INTERNATIONAL JOURNAL OF HYDROGEN ENERGY 37 (2012) 12805–12812

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Ultra thin gas diffusion layer development for PEMFC

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ARTICLE INFO

Article history:
Received 14 December 2011
Received in revised form
22 May 2012
Accepted 23 May 2012
Available online 23 June 2012

Keywords:
Gas diffusion layer
Carbon fiber paper
Proton exchange membrane fuel cell
Micro porous layer

ABSTRACT

This research studies an ultra-thin carbon fiber paper fabrication process for proton exchange membrane fuel cells (PEMFCs). Polyacrylonitrile (PAN) based carbon fibers 6 mm long were dispersed and formed at aerial weights of 15 and 20 g/m^2 using a slurry molding machine. Polyscrylamide (PAM) and *polyvinyl alcohol* (PVA) dispersal agent solutions for fiber binding were added to evenly distribute the carbon fibers and increase the paper mechanical strength. The carbon fiber papers were dried after resin impregnation using a convective oven at 120 °C temperature for 10 min. The hot press machine was heated to 160 °C temperature and the workpieces were pressed for 5 min. Graphitization completed the gas diffusion substrate (GDS) process. GDL involves immersing the paper in a 5% polytetrafluoroethylene (PTFE) solution, coating the paper with a micro porous layer (MPL). This study shows the proposed ultra-thin GDL fabrication method is suitable for PEMFC applications and exhibits feasible functionality for fuel cells.

1. Introduction

The fuel cell is a power generation module that converts the chemical energy stored in the fuels and oxidants directly into power using a non-burning electrochemical reaction. Among the various fuel cells, the proton exchange membrane fuel cell (PEMFC) possesses advantages such as non-polluting, non-erosion, high power, high energy conversion efficiency, fast activation and low working temperature. It is suitable for use as a mobile energy source and power supply for electrical vehicles. The fuel cell can be applied in the following three fields, transport, stationary and portable fields [1].

A gas diffusion layer is an important component of the PEMFC. It supports the catalyst layer and provides important mass transfer channels for reaction gas and water management, as well as electrical transfers. The gas diffusion layer uses the carbon fiber paper as the fuel cell electrode. The gas diffusion layer has a uniform cellular construction, high mechanical strength and stable size. Carbon fiber paper, it has the features of good conductivity, chemical stability and thermal stability. The porous construction in the gas diffusion layer has an important influence on fuel cell operation. Its primary function is to provide transfer channels for the H_2 and O_2 in the electrochemical reaction, ensure that the cell can output stable electrical current and remove the liquid water generated in the reaction, prevent system flooding [2].

Jordan et al. [3] discussed the influence of different gas diffusion layer physical parameters on PEMFC performance using theoretical analysis. They found that the gas diffusion layer thickness and film characteristics were both related to PEMFC performance. The operating temperature and gas humidity must be taken into account as well. Chua et al. [4] found that the gas diffusion layer porosity influenced cell performance and could cause non-uniform gas distribution. A

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0360-3199/$ – see front matter
http://dx.doi.org/10.1016/j.ijhydene.2012.05.110

change in gas diffusion layer porosity would affect oxygen transport. Large pores can obtain good oxygen transport, resulting in high current density and good cell performance. However, it also consumes a large amount of oxygen. Small pores may cause flooding and lower the cell performance. A change in porosity would not influence the cell performance in low density cased. In high current density cases, changes in porosity are influenced mainly by the mass transfer phenomenon, which in turn affects cell performance. Williams et al. [5] fabricated gas diffusion layers using five different kinds of commercial-use gas diffusion layers for comparison. The results showed that the gas permeation rate was related to the current density and increased with the gas permeability. The gas diffusion layer generates capillary force because of the small pores. The capillary force influence results in high gas permeability. Under gas diffusion layer saturation circumstances oxygen transfer limits and flooding can be ignored. Large pores diffuse and transmit gas more effectively.

Lufrano et al. [6] conducted PTFE impregnation in carbon fiber paper and measured its performance using the PTFE with a concentration from 10% to 60%. The study results showed that PTFE with a concentration of 20% obtains good cell performance. Prasanna et al. [7] conducted researches on different gas diffusion layer thicknesses impregnated using PTFE with different hydrophobic treatment concentrations. The study results showed that a small thickness would result in great contact resistance and mass transfer loss, causing the flooding phenomenon. A large thickness produced high ohmic resistance, mass transfer resistance and activation overpotential. As the thickness increased, the porosity would decrease. Good performance was achieved at a thickness of 175 μm. Park et al. [8] added different PTFE concentrations to the micro porous layer. They found that high PTFE concentration would easily block water from passing through the gas diffusion layer, reducing cell performance. A gas diffusion layer added with a micro porous layer had smaller pores. Adding PTFE at content from 10% to 40% to the micro porous layer showed that 20% PTFE produced good cell performance.

In the previous gas diffusion layer composition studies, post-processing was conducted using commercial carbon fiber papers. These studies were focused on the diffusion layer thickness, porosity, gas permeability and electrical resistance. PEMFC generates water during the electrochemical reaction. The flooding phenomenon in the cell has great influence on cell performance. Water management in the gas diffusion layer is therefore critical. To reduce the flooding phenomenon hydrophobic treatment is conducted on the carbon fiber paper and micro porous layer. There are many factors that influence gas diffusion layer performance, all of which are correlated with each other. The previous studies mentioned that a thin gas diffusion layer produced poor performance. Carbon fiber paper substrate processing is also a critical parameter. Mathias et al presented a good review and description for GDL materials and characterization [9]. Carbon-fiber products, processing and diffusion media production were recently reviewed by Park et al. [10]. One type of carbon fiber paper was prepared from oxidized fiber felt by Liu et al. [11]. The small yard weight can produce a thin carbon paper. The small yard felt produced using a needle punch created the fiber distribution uniformity problem. This defect can be observed by the naked eye. The thickness of commercially available carbon fiber papers is greater than 190 μm (Toray Carbon Paper TGP-H-120) or 108 μm at 140 N/cm^2 (Ballard Power Materials, Carbon Paper – AvCarb P50). These carbon papers were made using a wet paper processing machine with certain basic weights. The demand for a thinner GDL with good cell performance is pressing. This study develops an ultra-thin gas diffusion layer with feasible functionality verified in fuel cell applications.

2. Basic theories

In their fiber paper-making dispersion study, Kerekes et al. [12] improved the fiber flocculation in water phenomenon using a suspension, and proposed crowding factor concept, defined as the number of fibers allowed in a specific volume, represented as N. The equation is represented below:

$$N = \frac{2}{3} C_v \left(\frac{L}{d}\right)^2 \quad (1)$$

Where, C_v is the dispersant concentration in volume (%), L is the fiber length (μm) , d is the fiber diameter (μm). The crowding factor is calculated using Equation (1). A single fiber will not dilate or divide into smaller fibers. The diameter of a single fiber will therefore not change. The fiber diameter is a constant. The fiber length is the variable influencing the crowding factor under the same fiber length.

According to Eq. (1), when N is small, it indicates that there are few fibers in the space and these fibers can move freely. As a result, the fibers have little contact with each other, the length/diameter ratio is small and the fiber distribution is quite uniform. As the fiber length increases, N will increase accordingly. As a result, there are more chances for longer periods of fiber contact. As the fiber length increases, the length/diameter ratio becomes large, so the fiber distribution becomes rather non-uniform. If N is too large, the fibers become restricted by the limited space and the fiber fluidity is decreased. The increased contact between the fibers easily causes the fiber flocculation phenomenon.

3. Experimental study

This study produced an ultra-thin gas diffusion layer applicable to fuel cells that is expected to reduce the gas diffusion layer thickness and retain the same functions as the current market gas diffusion layer. The experimental steps in this study are divided into the fiber paper fabrication and post-processing.

3.1. *Fabrication of carbon fiber paper*

This study adopted wet paper-making technology to fabricate the carbon fiber paper. PAN carbon fiber manufactured by the Japanese Toho Corporation in lengths of 3, 6 and 12 mm was selected as the raw material. PAM dispersant at four concentrations: 0.01, 0.05, 0.13 and 0.25%, was mixed with the carbon fiber and at proportion of 0.025, 0.05 and 0.1%. PVA fibers were added to at 10% of the carbon fiber weight to increase the paper strength. Carbon fiber dispersion was conducted as the final step.

3.2. *Carbon fiber paper post-processing*

Phenolic resin impregnation, carbonization, graphitization, hydrophobic treatment and micro porous layer coating were conducted as carbon fiber paper post-processing steps. High fludic PF650 phenolic resin solution was used to fully and uniformly impregnate the raw fiber paper. Methyl alcohol was used to dilute the phenolic resin solution at concentrations of 16, 25 and 33%. The carbon fiber paper was baked in an oven for 10 min at the temperature of 120 °C. The phenolic resin impregnation ratio can be shown using Equation (2):

$$\text{Impregnation rate}(100\%) = \text{Weight after impregnation} - \text{Weight before impregnation}/\text{Weight after impregnation} \times 100\% \quad (2)$$

The produced raw fiber paper thickness is between 135 and 210 μm, so hot pressing is necessary to reduce the thickness to 100 μm or less. To make the resin sufficiently fluid during the hot pressing process and distributed it uniformly through the carbon fiber, the hot press machine temperature needs to be 160 °C. The pressure was set at 80 kg/cm^2 with 5 min hot press time.

After the hot pressing process, the mechanical strength, porosity and density of the carbon fiber paper are all increased. Carbonization and graphitization are conducted next to attain the proper carbon fiber paper electrical resistivity. Phenolic resin in the carbon fiber paper can remove non-carbon elements during high-temperature carbonization and produce carbon during the sintering process. The sintering temperature is 850 °C for 1 h. To reduce the through-plane fiber paper resistance and improve its chemical stability, a graphitization process must be conducted under high temperature. The sintering temperature is increased to 1750 °C for the graphitization process, lasting for 30 min.

To avoid the flooding phenomenon in the gas diffusion layer, the carbon fiber paper receives hydrophobic treatment. A 60% PTEE solution from Sigma–Aldrich Corporation is used for the hydrophobic treatment. The PTFE solution is stirred into pure water to produce the hydrophobic agent at a concentration of 5%. This solution is added onto the carbon fiber paper for a 10-min hydrophobic treatment. Following the solution treatment, the carbon fiber paper is baked on a hot plate for 10 min at a temperature of 200 °C and sintered for 20 min at 360 °C.

After the above processes, the carbon fiber paper receives micro porous layer fabrication processing. In the micro porous layer, carbon black was mixed with PTFE dispersed in a methyl alcohol solution. The solution contained 1.6 g of Vulcan XC-72R carbon black added into PTFE at 10% concentration mixed with 100 ml of methyl alcohol. The weight ratio of carbon black/PTFE in this solution is 70/30. This mixture is coated onto one side of the carbon fiber paper. Because the paper has an ultra-thin, porous structure, the MPL slurry penetrates into the bulk GDL. The coated paper is then baked on a hot plate for 10 min at a temperature of 200 °C, followed by sintering for 10 min at a temperature of 360 °C.

Commercially available CCM (catalyst coated membrane), from Pearl Hydrogen Technology, was used to make MEA. A fuel cell test station (PCFD PD50, APFCT) was employed to obtain the MEA polarization curve. The operating conditions are shown as follows.

(1) Stoichiometry: H_2/Air = 1.5/2.5
(2) Cell temperature: 60 °C
(3) Humidification temperature: 65 °C for both anode and cathode
(4) Scanning range and rate: 0.95 V ~ 0.3 V, 50 mV/min

4. Results and discussion

This section examines the carbon fiber length, dispersant concentration, dispersion theory verification and measures the carbon fiber paper functions. This study dispersed the carbon fiber with three different lengths 3, 6 and 12 mm. The experimental results show that the 3 and 6 mm lengths obtained good dispersion while the 12 mm length obtained rather bad results. That is mainly because the carbon fiber does not have bonding ability, allowing the long carbon fibers to wind together easily during stirring, resulting in flocculation as shown in Fig. 1. Non-uniform sediment is produced as shown in Fig. 2. The 6 mm carbon fiber produces better mechanical strength than the 3 mm carbon fiber. Therefore, the carbon fiber length not only affects the dispersion and uniformity of carbon fiber, it also affects the fiber paper gas permeability. Therefore, suitable carbon fiber paper must be fabricated based on selecting suitable carbon fiber length to obtain appropriate permeability, strength and uniformity.

Fig. 1 – Carbon fiber paper with flocculation.

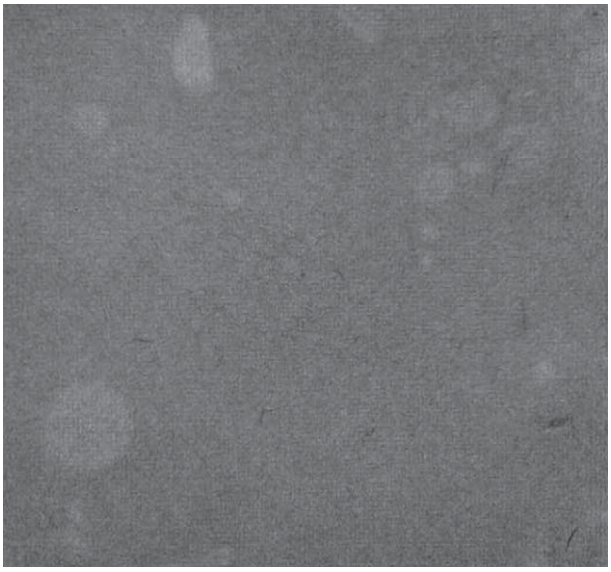

Fig. 2 – Carbon fiber paper with non-uniform sediments.

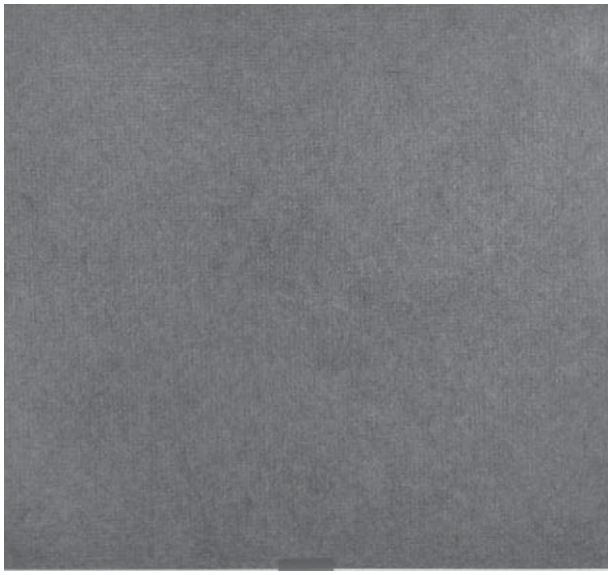

Fig. 4 – Carbon fiber paper with good dispersion.

This study selected 3 and 6 mm carbon fiber to fabricate the carbon fiber paper.

The experimental results show that the 3 mm carbon fiber can obtain good dispersion effect at all dispersant concentrations. However, at high dispersant concentrations the carbon stirring period is quite long. Therefore, low dispersant concentration should be used to disperse the 3 mm carbon fiber. When the carbon fiber is 6 mm long, 0.05 ~ 0.13% dispersant concentration can obtain good dispersion effects. Excessive dispersant concentration, 0.25% will produce holes during the paper-making process, as shown in Fig. 3. Under the appropriate dispersant concentration, the carbon fiber paper can get good dispersion effects, as shown in Fig. 4. The carbon fiber paper fabrication process can be helped by adding dispersant. Different dispersant concentrations will change the flow of the carbon fiber suspension. The adhesiveness of

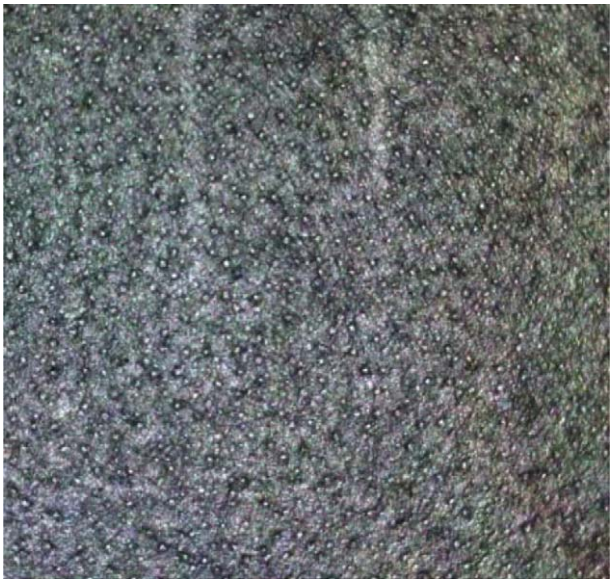

Fig. 3 – Carbon fiber paper with holes.

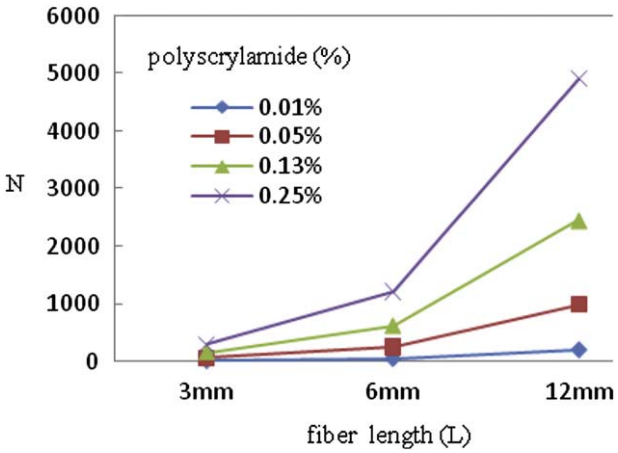

Fig. 5 – Correlation diagram of N in the dispersion theory.

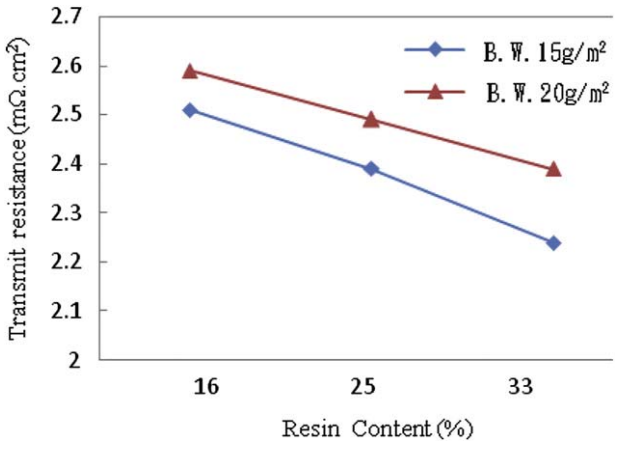

Fig. 6 – Through-plane resistance for paper impregnated with different resin concentrations.

Table 1 – Weight measured using different basic weights and impregnating into different concentrations.

Sample	Basic weight (g/m^2)	Resin content (%)	Carbon paper weight (g)	Phenolic resin weight (g)	Hot press weight (g)	Graphitize weight (g)	Impregnation ratio (wt%)
1	15	16	1.25	4.31	3.82	2.49	70
2	15	25	1.25	4.7	4.27	2.67	73
3	15	33	1.25	5.98	5.09	3.19	79
4	20	16	1.69	5.98	5.5	3.64	71
5	20	25	1.69	6.64	5.94	3.79	74
6	20	33	1.69	7.45	6.2	4.03	77

Table 2 – Thickness measured using different basic weights and impregnating with different concentrations.

Sample	Basic weight (g/m^2)	Resin content (%)	Carbon paper thickness (μm)	Hot press thickness (μm)	Graphitize thickness (μm)	Contract ratio (%)
1	15	16	145	68	48	29
2	15	25	145	70	51	27
3	15	33	145	75	52	30
4	20	16	200	80	58	27
5	20	25	200	85	59	30
6	20	33	200	87	62	28

the dispersant can indeed improve the fluidity of the carbon fiber in the water and slow down the sedimentation and flocculation.

This study verified the dispersion theory using Equation (1). Variables in the equation were substituted to calculate N. Four different dispersant concentrations 0.01, 0.05, 0.13 and 0.25% are taken as variable C_v, 3, 6 and 12 mm as variable L and diameter 7 μm as variable d. N is calculated as shown in Fig. 5. Fig. 6 shows that under different carbon fiber lengths, N changes with the dispersant proportion. Three and 6 mm carbon fibers can obtain better dispersion effect than 12 mm. According to the calculation results, the minimum N is 12, while the maximum N is 612. Therefore, when N is $612 \geqq N \geqq 12$, the carbon fiber paper exhibits good dispersion effect.

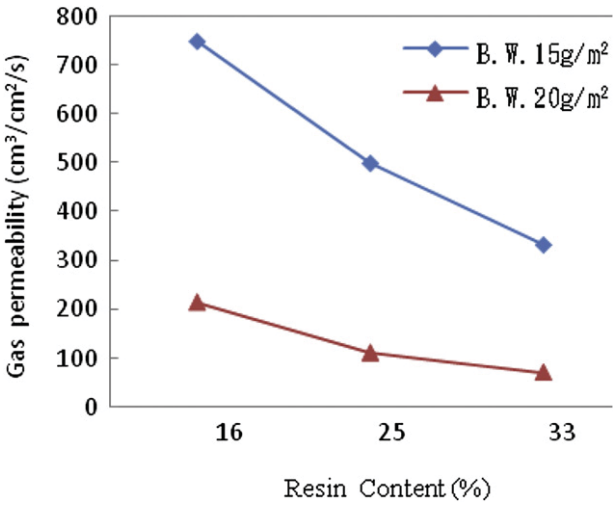

Fig. 7 – Gas permeability for paper impregnated with different resin concentrations.

This study measured the physical properties of the fabricated carbon fiber paper, including the thickness, weight, gas permeability, through-plane resistance, tensile strength, surface morphology and GDL performance test. The weight of the carbon fiber paper was measured after each process, as shown in Table 1. The phenolic resin impregnation ratio was obtained by measuring the weight after phenolic resin impregnation. By observing the weight change after hot pressing, the weight was found slightly reduced after hot pressing. This is because the methyl alcohol in the phenolic resin is volatile and the surplus phenolic resin was squeezed out during the hot-press process. The weight loss after graphitization is because the non-carbon elements were sintered under high temperature. As a result, the residual carbon on the phenolic resin contracted. When raw fiber paper with a basic weight of 20 g/m^2 was impregnated with phenolic resin at concentration of 16, 25 and 33%, the impregnation ratios

Table 3 – Tensile strength measured using different basic weights and impregnating into different concentrations.

Sample	Basic weight (g/m^2)	Resin content (%)	Tensile strength (N/cm)
1	15	16	32–39
2	15	25	32–39
3	15	33	32–39
4	20	16	35–41
5	20	25	35–41
6	20	33	35–41

Table 4 – Illustrated bare GDL material properties.

Properties	Thickness (μm)	Basic weight (g/m^2)	Tensile strength (N/cm)	Gas permeability ($cm^3/cm^2/s$)	Through-plane resistance ($m\Omega\ cm^2$)
Commercial (CeTech)	180	45	24	100	5.8
Sample 1	48	15	32	750	2.51
Sample 4	58	20	35	214	2.59

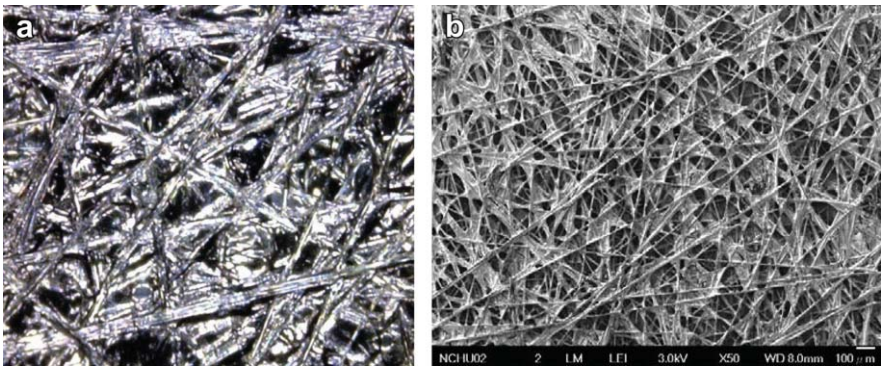

Fig. 8 – Surface morphology of carbon fiber paper impregnated with phenolic resin, (a) measured by optical microscope 200×, (b) SEM micrograph.

were 70, 73 and 79%, respectively. When carbon fiber paper with a basic weight of 20 g/m^2 was impregnated with phenolic resin at concentration of 16, 25 and 33%, the impregnation ratios were 71, 74 and 77%, respectively.

The major factors affecting the carbon fiber thickness include basic weight of carbon fiber paper, phenolic resin concentration, and hot-press pressure, as shown in Table 2. Using carbon fiber paper with a basic weight of 15 and 20 g/m^2 impregnated with phenolic resin at concentration of 16, 25 and 33%, the raw fiber paper thicknesses were 145 and 200 μm, respectively. After the hot-pressing process, the thicknesses were 68, 70, 75 and 80, 85, 87 μm, respectively. Through the carbonization and graphitization processes, the thicknesses were reduced to 48, 51, 52 μm and 58, 59, 62 μm, respectively. Compared with the thickness before the hot-pressing process, the thicknesses were reduced by 20, 19, 23 μm and 22, 26, 25 μm, respectively. Calculating the phenolic resin contraction ratios, the results were 29, 27, 30% and 27, 30, 28%, respectively.

The GDL through-plane resistance test used carbon fiber paper with a basic weight of 15 and 20 g/m^2 impregnated with phenolic resin at concentrations of 16, 25 and 33%. As shown in Fig. 6, as the phenolic resin content increased, the through-plane resistance is reduced. Moreover, carbon fiber paper with a basic weight of 15 g/m^2 has low through-plane resistance. Table 2 shows that carbon fiber paper with a basic weight of 15 g/m^2 attains thinner thickness than paper with a basic weight of 20 g/m^2. Therefore, the carbon fiber paper thickness has influence on the through-plane resistance. A thinner GDL has relatively small through-plane resistance.

The carbon fiber paper exhibits high gas permeability before impregnation with phenolic resin. The carbon fiber is composed of a stack of crossing fibers. There are numerous pores between the fibers. The most common carbon fiber paper on the market is fabricated using the carbon fiber with a basic weight of 40 g/m^2. However, the thin gas diffusion layer fabricated in this study has basic weights of 15 and 20 g/m^2. The gas permeability is relatively high as shown in Fig. 7. After impregnation with phenolic resin at a concentration of 16, 25, 33%, the carbon fiber paper exhibits good gas permeability. Comparing carbon fiber papers with basic weights of 15 and 20 g/m^2, the former exhibits a better gas permeability. Carbon fiber paper impregnated with phenolic resin at low concentration exhibits the best gas permeability. Thus, the carbon

Fig. 9 – SEM diagram of coating micro porous layer.

Table 5 – Parameters for fuel cell test.

Cathode fuel	Anode fuel	Cathode temperature	Anode temperature	Cathode gas-flow rate	Anode gas-flow rate	Cell temperature	Humidity	Back pressure	Reaction area
H_2	Air	65 °C	65 °C	1.5	2.5	60 °C	90%	None	25 cm^2

fiber content and phenolic resin concentration both have great influence on the gas permeability.

The purpose of the tensile strength measurement is to identify carbon fiber paper with low basic weight. As shown in Table 3, using carbon fiber paper with basic weights of 15 and 20 g/m^2 impregnated with phenolic resin at concentrations of 16, 25 and 33% the measured tensile strengths are 32 ~ 39 N/cm and 35 ~ 41 N/cm, respectively. Both fiber papers have good mechanical strength. An illustrated table listed some bare GDL material properties is shown in Table 4. Thin GDLs have lower through-plane resistance than commercial GDL. The ultra-thin gas diffusion layer fabricated in this study through high-pressure hot-pressing forms thin carbon fiber papers. The pressed carbon fibers bond with each other tightly, giving the thin gas diffusion layer good mechanical strength.

This study used optical microscopy to observe the surface morphology of the carbon fiber paper after carbonization and graphitization sintering. As shown in Fig. 8, the carbon fiber surface is covered by residual carbon after phenolic resin sintering under high–temperature. The pores between the carbon fibers are covered by phenolic resin. The phenolic resin contracts after the carbonization and graphitization processes. As a result, the pores between the carbon fibers become larger and the gas permeability is improved. The smoothness of the micro porous layer coating is shown using SEM observation (Fig. 9). As shown in Fig. 9, the carbon fiber structure of bare GDL was still apparent after MPL coating. This indicated that MPL modified the GDL pore structure without increasing the GDL thickness.

The performance test used carbon fiber paper with basic weights of 15 and 20 g/m^2 impregnated with phenolic resin at concentration of 16, 25 and 33%, with a micro porous layer coated on one side. The fabricated carbon fiber paper was assembled into a single cell for conducting the cell performance test. The parameter settings during the performance test are shown in Table 5. The performance test results are shown in Fig. 10. When the cell voltage was 0.6 V, the maximum current density and power density were achieved using carbon fiber paper with basic weights of 15 and 20 g/m^2 impregnated with phenolic resin at 25% concentration. The maximum current densities were 1701 and 1805 mA/cm^2 , when the maximum power densities were 576 and 615 mW/cm^2. When the phenolic resin concentration was 16%, current densities of 1680 and 1611 mA/cm^2, and power densities of 569 and 564 mW/cm^2 were achieved. When the impregnated phenolic resin was concentrated at 33%, the maximum current densities of 1492 and 1355 mA/cm^2 , and maximum power densities of 521 and 471 mW/cm^2 were achieved. Therefore, it can be concluded that the phenolic resin concentration will affect carbon fiber paper performance. Using the phenolic resin at a 25% concentration, the GDL obtains the best performance. Conversely, phenolic resin impregnation concentration at 33% achieves the poorest performance.

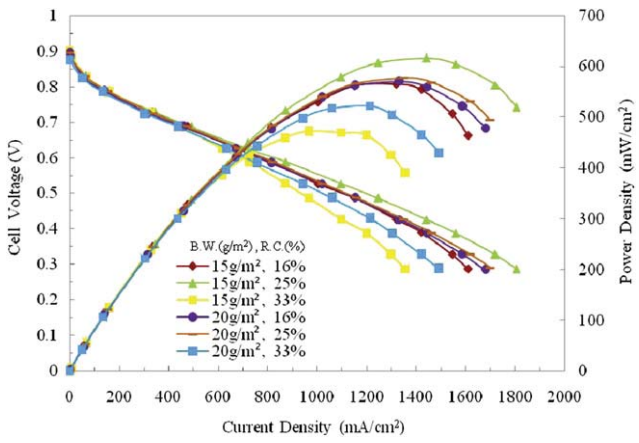

Fig. 10 – Current density-voltage and current density –power density curve.

5. Conclusions

This study proposed GDL fabrication by adjusting the material composition. A large-capacity dispersion water tank added with the appropriate amount of carbon fibers was used to produce GDL with excellent dispersion and uniformity. The carbon fiber dispersion is closely related to three carbon fiber characteristics, length, dispersant concentration and carbon fiber content. The fabricated ultra-thin gas diffusion layer possessed good mechanical strength, gas permeability and electrical resistance with low aerial weight carbon fiber, which reduces the carbon fiber material cost. This study successfully fabricated an ultra-thin GDL. According to the measurement results, the minimum thickness can reach 48 μm, 32 ~ 39 N/cm tensile strength, 2.5 mΩ-cm^2 through-plane resistance and 750 $cm^3/cm^2/s$ gas permeability. When the test cell voltage was 0.6 V, the air-hydrogen flow ratio was 1.5/2.5, and the working temperature 60 °C. The maximum current density reached 815 mA/cm^2 without back pressure. The proposed ultra-thin GDL fabrication process can be applied in PEMFCs, to reduce the fuel cell stack volume. Feasible functionality for the proposed process was verified for fuel cell applications.

Acknowledgment

This research was supported by the National Science Council (NSC-99-2221-E-005-073), Taiwan. The processing equipment provided by CeTech Co., Ltd. in Taiwan is acknowledged.

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