



Fast stack activation procedure and effective long-term storage for high-performance polymer electrolyte membrane fuel cell



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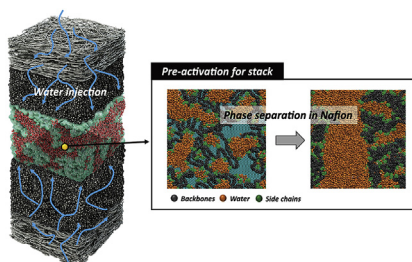
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HIGHLIGHTS

- Time-saving stack activation and long-term storage for PEMFC were investigated.
- The pre-activation by using DI water significantly reduces a whole activation time.
- The humidified N₂ gas minimizes the Pt oxidation and dying of polymer membrane.

GRAPHICAL ABSTRACT



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ABSTRACT

Time-saving stack activation and effective long-term storage are one of most important issues that must be resolved for the commercialization of polymer electrolyte membrane fuel cell (PEMFC). Herein, we developed the cost-effective stack activation method to finish the whole activation within 30 min and the long-term storage method by using humidified N₂ without any significant decrease in cell's performance for 30 days. Specifically, the pre-activation step with the direct injection of DI water into the stack and storage at 65 or 80 °C for 2 h increases the distinctive phase separation between the hydrophobic and hydrophilic regions in Nafion membrane, which significantly reduces the total activation time within 30 min. Additionally, the long-term storage with humidified N₂ has no effect on the Pt oxidation and drying of Nafion membrane for 30 days due to its exergonic reaction in the cell. As a result, the high water content in Nafion membrane and the decrease of Pt oxidation are the critical factors that have a strong influence on the activation and long-term storage for high-performance PEMFC.

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1. Introduction

The worldwide demand for the dramatic reduction of greenhouse gas emissions resulting from the use of fossil fuels has generated extensive interest in the possible use of polymer electrolyte membrane fuel cells (PEMFCs) as a clean and highly-efficient source of electric power for stationary and portable

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power applications [1–6]. In order for these cells to gain extensive acceptance in the world's energy markets by 2020, significant progress must be made in the areas of ensuring their durability, reducing their cost, enhancing their performance, and providing the infrastructure required for the production, storage, and transportation of hydrogen. Among the many technological issues that must be addressed, time-saving stack activation and effective long-term storage methods will be one of most important issues that must be resolved for the commercialization and the mass production of PEMFCs. Specifically, the most negative element of the stack activation is the high cost for initial installation and operation expenses of test station because the activation process needs a lot of time and hydrogen to increase the water content in polymer membrane and decrease the platinum (Pt) oxidation. Furthermore, PEMFCs' stacks frequently can be kept at atmospheric conditions for several days or months without any operations in the fabrication process and shipment, and this can result in a significant decrease the stack's performance, due primarily to the severe drying of the polymer membrane and the Pt oxidation. Interestingly, the both of the water content in polymer membrane and the Pt oxidation are the critical factors that have a strong influence on the activation and long-term storage. In this work, we suggested a new method to reduce the whole activation time to an hour only by applying the pre-activation of stack. The main processes of the pre-activation are the sufficient injection of distilled water into the stack and the storage at 65 or 80 °C for 2 h, leading to apparent phase separation between the hydrophobic and hydrophilic parts in Nafion membrane. In addition to stack activation, an effective long-term storage method for PEMFC was also investigated by using the humidified nitrogen (N₂) as a representative inert gas to minimize the decrease in the performance of the cells. To date, there have been no studies that tried to minimize the decrease in the performance of the cells during long-term storage of PEMFCs. In this work, we showed that long-term storage in N₂ had little influence on the performance of the cells due to its endergonic reaction.

2. Experimental

2.1. Activation of PEMFC

The commercial Gore PRIMEA 57 membrane-electrode-assembly (MEA) with active area of 25 cm² was used to conduct the activation of 10-cell stack. A membrane thickness was 18 μm. The Pt loadings on the anode and cathode electrodes were 0.1 and 0.4 mg_{Pt} cm⁻², respectively. In this work, total activation process was classified into pre- and vacuum-activations. Firstly, the pre-activation was conducted in the following the order: *i*) DI water was injected into the stack until void spaces in bipolar plate and catalyst layer were filled with water, and then the stack was kept in environmental chamber at 65 or 80 °C for 2 h; *ii*) after the pre-activation procedure, the residual DI water was removed from the stack by purging the humidified N₂ gas and then H₂ and air were supplied to the anode and cathode, respectively; *iii*) after confirming a stable open circuit voltage (OCV) of stack, the constant current at 0.65 V was retained for 30 min to decrease the Pt oxidation and produce the water molecules *via* oxygen reduction reaction (ORR). Finally, the vacuum-activation of continuous three steps were repeated for 30 min as follows: *i*) a high current density of 1.2 A cm⁻² was retained for 2 min to produce water molecules; *ii*) a low current density of 10 mA cm⁻² was retained to slowly reducing the voltage to 0.4 V without supplies of H₂ and air; *iii*) H₂ and air were supplied to anode and cathode, respectively. For the vacuum-activation process, the constant flow rates of H₂ and air were 4 and 12 L min⁻¹, respectively. All of the stack experiments

were conducted at 65 °C under relative humidity (RH) of 100% and ambient pressure. The coolant flow rate was 1 L min⁻¹. For the polarization curve of stack, the average value of the voltages that were obtained for the last 1 min of the 2-min period at each current was used. The stoichiometric ratios of H₂ and air were 1.43 and 2.0, respectively. The gas flow channel was single-serpentine parallel channels. The width, depth and land width in gas flow channel are 1 mm. The flow mode was counter-flow. In this work, we conducted a conventional activation by repeating the currents in a range of potential from OCV to 0.4 V until stabilizing the stack's performance for 8 h.

2.2. Long-term storage of PEMFC

The Gore PRIMEA 57 membrane-electrode-assembly (MEA) with a membrane thickness of 18 μm was used to conduct the long-term storage of the PEMFCs. The Pt loadings on the anode and cathode electrodes were 0.1 and 0.4 mg_{Pt} cm⁻², respectively. The pre- and vacuum-activations were used to increase an initial cell performance. After the cell activation, the MEAs with active areas of 1 and 25 cm² were kept in various gases in both dry and humidified conditions at room temperature for 30 days. All of the unit cell experiments were conducted at 65 °C and ambient pressure. After stabilizing at each current for 5 min, electrochemical impedance spectroscopy (EIS) spectra were obtained using an HCP-803 analyzer (BioLogics, Science Instruments) by sweeping frequencies over a range of 1000 kHz–100 mHz. For the polarization curve, we used the average value of the voltages that were obtained for the last 2 min of the 5-min period during the impedance measurement at each current. At currents less than 10 A, the flow rates of H₂ and air were 100 and 410 mL min⁻¹, respectively. At current values greater than 10 A, the stoichiometric ratios of H₂ and air were 1.43 and 2.5, respectively. In order to specifically investigate the Pt oxidation, we selected dry/humidified N₂ gas as the inert gas. X-ray photoelectron spectroscopy (XPS) was performed using a K-alpha instrument (Thermo Fisher) to analyze the Pt oxidation. For the XPS analysis, an MEA with an active area of 25 cm² was cut into small areas of 1 cm², and, then, the MEA was kept in dry/humidified conditions using various gases at room temperature for 30 days.

3. Results and discussion

3.1. Activation

A large portion of whole stack activation is spent on the swelling of polymer membrane for the development of distinctive phase separation between hydrophobic and hydrophilic regions in Nafion membrane. While, the Pt oxide such as Pt(OH)_{ads} can be easily removed by applying the potential below 0.65 V during quite a short time. Therefore, the pre-activation process is important to increase the swelling of Nafion membrane at a high temperature in a short time. In this pre-activation process, the mobility of stiff backbones in polymer structures increased at high temperature, leading to the distinctive phase separation between hydrophobic regions of perfluoroethylene backbone and hydrophilic regions of sulfonic groups. These properties are beneficial to increase the proton conductivity in a short time.

In addition to the swelling of polymer membrane, the reduction of oxidized Pt surfaces is also important to increase the oxygen reduction reaction (ORR) on Pt surface. The reduction of oxidized Pt is directly related to applied potential. Casalogue et al. specifically showed by using XPS study and density functional theory (DFT)/Monte Carlo simulations that *i*) H₂O_(ad) is the dominant species on Pt surfaces at a potential below 0.65 V; *ii*) above 0.65 V, OH_(ad)

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