

# Effect of Particle Size of Platinum and Platinum-Cobalt Catalysts on Stability Against Load Cycling

Towards the development of high performance, stable fuel cell catalysts with low platinum loadings

doi:10.1595/147106710X523698

<http://www.platinummetalsreview.com/>

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*To investigate the effect of load cycling, platinum (Pt) and platinum-cobalt (PtCo) fuel cell catalysts with different particle sizes were prepared and evaluated for their durability against load cycling. The particle size of the Pt and PtCo catalysts was controlled by changing the catalyst loading and by applying heat treatment. Pt catalysts with particle sizes of 2–3 nm and 4–5 nm and PtCo catalysts with sizes of 3–4 nm, 4–5 nm and 7–8 nm were obtained. A potential sweep from 0.65 V to 1.05 V was applied to the cathode of membrane electrode assemblies (MEAs) prepared with these catalysts, and the degradation of their mass activity and cell voltage were evaluated. As a result of this investigation, it was found that Pt catalysts with particle sizes of 4–5 nm and PtCo catalysts of particle sizes 7–8 nm showed better stability against potential sweep, with the Pt catalysts of sizes 4–5 nm showing the best stability of all the catalysts tested.*

## 1. Introduction

Polymer electrolyte membrane fuel cells (PEMFCs) are becoming more attractive and practical as power sources for automotive, small stationary and portable applications. However, there are still some issues which have to be overcome in order to realise the full commercial potential of fuel cell systems. These include improving the performance of the platinum-based catalyst and its stability against load change during fuel cell operation, improving the durability of the support material and decreasing the overall cost of the fuel cell system. Of these, improving catalyst stability in order to prevent catalyst degradation is one of the most significant.

Several authors (1–3) have studied cathode catalyst degradation during fuel cell operation by using an accelerated degradation test, for example applying a potential sweep or a series of potential steps to the

catalyst. Kinoshita *et al.* (1) reported that the decrease in surface area of the catalyst was accelerated by the application of a potential sweep in sulfuric acid solution. Patterson *et al.* (2) reported that the surface area of the catalyst was reduced to half of its initial value after a potential sweep of 6500 cycles from 0.87 V to 1.2 V in a PEMFC. Yu *et al.* (3) demonstrated a similar experiment in which they reported that platinum band formation in the membrane was observed for both a platinum catalyst and a platinum-cobalt catalyst after 2400 cycles of potential sweep.

Ferreira *et al.* (4) made a detailed investigation into the MEA after potential sweep, and reported that the degradation of the Pt catalyst was caused by the dissolution and redeposition of Pt, which leads to dissolved Pt being redeposited within the ionomer. Makharia *et al.* (5) investigated the durability of the carbon support at several voltages (1.0, 1.1, 1.2 and 1.3 V) and found that carbon weight loss is dependent on the level of cell voltage. Weight loss increases with increasing cell voltage. On the other hand, Tada *et al.* (6) and Chen *et al.* (7) reported that a significant decrease in catalyst surface area occurred even under constant current operation.

The purpose of the present study was to improve the stability of Pt and PtCo catalysts, especially against load change. A potential sweep was applied to the cathode to simulate load change during fuel cell operation. To evaluate the stability of the catalyst, MEA performance was measured before and after potential sweep and the values were compared. Pt and PtCo catalysts with different catalyst loadings were tested. As a result of these investigations it was

found that changing the catalyst particle size by heat treatment was the most effective method of stabilising the catalyst. Here, the effect of the particle size of Pt and PtCo catalysts on their stability against load cycling is reported.

## 2. Experimental Details

### 2.1 Catalyst Preparation

The Pt catalysts (denoted 30% Pt and 50% Pt in Table I) were prepared by chemical deposition of Pt in a water-based solution onto a high surface area carbon support (surface area: 800 m<sup>2</sup> g<sup>-1</sup>). The metal loading of the catalysts was controlled at 30 wt% and 50 wt%. After the deposition of Pt, the catalysts were well washed and dried out in an oven at 60°C.

To control Pt particle size, heat treatment was then applied to the 30% Pt and 50% Pt catalysts to produce the heat-treated Pt catalysts (denoted 30% Pt-HT and 50% Pt-HT in Table I). Each catalyst was put into a quartz tube and the tube was purged with nitrogen. Then the tube was inserted into an electrical furnace and heated up to 900°C under reducing conditions under a flow of hydrogen and nitrogen. The heat treatment was applied to the catalyst for 30 minutes.

The PtCo alloy catalysts were prepared by chemical deposition of Co onto the 30% Pt and 50% Pt catalysts followed by heat treatment (these are denoted 30% PtCo-HT and 50% PtCo-HT in Table I). The heat treatment conditions were the same as those used for the Pt catalysts. A PtCo catalyst with larger particle size (denoted 50% PtCo-HHT in Table I) was prepared by higher-temperature heat treatment. After the heat treatment, a leaching treatment with nitric

Table I

Metal Loading and Particle Size of Platinum and Platinum-Cobalt Catalysts

| Catalyst description | Platinum loading, wt% | Cobalt loading, wt% | Pt:Co molar ratio | Particle size (XRD), nm | Particle size (TEM), nm |
|----------------------|-----------------------|---------------------|-------------------|-------------------------|-------------------------|
| 30% Pt               | 28.0                  | –                   | –                 | 2.5                     | 2–3                     |
| 30% Pt-HT            | 30.5                  | –                   | –                 | 4.3                     | 4–5                     |
| 50% Pt               | 46.5                  | –                   | –                 | 2.5                     | 2–3                     |
| 50% Pt-HT            | 50.5                  | –                   | –                 | 5.0                     | 4–5                     |
| 30% PtCo-HT          | 28.0                  | 2.7                 | 3.1:1             | 3.5                     | 3–4                     |
| 50% PtCo-HT          | 46.3                  | 5.0                 | 2.8:1             | 5.0                     | 4–5                     |
| 50% PtCo-HHT         | 45.0                  | 7.0                 | 1.9:1             | 7.0                     | 7–8                     |

acid was carried out on each PtCo catalyst to remove excess Co.

## 2.2 Catalyst Characterisation

**Table I** shows the metal loading and particle size of the Pt and PtCo catalysts. The Pt loading of the Pt catalysts was obtained by the ash method as follows: about 0.1 g of catalyst sample was weighed and put into a crucible. A lid was placed on the crucible which was then placed in a muffle furnace. The temperature was increased from room temperature to 800°C and maintained for 1 hour to burn out all the carbon support. After cooling to room temperature, the crucible was weighed again. The Pt loading was calculated from the initial sample weight and the weight of the residue after burning out the carbon support.

The total metal loadings of the PtCo catalysts were also obtained by the ash method. The Pt:Co ratio of each PtCo catalyst was obtained by X-ray fluorescence (XRF) analysis and used to calculate the respective loadings of Pt and Co as shown in **Table I**. The average particle size of each catalyst was estimated from transmission electron microscopy (TEM) and X-ray diffraction (XRD). These values are also shown in **Table I**.

## 2.3 Ink Preparation

The catalyst ink was prepared as follows. About 1 g of catalyst powder containing 0.5 g of carbon was put into a zirconia pot of volume 200 ml with 0.6 g of ionomer (Nafion<sup>®</sup> DE521CS) powder, 18 ml of solvents (1:1:1 water:*n*-propanol:2-propanol by volume) and 200 g of 5 mm-diameter zirconia balls and mixed by planetary ball milling for 50 minutes at 200 rpm with a rotation:revolution ratio of 1:1. The carbon:ionomer ratio in the ink was fixed at a dry weight ratio of 1:1.2 for all catalysts. After storage for 3 days in a refrigerator, the catalyst ink was used to prepare the catalyst layer of the MEA.

## 2.4 Membrane Electrode Assembly Preparation

The gas diffusion layer was prepared by the following two steps. First, a slurry containing a mixture of fluorinated ethylene-propylene (FEP) copolymer and carbon powder was printed onto carbon paper using a bar coater, then dried and calcined. This layer works as a hydrophobic layer. Second, a slurry containing a mixture of Nafion<sup>®</sup> and carbon powder was printed onto the carbon/FEP layer using the bar coater then

dried and hot pressed. This layer prevents penetration of the catalyst ink into the gas diffusion layer during coating of the catalyst layer.

Next, the catalyst ink was printed onto the gas diffusion layer using the bar coater then dried and hot pressed at 130°C for 30 minutes with a pressure of 20 kg cm<sup>-2</sup>. The Pt loading of each electrode was controlled at 0.4 mg cm<sup>-2</sup> for the 30% catalysts and 0.5 mg cm<sup>-2</sup> for the 50% catalysts by changing the coating times and bar size. These electrodes were used for the cathode. The anode was prepared in the same manner using the 50% Pt catalyst. The Pt loading of all anode catalyst layers was controlled at 0.5 mg cm<sup>-2</sup>.

A single fuel cell of area 25 cm<sup>2</sup> was assembled by sandwiching a membrane (Nafion<sup>®</sup> NRE212CS, membrane thickness: 50 μm) with the anode and the cathode using carbon plates with a single serpentine gas flow channel. A schematic image of the cell is shown in **Figure 1**. The compression was controlled by a spring at 25 kg cm<sup>-2</sup>. No hot press was applied to the MEA before assembling the single cell.

## 2.5 Membrane Electrode Assembly Measurement

MEA measurement was carried out before and after potential sweep to evaluate the catalyst stability. The procedure was as follows:

- Measurement of initial MEA performance*: after a pretreatment, current–voltage (I–V) performance and mass activity at 0.9 V were measured with hydrogen (1000 standard cubic centimetres per minute (sccm), fully humidified) and oxygen (1000 sccm, dry) at 80°C. Cyclic voltammetry was also performed with hydrogen (50 sccm, fully humidified) and nitrogen (20 sccm, dry) at a scan rate of 10 mV sec<sup>-1</sup> at the same temperature;
- Slow potential sweep*: a potential sweep from 650 mV to 1050 mV was applied to the cathode at 40 mV sec<sup>-1</sup> for 3600 cycles (20 hours) at 80°C (to confirm whether the pretreatment was sufficient);
- Measurement of MEA performance after slow potential sweep*: measurement of the MEA performance and cyclic voltammetry were carried out in the same way as described for step (a);
- Fast potential sweep*: a potential sweep from 650 mV to 1050 mV was applied to the cathode at 100 mV sec<sup>-1</sup> for 10,800 cycles (24 hours) at 80°C;

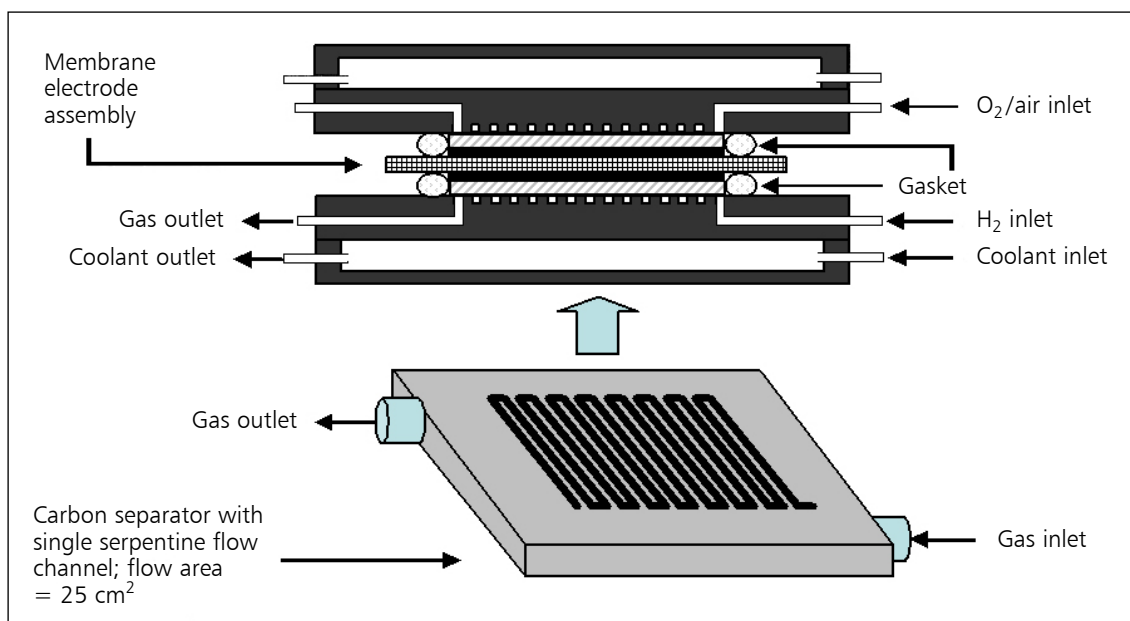


Fig. 1. Schematic image of the single fuel cell with a serpentine gas flow channel

- (e) *Measurement of MEA performance after the fast potential sweep for 10,800 cycles*: measurement of the MEA performance and cyclic voltammetry were carried out in the same way as described for step (a);
- (f) *Second fast potential sweep*: a potential sweep was applied in the same way as described for step (d);
- (g) *Measurement of MEA performance after the fast potential sweep for 21,600 cycles*: measurement of the MEA performance and cyclic voltammetry were carried out in the same way as described for step (a).

### 3. Results and Discussion

#### 3.1 Particle Size of Platinum Catalysts

Figure 2 shows TEM images of the 30% Pt, 30% Pt-HT, 50% Pt and 50% Pt-HT catalysts. Fine particles and good metal dispersion were obtained for the 30% Pt and 50% Pt catalysts. The Pt dispersion of the 30% Pt-HT and 50% Pt-HT catalysts remained good even after heat treatment. As shown in Table I, the average particle size of the 30% Pt and 50% Pt catalysts was estimated at 2–3 nm by TEM observation. The average particle sizes of the heat-treated catalysts (30% Pt-HT and 50% Pt-HT) were 3–4 nm and 4–5 nm, respectively. It was clear that Pt particles became

larger during heat treatment at high temperature, and this was thought to be due to agglomeration and sintering (8). It was also found that smaller Pt particles (<1 nm) disappeared after heat treatment. The catalyst particle size was also checked by XRD analysis; the average particle size of each catalyst was consistent with that obtained by TEM observation.

Han *et al.* (8) reported the effect of heat treatment on the growth of Pt-based catalyst particles in detail. They found that growth of catalyst particles occurs by agglomeration and sintering during heat treatment. This process depends on the catalyst loading, and catalysts with a higher metal loading are expected to show more significant agglomeration and sintering. This was found to be the case in the present study, as slightly more catalyst particle growth was observed in the 50% Pt catalyst than in the 30% Pt catalyst after heat treatment, as shown by the XRD results (Table I).

The increase in catalyst particle size for both the 30% Pt catalyst and the 50% Pt catalyst was less than that observed by Han *et al.* (8), even though a higher heat treatment temperature was used in the present study. This is thought to be due to the use of a higher surface area carbon support ( $800 \text{ m}^2 \text{ g}^{-1}$  vs.  $250 \text{ m}^2 \text{ g}^{-1}$  used by Han *et al.* (8)), which led to a smaller number of catalyst particles per unit area.

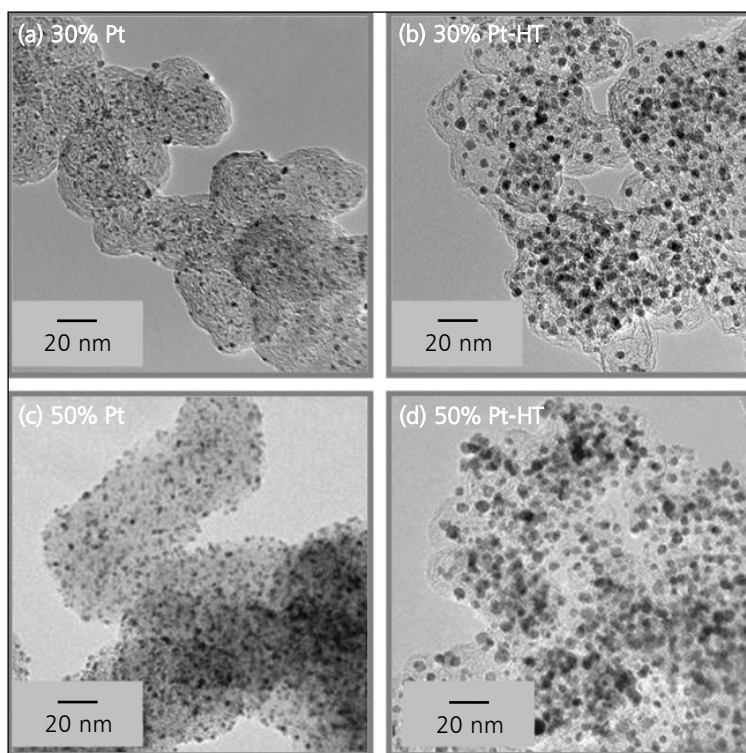


Fig. 2. Transmission electron microscopy (TEM) images of platinum catalysts with 30% or 50% metal loading, before and after heat treatment (HT) (a) 30% Pt; (b) 30% Pt-HT; (c) 50% Pt; (d) 50% Pt-HT

### 3.2 Performance of Platinum Catalysts

Figure 3 shows the MEA performance of the Pt catalysts before and after potential sweep measured with hydrogen and oxygen. There was no significant difference in initial MEA performance between the evaluated catalysts, indicating that heat treatment did not affect the initial MEA performance of either the 30% Pt catalyst or the 50% Pt catalyst.

As the number of cycles increased, the MEA performance of the 30% Pt and 50% Pt catalysts declined significantly. However, the decline in MEA performance of the heat-treated catalysts 30% Pt-HT and 50% Pt-HT was very small even after potential sweep for 21,600 cycles. This result suggests that heat treatment improved the catalyst stability in both cases.

The 30% Pt catalysts showed almost the same initial cell voltage as did the 50% Pt catalysts at every current density, even though the 30% Pt catalysts had a lower Pt loading on the electrode ( $0.4 \text{ mg cm}^{-2}$ ) than did the 50% catalysts ( $0.5 \text{ mg cm}^{-2}$ ). This suggests that the initial catalyst activity was increased by lowering the catalyst loading.

The cell voltage of all the Pt catalysts except for the 50% Pt catalyst was increased after slow potential sweep, as shown in Figure 3. This may be due to the

effect of heat treatment of the catalyst on the hydrophobicity or hydrophilicity of the electrode. Further optimisation of the electrode would therefore be necessary in order to use heat-treated catalysts efficiently.

Figure 4 shows the mass activities of the Pt catalysts before and after potential sweep for 21,600 cycles and the electrochemically active surface area (ECSA) loss after potential sweep for 21,800 cycles. The initial mass activity of the 30% Pt catalyst was higher than that of the 50% Pt catalyst for both the non-heat-treated and the heat-treated catalysts. The initial mass activity of the 30% Pt catalysts was increased after heat treatment, while that of the 50% Pt catalysts declined after heat treatment. Further investigation would be necessary to explain this discrepancy.

Mass activity was decreased after potential sweep for all the catalysts, and degradation was especially significant for the 50% Pt catalyst which showed a mass activity loss of 75%. The mass activity losses of the 30% Pt and the 50% Pt-HT catalysts were 20% and 19%, respectively. The 30% Pt-HT catalyst showed the smallest mass activity loss of all the Pt catalysts at only 11%. Similar trends were observed for the ECSA loss after the potential sweep for 21,800 cycles, therefore

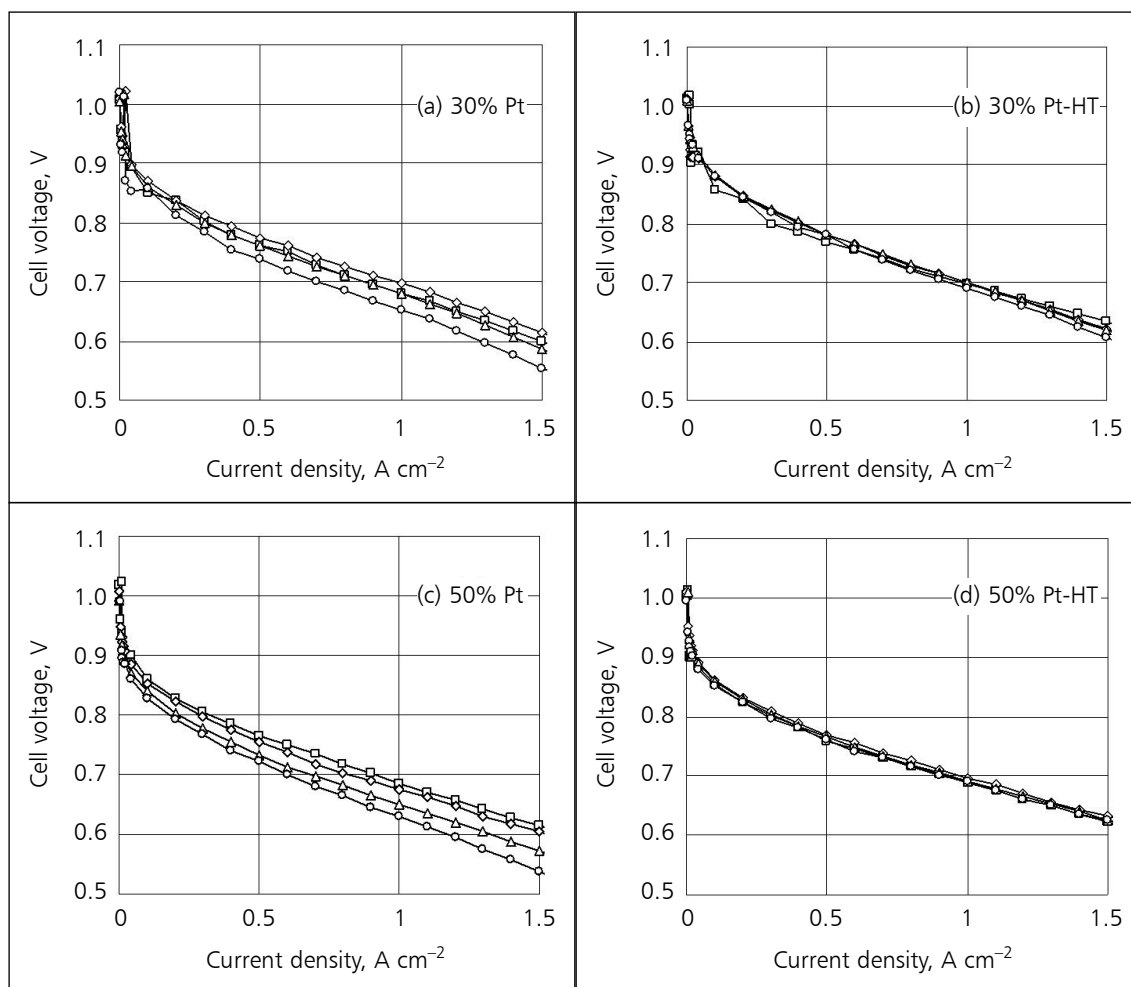


Fig. 3. Membrane electrode assembly (MEA) performance of platinum catalysts with 30% or 50% metal loading, before and after heat treatment (HT): (a) 30% Pt; (b) 30% Pt-HT; (c) 50% Pt; (d) 50% Pt-HT. Current-voltage performance measured with hydrogen and oxygen at 80°C: before pretreatment ( $\square$ ), after slow potential sweep ( $\diamond$ ), after fast potential sweep for 10,800 cycles ( $\triangle$ ), and after fast potential sweep for 21,600 cycles ( $\circ$ ). Flow rate of  $H_2/O_2$  was 1000/1000 sccm, humidity condition of  $H_2/O_2$  was 90°C/dry, gas pressure of  $H_2/O_2$  was ambient

it may be supposed that the mass activity loss was related to the decrease in ECSA.

### 3.3 Stability of Platinum Catalysts

In this study, a potential sweep from 0.65 V to 1.05 V was applied to the cathode to accelerate catalyst degradation. The upper voltage was limited to 1.05 V in order to prevent carbon corrosion from affecting the total performance degradation of the MEA. Makharia *et al.* (5) reported the effect of voltage on carbon weight loss. Based on their data, it was expected that the carbon support would not be

damaged if the upper voltage was limited to 1.05 V. Therefore, it was assumed that the catalyst degradation observed in the present study was dominated only by the dissolution and redeposition of Pt, as reported by Ferreira *et al.* (4). Figure 3 shows that the degradation of cell voltage could be controlled by heat treatment of the Pt catalysts. This is thought to be due to a higher initial surface area which reduces the possibility of dissolution and redeposition of Pt and hence particle size growth. In addition, the disappearance of very fine Pt particles (less than 1 nm) may also have a stabilising effect as these very fine particles are unstable.



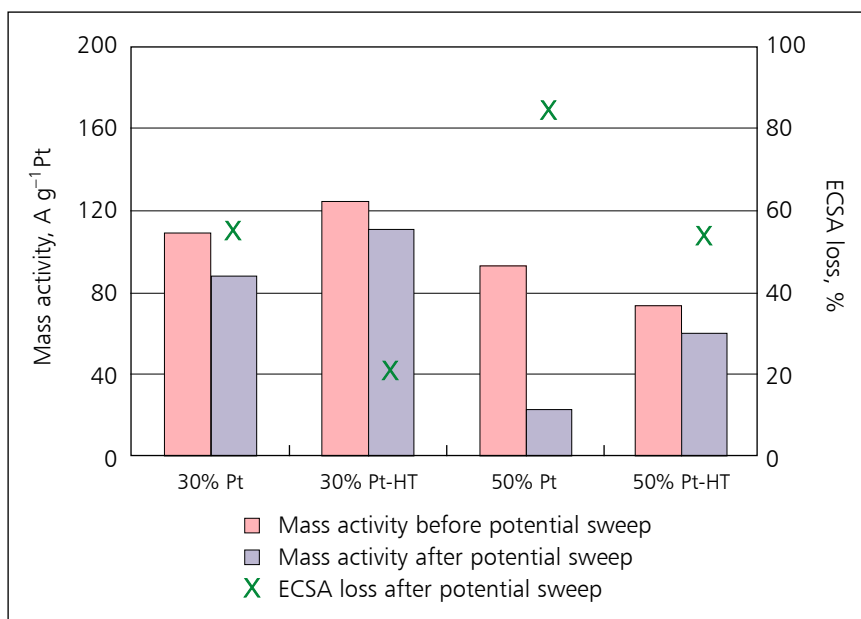


Fig. 4. Mass activity of platinum catalysts with 30% or 50% metal loading, before and after heat treatment (HT) at 0.9 V with hydrogen and oxygen at 80°C: before pretreatment and after fast potential sweep for 21,600 cycles. On the secondary axis the electrochemical surface area (ECSA) loss after 21,800 cycles is plotted

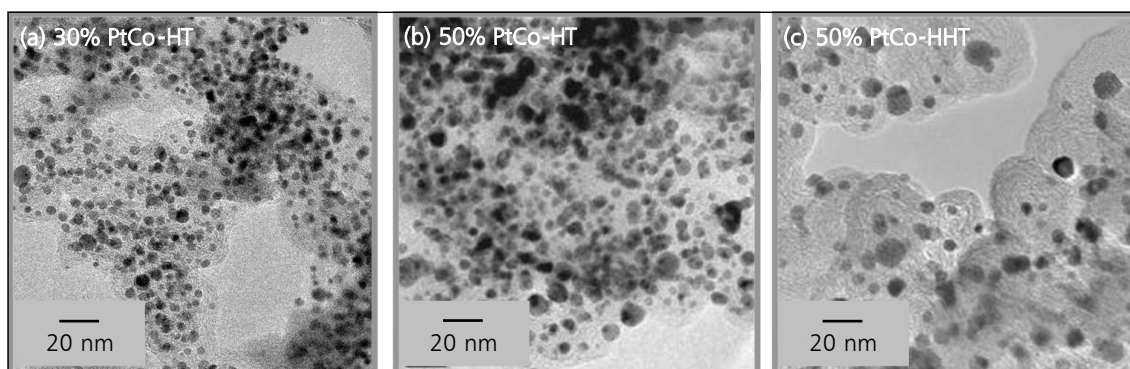


Fig. 5. Transmission electron microscopy (TEM) images of platinum-cobalt catalysts with 30% or 50% metal loading after heat treatment (HT) and after heat treatment at a higher temperature (HHT): (a) 30% PtCo-HT; (b) 50% PtCo-HT; (c) 50% PtCo-HHT

A decrease in metal loading of the catalyst would normally be expected to increase the catalyst surface area, and hence increase the catalyst activity. On the other hand, catalysts with a low metal loading tend to be less stable due to their small particle size. In this study, however, little difference was observed between the cell voltage of the 30% Pt catalyst and that of the 50% Pt catalyst, even though the 30% Pt catalyst electrode had a lower Pt loading. The 30% Pt catalyst showed better stability than the 50% Pt catalyst. This may be due to the effect of the distance between Pt particles, although further theoretical investigation would be necessary to fully understand this result.

### 3.4 Particle Size of Platinum-Cobalt Catalysts

Figure 5 shows TEM images of the 30% PtCo-HT, 50% PtCo-HT and 50% PtCo-HHT catalysts. The average particle sizes of the catalysts are given in Table I. The particle size and dispersion of the 30% PtCo-HT and 50% PtCo-HT catalysts were very similar to those of the 30% Pt-HT and the 50% Pt-HT catalysts, respectively, as both were subjected to heat treatment at the same temperature. There were no small metal particles on the carbon support for any of these catalysts. The particle size distributions of both the 30% and 50% PtCo-HT catalysts were uniform, but that of the 50% PtCo-HHT catalyst was not uniform and included

some very large particles due to the higher heat treatment temperature used.

### 3.5 Performance of Platinum-Cobalt Catalysts

Figure 6 shows MEA performances of the PtCo catalysts before and after potential sweep with hydrogen and oxygen. Although the surface area of the 30% PtCo-HT catalyst was higher than that of the 50% PtCo-HT catalyst, its initial cell voltage was lower. This suggests that more pretreatment is needed for the lower-loading catalysts.

The 30% PtCo-HT catalyst showed better performance than the 50% PtCo-HT catalyst despite its lower Pt loading. The performance loss of the 50% PtCo-HT catalyst after potential sweep for 21,600. The performance loss of the 50% PtCo-HT catalyst after the 21,600 cycles of fast potential sweep was the largest of all the evaluated catalysts. As the number of cycles increased, the MEA performance of all the heat-treated PtCo catalysts declined, unlike the heat-treated Pt catalysts. The high initial performance of the PtCo catalysts was thought to be due to the effect of Co increasing oxygen reduction reaction (ORR) activity, therefore the loss in MEA performance observed for the PtCo catalyst after potential sweep may be caused by the dissolution of Co into the ionomer during the accelerated degradation test. However, further investigation would be required to prove this and to fully understand the degradation mechanism.

Figure 7 shows the mass activity of the PtCo catalysts at 0.9V before and after potential sweep. The initial mass activity increased with decreasing metal loading (that is, increased metal surface area). The 50% PtCo-HHT catalyst showed high mass activity even though it had large particles (that is, decreased metal surface area). This may be due to the relatively high Co content of the 'as prepared' 50% PtCo-HHT catalyst (Pt:Co ratio was 1.9:1).

The 50% PtCo-HHT catalyst showed the highest mass activity of all the evaluated PtCo catalysts after the 21,600 cycles of fast potential sweep. During the cycling, Pt and Co dissolved into the ionomer. Less Co was dissolved out of the 50% PtCo-HHT catalyst, enabling it to maintain its mass activity even after cycling.

### 3.6 Stability of Platinum-Cobalt Catalysts

The degradation rate of the 50% PtCo-HT was 7% and that of 50% PtCo-HHT was 4% at a current density of 0.5 A cm<sup>-2</sup>. The initial performance of the 50%

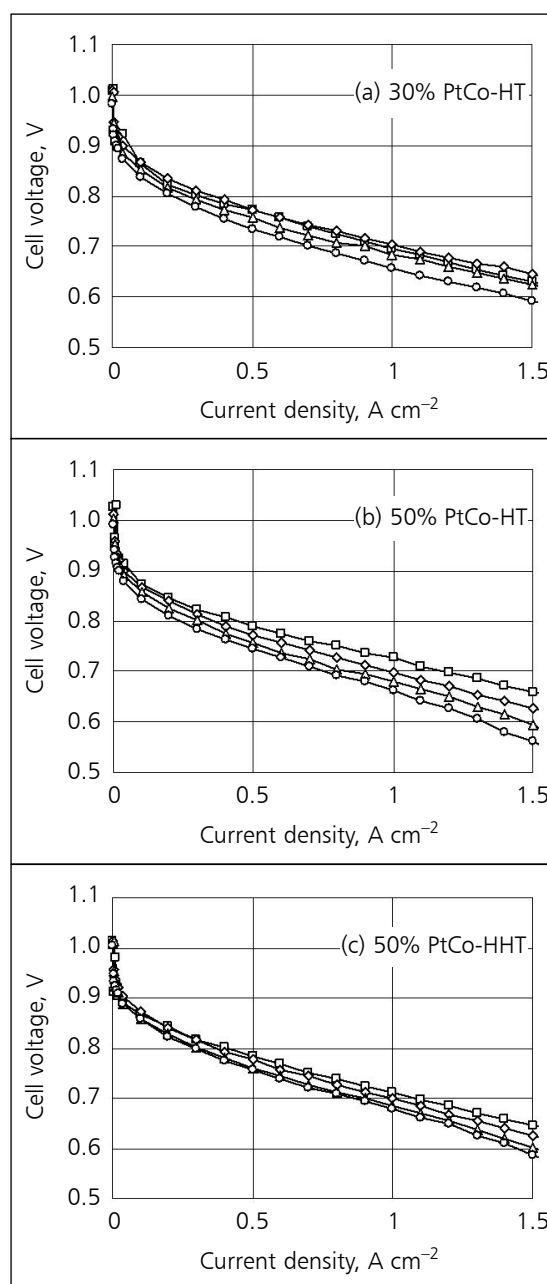


Fig. 6. Membrane electrode assembly (MEA) performance of platinum-cobalt catalysts with 30% or 50% metal loading, before and after heat treatment (HT) and after heat treatment at a higher temperature (HHT): (a) 30% PtCo-HT; (b) 50% PtCo-HT; (c) 50% PtCo-HHT. Current-voltage performance measured with hydrogen and oxygen at 80°C: before pretreatment ( $\square$ ), after slow potential sweep ( $\diamond$ ), after fast potential sweep for 10,800 cycles ( $\triangle$ ), and after fast potential sweep for 21,600 cycles ( $\circ$ ). Flow rate of H<sub>2</sub>/O<sub>2</sub> was 1000/1000 sccm, humidity condition of H<sub>2</sub>/O<sub>2</sub> was 90°C/dry, gas pressure of H<sub>2</sub>/O<sub>2</sub> was ambient



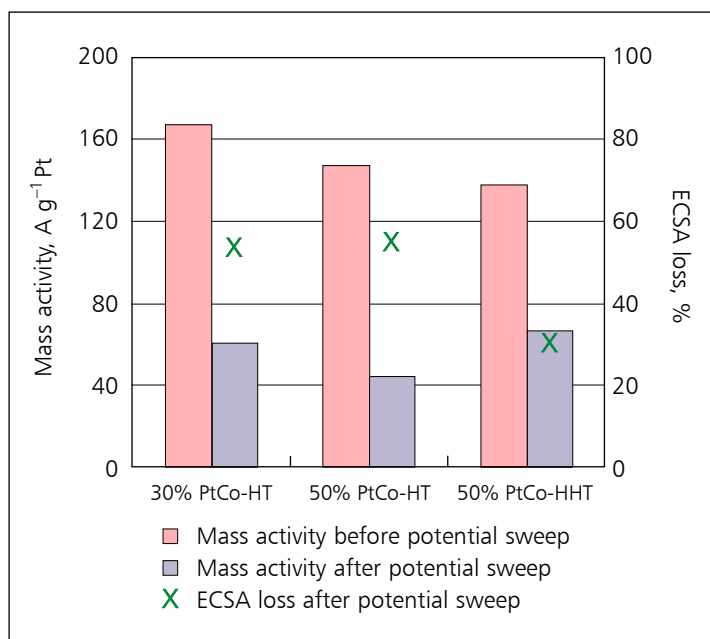


Fig. 7. Mass activity of platinum-cobalt catalysts with 30% or 50% metal loading, before and after heat treatment (HT) and after heat treatment at a higher temperature (HHT) at 0.9 V with hydrogen and oxygen at 80°C: before pretreatment and after fast potential sweep for 21,600 cycles. On the secondary axis the electrochemical surface area (ECSA) loss after 21,800 cycles is plotted

PtCo-HHT was lower than that of 50% PtCo-HT. However, this catalyst showed the highest MEA performance after the 21,600 cycles of fast potential sweep. This result suggests that catalysts with large particle sizes were more stable against load change.

### 3.7 Comparison between Platinum and Platinum-Cobalt Catalysts

The MEA performance of the 30% PtCo-HT catalyst was slightly better than those of the 30% Pt and 30% Pt-HT catalysts (see Figures 3 and 6). Furthermore, the 30% PtCo-HT showed the highest mass activity of all the 30% catalysts (see Figures 4 and 7). This confirms the positive effect of Co addition on catalyst performance. In terms of stability, however, the 30% PtCo-HT catalyst showed poor resistance against potential sweep, and the rates of degradation of cell voltage and mass activity of this catalyst were much larger than those of the 30% Pt and 30% Pt-HT catalysts. The 30% Pt-HT catalyst showed the best stability of all the 30% catalysts studied.

The 50% PtCo-HT and 50% PtCo-HHT catalysts showed high cell voltage (see Figures 3 and 6) and mass activity (see Figures 4 and 7). The 50% PtCo-HHT catalyst had a more stable cell voltage and mass activity compared to the 50% PtCo-HT catalyst; however, its cell voltage and mass activity degradation rates

were much higher. Therefore, it was also concluded that the 50% Pt-HT catalyst was the most stable of all the 50% catalysts studied.

### 4. Summary

In this study, it was found that 30% Pt catalysts showed excellent performance even though their Pt loading was low. The 30% Pt catalysts also had good stability against potential sweep. Heat treatment of the Pt catalysts (to give 30% Pt-HT, and 50% Pt-HT catalysts, respectively) improved their stability against potential sweep compared to the non-heat-treated catalysts.

The heat-treated PtCo catalysts 50% PtCo-HT and 50% PtCo-HHT showed better catalyst performance, indicating that the addition of Co as an alloying element could improve catalyst activity. The higher-temperature heat-treated 50% PtCo-HHT catalyst had a higher stability than the 50% PtCo-HT catalyst, although it was still lower than that of the heat-treated Pt catalysts.

There is normally a trade-off between improving the catalyst activity and the catalyst stability. However, the results of this study suggest that it is possible to prepare a catalyst with low metal loading which has both high performance and good stability. Further investigation of catalysts with low pgm loadings is ongoing.

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