cathode-cell}. The high ohmic resistance of the ES electrode was caused by the ionic resistance due to the large CL thickness and small ionomer thickness. At high relative humidity, the large CL thickness caused long proton paths in the CL, and the ohmic resistance increased. However, the ionomer distribution in the ES electrode was uniform and continuous, based on the ECA and MA values, and the difference in the ohmic resistance was
small. At low relative humidity, the small ionomer thickness in the ES electrode was affected more strongly, because the ionomer network was easy to interrupt.

The $O_2$ gains for both cells, which were obtained under pure $O_2$ and air feed, are shown in Fig. 11 as a function of the current density at 80 $^\circ$C and various RH values. The $O_2$ gain values measured for the ES-cathode-cell at current densities lower than 0.5 A cm$^{-2}$ at 80–100% RH were lower than those for the PSS-cathode-cell. This indicates that the gas supply network of the cathode was improved under these conditions. The highly porous CL obtained using the ES method was considered to have contributed to the improvement of the gas mass transport at current densities below 0.5 A cm$^{-2}$ for 80–100% RH. However, for higher current densities and lower RH, the superiority of the ES-cathode-cell disappeared. We consider that the thick CL obtained with the ES method led to losses of mass transport capability for gas or protons in the high current density range and led to low proton conductance under low RH conditions. For the ES-cathode-cell, there is clearly room for further improvements in ionomer content, Pt loading percentage, type of carbon, and types of other support materials. It is true that the absolute value of the power density for the ES-cathode-cell was far below the USDOE performance target. However, our evaluation conditions were different from the DOE conditions, including the Pt loading and the use of backpressure and so on. It should be noted that our purpose was the improvement of the pore structure in the CL, the CL thickness and the performance of the cell. Therefore, our cell performance should not be compared directly with DOE performance targets. We believe that if we adopt alloy catalysts, and improve the Pt-Pt particle distance in the CL, we might be able to match the DOE performance and durability targets in the near future.

**Conclusions**

We prepared and evaluated cathode CLs by use of the ES method from Pt nanoparticle catalysts supported on CB. By optimizing the various preparation conditions, i.e., distance between the nozzle and membrane, the applied voltage, and the nozzle diameter, etc., we were able to obtain a stable ejection of the submicrometer ink droplets. CLs with highly uniform characteristics were obtained. The ES method enabled us to precisely control the coated area and to reduce the loss of catalyst ink. The ES method also led to improved ionomer coverage, increased ECA values, and more highly porous CLs. The highly porous CL prepared by the ES method contributed to the improvement of the MA and the cell performance due to the increased efficiency of gas mass transport at current densities below 0.5 A cm$^{-2}$ at 80–100% RH. We propose that the new method using ES method for the preparation of thin CLs with very Pt low loading might be attractive in order to improve the performance and reduce costs for PEFCs.

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