

## Water permeability study of nafion<sup>®</sup> 117 membrane after undergoing hot press

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### Abstract

Influence of various hot press treatment on Electrode Assembly (MEA) fabrication was investigated. The correlation between press and water permeability through both side of electrode was observed also. The hot press with temperature at 130°C was found to be more relevant to produce a more pore sizes in the catalyst layer and supporting higher surface area. The hot press temperature of 130°C was observed to develop a better pores spaces. The MEA produced was analyzed for water flux test and porosity of membrane at various of temperature was investigated also. The balance between the hot press temperature and the pressure will allow the right amount of water to exit the electrode thus making the electrode more resistant, with better efficiently and long lasting.

*Keywords* : water permeability, humidifier, hot press, PEM fuel cell.

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### 1. Introduction

Membrane characterization in MEA is necessary to mass transport of hydrogen to the cathode to form water. The potential of membrane electrode assembly for PEMFC indicated that a membrane treatment and untreated. The Nafion<sup>®</sup> 117 Membrane in MEA is called “Proton Exchange Membrane”. It should be noted that the electrolyte must only allow H<sup>+</sup> ions pass through it. Certain polymer's can also be made to contain mobile H<sup>+</sup> ions.

The conductivity behavior of Nafion 117 membranes is so almost importance for the performance of proton exchange membrane fuel cell (PEMFC), where Nafion 117 can be used as electrolyte and highly permeable to water. This property is caused sulfonic acid groups in Nafion 117. The sulfonic acid groups have function for absorbs water and lead to very rapid transfer of water through Nafion is shown in fig 1<sup>[3]</sup>.

In the literature, mechanism of this water transport is usually identified as diffusion. However, in this approaches the influence of the membrane microstructure on its transport takes place in the hydrophilic pores of the membrane, which are surrounded by

the hydrophobic polymer backbone forming a sponge-like structure. Water is not the solvent in the hydrophilic pores but the solvent. Therefore, diffusion describing transport of the solutant is obviously not the adequate transport mechanism for water transport. In contrast, Frank Meier and Gerhart Eigenberger assumes that the back transport of water in the H<sub>2</sub>-PEC is caused by a gradient of the capillary pressure inside the membrane, which is caused by the gradient in the membrane water content [6].

An interesting attempt to investigate the conduction phenomena in Nafion was performed by Uosaki et. al. (1990), who investigated Nafion 117 membranes by means of impedance spectroscopy and differential scanning calorimetry between 180 K and room temperature. They showed that conductivity of Nafion depends on the water structure in the membrane, and that this can be influenced by the cation exchange.

In this paper presented here concern the determination of the relationship of the hot pressing at a temperature 130°C and a pressure of 35 kgf/cm<sup>2</sup> for 180 s on the water permeability.

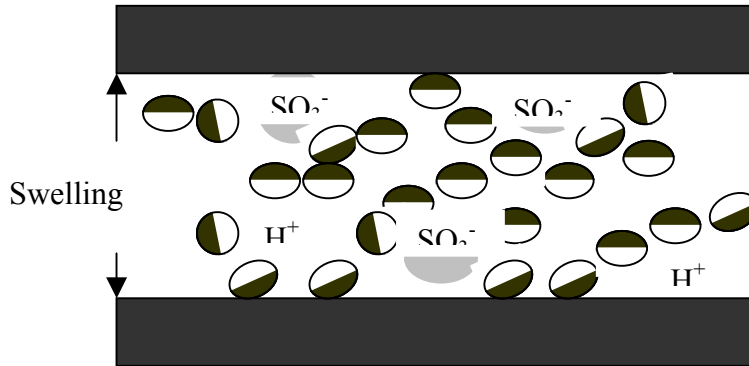


Figure 1. Distribution of proton in Nafion 117 membrane

## 2. Theory

Concept of water permeability is very simple transfer of water through the Nafion, which is measured as function of the applied pressure. At a certain minimum pressure the largest pores become permeable, while the smaller pores still remain impermeable. This minimum pressure depends mainly on the type of membrane material present (contact angle), type of permeate (surface tension) and pore size. The flux water is proportional to the increase in applied pressure according to Hagen-Poiseuille:

$$N = \left( \frac{\epsilon r^2}{8\eta\tau\delta x} \right) \Delta P_{eff} \dots\dots\dots 1$$

Here N is the (water) flux through the membrane at a driving force  $\Delta P/\delta x$ , with  $\Delta P$  being the pressure difference and  $\delta x$  the membrane thickness. The proportionality factor

contains the pore radius  $r$ , the liquid viscosity  $\eta$ , the porosity of the membrane  $\varepsilon$  ( $=n\pi r^2$ ) and the tortuosity factor  $\tau$ .

According to Marcel Mulder, 1991, the smaller pore diameters require a high pressure to wet the membrane. If the membrane becomes wetted and permeable, and thereafter the flux increases linearly with increasing pressure. Permeability coefficient was obtained from linear regression of curve.

### **3. Experimental**

#### *3.1. The Nafion 117 membrane treatment*

Nafion 117 membrane was first cleaned with distilled water in a water bath at 80-90° C, followed by heating in 5% H<sub>2</sub>O<sub>2</sub> for 1 hour at 70-80° C to remove organic impurities. It was then heated in 0.5 M H<sub>2</sub>SO<sub>4</sub> for 1 hour at 70-80° C. The H<sub>2</sub>SO<sub>4</sub> was removed by repeated washing in boil water. The membrane was stored in the dark overnight in distilled water before assembling with the electrodes.

#### *3.2. Membrane and electrode assembly (MEA)*

Activated carbon, Iso propyl alcohol (IPA), water, Nafion solution and polytetrafluoroethylene (PTFE) were mixed using magnetic stirrer for a 10 minutes as a carbon ink. The viscous mixture was sprayed on the carbon cloth and then dried for one day (Heo et. al., 2002). It is called the Gas diffusion layer, next hot pressed using hot press machine (Hung Ta Instrument Co. LTD). The sheet placed in the stainless steel frame. Each heating plate was drilled to accept an electric cartridge heater and thermocouple. A temperature controller was connected to the heater and thermocouple on each heating plate. Setting the temperature at 130° C, pressure at 35 kgf/cm<sup>2</sup> and for long 180 s. The GDL was then cooled at room temperature for 30 minutes.

Then catalyst layer was prepared, that is carbon-support platinum, Nafion solution and iso-propyl alcohol were mixed and coated on to the diffusion layer and the latter was dried in an oven at 80° C to remove water and iso-propyl alcohol. Nafion 117 membrane is sandwiched between two electrodes using hot press similar to that of the GDL. The MEA fabrication process can see in the fig 1.

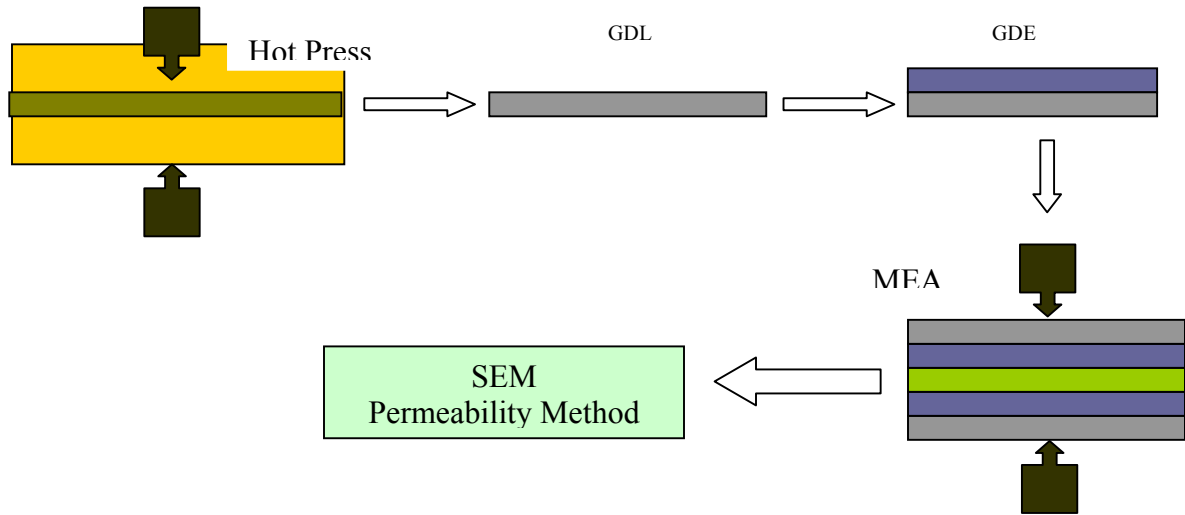


Figure 2. Diagram flow MEA Fabrication

### 3.3. Porosity

Vinod Kumar Gupta used Mizutani and Nishimura (1847) methods for the estimation of porosity of the membrane, which is calculated from water content data by using the following formula:

$$\varepsilon = \text{water content} / A.L.\rho_w \dots\dots\dots 2$$

Where, A = Area of the membrane, L = thickness and  $\rho_w$  = Density of water

### 3.4. Water content

Membrane after treatment and the adhering liquid was wiped off with tissue paper. Then the membrane was weighed and dried at the temperature 130° C to a constant weight. The difference in the two weightings divided by the weight of the wet membrane was taken as the water content. <sup>[9]</sup>

Characteristics of MEA and Nafion 117 membrane were analyzed by Scanning Electron Microscopy (SEM) and determine parameter of water permeability used linier regression.

#### 4. Results and discussion

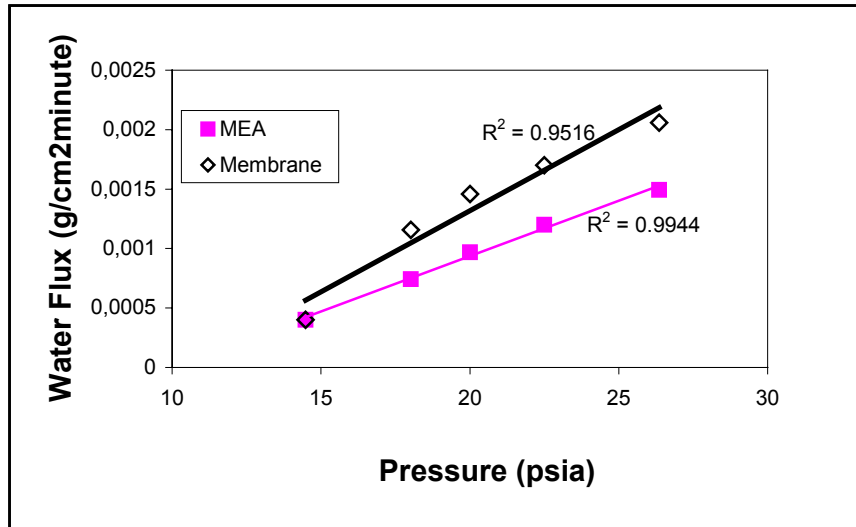


Figure3. Water Flux vs Pressure

Permeability coefficient of nafion 117 membrane was obtained from linear regression of curve. This curve looks similar to the curve of Hagen-Poiseuille equation. The idealised flux version pressure curve is shown in fig 3. However, synthetic micro filtration and ultra filtration membranes do generally not posses a uniform pore size, and hence breakthrough curves of the type shown in fig 3 will not be observed. At a pressure below  $P_{min}$  the membrane is impermeable. At  $P_{min}$  the largest pore become permeable, and as the pressure increases smaller and pores become permeable when the pressure increased further the flux increases linearly with pressure (fig 3). From the regression linier of both the curve is

$$Y = 0.0002x - 0.0014 \dots\dots\dots 3$$

$$N = 2.10^{-4} \Delta P_{eff} - 0.0014$$

$$Y = 0.00009x - 0.0009 \dots\dots\dots 4$$

$$N = 9.10^{-5} \Delta P_{eff} - 0.0009$$

Permeability coefficient of Nafion 117 membrane before hot press is  $2.10^{-4}$  g/cm<sup>2</sup>/psia and MEA is  $9.10^{-5}$  g/cm<sup>2</sup>/psia. Decreasing permeability coefficient by up to  $9.10^{-5}$  g/cm<sup>2</sup>/psia after assembling Nafion 117 membrane between electrodes given in equation 3 or fig 3. There is decreasing of permeability coefficient of membrane after hot press caused temperature and pressure treatment that have been done at temperature of 130° C and pressure of 35 kgf/cm<sup>2</sup> for 180s. Hot pressing method can influence microstructure of Nafion 117 membrane shown in fig 4. By treating temperature will to removal the water content. The water content influence microstructure of membrane such the hydrophilic pores of the Nafion 117 membrane. Decreasing of water content regions

around the cluster of sulphonated side chains can lead to the absorption of smaller quantities of water influenced to proton mobility in Nafion 117 membrane. The pressure very influence pore size of Nafion 117 membrane. By pressure treatment of hot press the Gas diffusion layer will impregnate a part of membrane pore. The smaller pore in membrane become impermeable that will cause the current of MEA reducing directly from 522 mA/cm<sup>2</sup> up to 10 mA/cm<sup>2</sup> in the time 30 minutes than the MEA commercial has current is 643 mA/cm<sup>2</sup> and reducing after 30 minutes up to 300 mA/cm<sup>2</sup>.

Table 1  
Water content and porosity of Nafion 117 membrane at temperature 130° C.

Membrane	Membrane Weigh (g)	Constant weight (g)	Thickness (cm)	Area (cm <sup>2</sup> )	Water content per gram of wet membrane G(H <sub>2</sub> O)/g(w.mem) ;g/g	Porosity
Nafion 117	0.2513.	0.2210	0.028	8.41	0.1152	0.4892

From the table 1, can be seen the temperature influence water content in membrane. The water removal from membrane obtains porosity 0.4892 at temperature of 130°C. The porosity of membrane depends of remove of water content and temperature.

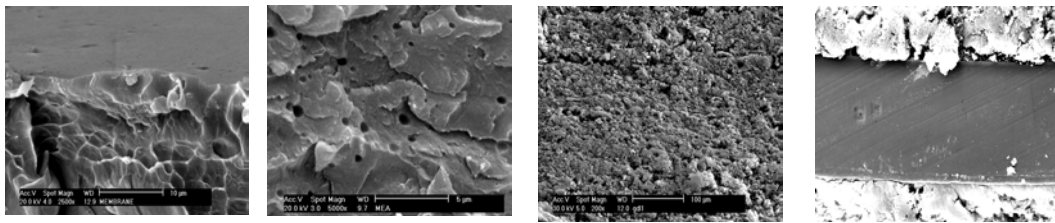


Fig 4. Scanning electron microscopy of a cross-section of Nafion 117 membrane and MEA (a) Nafion 117 membrane before hot press method (b) Nafion 117 membrane after hot press (c) diffusion layer after hot press and (d) Membrane Electrode Assembly

By assembling the electrodes on the both side of Nafion 117 membrane impregnate the pore of membrane, which influence the permeability of membrane. According to SEM result shows the different of structure of membrane before hot press and after assembling membrane between two electrodes.

## 5. Conclusions

Hot press method at temperature of 130°C and pressure of 35 kgf/cm<sup>2</sup> for 180s influenced of the membrane microstructure such as pore size, porosity and lead to transfer of water through the Nafion. The membrane microstructure influence the water permeability of Nafion 117 membranes. At a certain minimum pressure the largest pores become permeable, while the smaller pores still remain impermeable. The temperature of hot press in MEA fabrication influence to remove water in membrane.

## 6. References

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