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Degradation on a PTFE/Nafion membrane electrode assembly with accelerating degradation technique

Ting-Chu Jao^a, Shih-Tsung Ke^b, Pei-Hung Chi^c, Guo-Bin Jung^{a,b,*}, Shih-Hung Chan^{a,b}

^a Department of Mechanical Engineering, Yuan Ze University, No. 135, Yuandong Rd., Zhongli City, Taoyuan County 320, Taiwan

^b Fuel Cell Center, Yuan Ze University, No. 135, Yuandong Rd., Zhongli City, Taoyuan County 320, Taiwan

^c Department of Mechanical Engineering, Lee-Ming Institute of Technology, No. 2-2, Lee Zhuan Road, Taishan Township, Taipei Country 243, Taiwan

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ABSTRACT

Cost and durability are the main issues of Proton exchange membrane fuel cells (PEMFCs) commercializing. This study uses the accelerate degradation technique to analyze the durability of low cost PTFE/Nafion membrane electrode assembly (MEA). Before the MEA degradation experiment, the MEA must be activated at 65 °C until the performance is stable. Then increase the operation temperature to 80 °C. The experimental process for MEA degradation contains three steps in one cycle. The first step is to open circuit voltage (OCV) for 30 s under R.H. 100%. Then, set 0.6V for 150 s under R.H. 100%. The final step is to set 0.6V for 120 s under R.H. 0%. These three steps take around 5 min to complete. This MEA degradation experiment process includes the OCV, potential cycles, and R.H. cycles. This study uses the polarization curve, AC impedance, cyclic voltammetry (CV), linear sweep voltammetry (LSV), equations and equivalent circuit to analyze state of the MEA. At less than 160 experiment cycles, the result show that catalyst degradation is the main reason for the decay of MEA. After 280 cycles, the MEA begins to exhibit creep deformation due to the R.H. cycle. Electrochemical surface area and high frequency resistance can be used to estimate the degree of MEA degradation approximately.

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1. Introduction

Nafion membrane (DuPont Co.) is widely used in PEMFCs due to Nafion owns low resistance, excellent thermal stability and chemical stability. The high cost of the MEA is one of the most important factors for retarding the commercialization of PEMFC. Many studies have tried to develop low cost composite membranes. Recent research reports demonstrated that composite membranes can be prepared by impregnation of a low cost sub- μm porous support material, such as polytetrafluoroethylene (PTFE) membranes with a Nafion solution [1–6].

In virtue of the high mechanical strength of PTFE, inserting porous PTFE film into Nafion membrane allows it to reduce membrane thickness (the thickness of composite membranes, prepared in our lab is about 15–25 μm , and it can be roughly controlled by fabrication conditions, the thicknesses of Nafion-211, Nafion-212, and Nafion-117 membrane are about 25, 50, and 175 μm , respectively). Owing to the lower thickness of PTFE/Nafion (PN) composite membranes, the MEA have a lower proton resistance. Thus PN composite membranes have chance to have better performance in PEMFCs. Some studies have been reported that PN composite membranes

* Corresponding author. Department of Mechanical Engineering, Yuan Ze University, No. 135, Yuandong Rd., Zhongli City, Taoyuan County 320, Taiwan. Tel.: +886 3 4638800x2469; fax: +886 3 455 5574.

E-mail address: guobin@saturn.yzu.edu.tw (G.-B. Jung).

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have similar PEMFC performance to Du Pont Nafion-112 and better PEMFC performance than Nafion-115 and Nafion-117 [2–5]. The advantages of PN composite membranes are not only low cost and low thickness, but also good mechanical strength in both swollen and un-swollen state, good thermo-stability, and low dimension change ratio.

Tang et al. manifested that PN composite membrane can be kept in a stable condition for more than 5000 cycles, and it is about 40% higher than the pure Nafion membrane (about 3500 cycles.) [7]. There are several factors that affect the durability of PEMFCs [8–12]. Tang et al. did not test the durability of PN MEA operated in fuel cell discharge mode, and that meant the catalyst layer degradation was not being concerned. Electrocatalysts play an important role in improving durability and lowering the cost of the PEMFCs [13]. Many studies have shown that none recoverable deterioration of PEMFC performance is primarily attributed to degradation of Pt based catalysts and PEM in membrane electrode assembly (MEA) [14–19].

There are different accelerating degradation techniques that being discussed in the durability test field which is as below: Huang et al. demonstrated the R.H. cycling reduces the strain-to-failure of MEA by ex-situ experimental [20]. Also, the mechanical behavior of fuel cell membrane under R.H. cycles had been simulated [21]. Singaram diagnosed the MEA failure modes under R.H. cycling [22]. F. Rong used finite element method to simulate the microstructure change in the catalyst layers by load cycling [23]. Mathias suggested the use of the material characterization tests described here as screening tools for automotive fuel cell materials development: (i) membrane conductivity vs. R.H. and water uptake, (ii) membrane RH-cycling test, (iii) catalyst voltage cycling test, and (iv) support corrosion test at 1.2 V [24].

To sum up, durability and cost are key issues for commercialization of PEMFCs. Normally, a steady-state lifetime test can assess the lifetime of a fuel cell. However this method is impractical for durability test because it is time-consuming and costly. This study uses the accelerate degradation technique to show the durability of PN membrane electrode assembly.

2. Experiment

2.1. Preparation of PTFE/Nafion membrane electrode assembly and cell specifications

The porous PTFE membrane with a thickness of $18 \pm 3 \mu\text{m}$ pore sizes of $0.5 \pm 0.1 \mu\text{m}$, and a porosity of $52 \pm 5\%$ was used as a supporting material or the composite membrane. And the Nafion solution was 5wt% of 1100EW Nafion diluted in a mixture solvent which contains water, isopropyl alcohol, methanol, and unspecified ethers. The procedure for the preparation of PN composite membranes was described in previous paper [6]. The catalyst ink contains catalyst (HiSPEC 4310, 39.6wt% Pt/C, Johnson Matthey), 5wt% Nafion solution (DuPont Co.) and extra solvent isopropyl alcohol. The weight ratio of Pt loading to solid Nafion was maintained at 2:1.

The MEA was fabricated by catalyst coated membrane (CCM) method. The catalyst ink was ultrasonic treated for

30 min before it applied onto the PN membrane by spray gun. The anode and cathode Pt loading was 0.5 mg Pt/cm^2 . The CCM is sandwiched between two gas diffusion layers (SGL, 10BC) without hot press. The active area of MEA was 25 cm^2 .

In this study, a homemade cell was utilized, and the homemade cell had three components which are insulating plate, collecting plate and flow field plate. The materials of the components were glass fiber, gold coated brass and graphite, respectively. The channel depth, channel width, and rib width of the serpentine flow field plate were all 1 mm.

2.2. Experimental procedures, parameters, and apparatus

The operation conditions of the activation process were as follows: (i) cell temperature at 65°C ; (ii) hydrogen flow rates 500 sccm and air flow rates 1250 sccm, both with R.H. 100%. The discharge voltage was kept at 0.4 V for 30 min. Performance tests were carried out to ensure that specimen was successfully activated. The criterion of stability was defined as follows: The change rate of current density at 0.6 V was measured and recorded; the change rate was defined in Eq. (1). The activation process was considered to be complete when the change rate of any two consecutive events was within $\pm 5\%$. Once the specimen condition was stable, the operation temperature must be raised to 80°C gradually to accelerate the degradation process. Fig. 1 demonstrates the time history of the change rate.

$$\text{Change rate} = \frac{i_{0.6\text{V},n} - i_{0.6\text{V},n-1}}{i_{0.6\text{V},n-1}} \times 100\% \quad (1)$$

The three steps of an accelerated degradation cycle were:

- Step 1: maintain OCV at R.H. 100%;
- Step 2: discharge at 0.6 V and R.H. 100%;
- Step 3: discharge at 0.6 V and R.H. 0% (by pass).

Step 1 represented the aging mode at high temperature and humidity. Step 2 was the discharge aging mode at high temperature and humidity, and Step 3 represented the

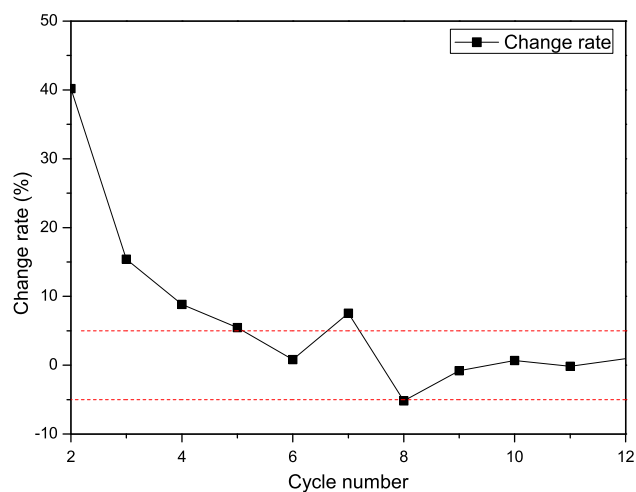


Fig. 1 – Performance change against activity cycle.

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