



Short communication

Effect of carbon fiber cloth with different structure on the performance of low temperature proton exchange membrane fuel cells

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ABSTRACT

This study uses fuel cell gas diffusion layers (GDLs) fabricated in the laboratory from carbon fiber cloth with different structure in proton exchange membrane fuel cells (PEMFCs), and investigates the relationship between the structure of the carbon fiber cloth and fuel cell performance. The paper discusses the relationship between fuel cell performance and structure of the carbon fiber cloth, and also examines the effect of the carbon fiber cloth's thickness, air permeability, surface resistivity, XRD and elemental analysis. Carbon fiber cloth is carbonized at rates of 190, 220, 250, 280, and 310 °C min⁻¹ respectively, and the resulting carbon fiber cloth is tested in cells. When the test piece area is 25 cm², the test temperature 40 °C, the gasket thickness 0.36 mm, and the carbonization rate 280 °C min⁻¹, a fuel cell using the carbon fiber cloth achieves a current density of 1968 mA cm⁻² and a maximum power density of 633 mW cm⁻² at 0.3 V.

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1. Introduction

Because of their high efficiency, high power density, low operating temperature, and low noise, proton exchange membrane fuel cells (PEMFCs) are thought to be superior to other fuel cell systems in vehicle applications [1–4]. Although gas diffusion layers (GDLs) are only one part of a fuel cell, differences in their internal microstructure can affect the performance of the cell as a whole. Carbon fiber fabric or carbon fiber papers are currently the most common materials used to make GDLs. Because carbon possesses the advantages of high conductivity and corrosion resistance, it is very well suited to the special environment inside a fuel cell. GDLs are usually produced by subjecting oxidized carbon fiber felt or oxidized carbon fiber cloth to high-temperature carbonization in a carbonizing furnace. This alters the structure of the material and increases conductivity, yielding carbon fiber paper or carbon fiber cloth after processing [5].

Past research on carbon fiber has shown that graphitization temperature has a significant effect on the microstructure and properties of the material, and conductivity increases with graphi-

tization temperature [6–7]. We choose a carbonization temperature of 1300 °C in this paper. The structure of carbon fiber gradually changes at a carbonization temperature of 1000–1300 °C as non-carbon elements are released [8]. Above 1300 °C, changes in the microstructure mostly consist of growth in the width of the microcrystallites appearing as elongated ribbons at the graphitization temperature [9]. Since bonding between crystal planes is weak, the carbon can be regarded as having two-dimensional bonding. Furthermore, the carbon atoms are bonded to one another by the π orbital composed of the sp² orbital and p orbital [10]. The carbonization process is quite time consuming [11–17] and the temperature increase rate is usually maintained at roughly 2–10 °C min⁻¹. In contrast, this study employed a temperature increase rate of 190–310 °C min⁻¹.

Although there are several compelling reasons for operating at a higher temperature [18], we selected 40 °C for the temperature of the fuel cell. While Chen [19] opted to study direct ethyl formate fuel cells at room temperature, we chose to work at 40 °C because in the future we plan to use fuel cells in 3C products. But while 3C products operate at room temperature, they usually produce heat, which may cause the temperature of the fuel cell to rise above room temperature and reach roughly 40 °C. We focus on GDL fabrication technology, and hope to boost fuel cell performance through improved GDLs. We also discuss the relationship

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between fuel cell performance and structure in carbon fiber fabric GDLs, and investigate the effect of thickness, air permeability, and surface resistivity. In the carbonization process, a high temperature causes non-carbon elements to volatilize, which results in the growth and linking of the carbon's crystal domains. While the conductivity of carbon fabric increases, resistivity rises with carbonization rate. XRD is consequently used to determine L_c (L_c : crystallite size in c direction (stacking size)), the effect of carbonization rate on the crystal structure of the carbon layer, and the effect of the carbon fiber cloth's crystal structure on cell performance. Furthermore, elemental analysis is used to determine the amount of carbon and nitrogen in carbon fiber cloth, and thereby gain an understanding of the effect of how the carbonization rate affects carbon and nitrogen content, and how these elements affect cell performance.

2. Experiment

The oxidized carbon fiber cloth used to fabricate GDLs in this study consisted of type LOW6713 made by the CeTech Co., Ltd. The oxidized carbon fiber cloth was first cut into pieces of an appropriate size, and then subjected to rapid carbonization. The temperature of the carbonization furnace was set at 1300 °C. The oxidized carbon fiber cloth was then rolled on a quartz tube, and carbonized in a carbonization furnace at different drawing roller rates (190, 220, 250, 280, 310 °C min⁻¹).

The thickness, air permeability, and surface resistivity of each piece was measured, and each piece was subjected to XRD and elemental analysis. A Teclock SM-114 thickness tester was used to measure the thickness of the carbon fiber paper, and thicknesses were the average of measurements taken at five random points. A Gurley Model 4320 meter was used to measure air permeability. Testing and analysis of air permeability was performed in accordance with Model 4110 regulations. A Loresta GP MCP-T600 meter was used to measure surface resistivity. Testing and analysis of surface resistivity was performed in accordance with JIS K 7194 regulations. The MXP-3 X-ray diffraction instrument had a scan angle of 60–10° and a scan rate of 2° min⁻¹, and used a copper target and a Ni filter. The Scherrer formula was used to derive L_c : $L_c = K\lambda / (B_{002} \cdot \cos \theta) - 1$, where L_c is the planar stacking thickness, K is the Scherrer constant (0.9), and λ is the diffraction line diffraction wavelength (0.1543 nm). A Universal CHNOS Elemental Analyzer Vario EL II from the German firm Elementar was used for elemental analysis. Samples to be analyzed were oxidized in pure oxygen at 1150 °C to CO₂ and NO_x, which were then reduced to CO₂ and N₂ using pure copper and separated in a GC column, after which the analyzer determined the weight percentages of carbon and nitrogen.

Preparing the carbon fiber fabric for use as a fuel cell GDL entailed cutting the paper into 5 cm × 5 cm pieces and then forming a three-layer MEA with catalyst-coated membrane (CCM) from Dupont™ (type NRE-211). This study focused exclusively on the material used for the GDL, and we plan to study how to spray or coat a microporous layer and PTFE on the GDL in the future. We did not use a microporous layer on the carbon fiber paper in this study. Furthermore, we did not bond the CCM and carbon fiber paper together by hot-pressing, but only used 40 kgf cm⁻¹ torsion to ensure close contact between the layers. The MEA was placed in a fuel cell testing module. The activated area was 25 cm², and the bipolar plates were gate-type grooved graphite plates made of highly compacted graphite. Stainless steel plates and PTFE washers were used to seal the module and create a fuel cell. Gas flow at the anode (H₂) was 500 cm³ min⁻¹, and at the cathode (O₂) was also 500 cm³ min⁻¹; the temperature of the anode and cathode was

40 °C. All single cell operations were performed using humidified pure hydrogen and oxygen gases without external pressures. Gas inlet pressure was 1 kg cm⁻², and cell temperature during testing was set as 40 °C. Gasket thickness was 0.36 mm.

3. Results and discussion

The success of the production of carbon fiber cloth for use in fuel cells GDLs primarily hinges on the carbonization process. The carbonization of carbon fiber cloth must be performed at high temperature in an anoxic environment in order to cause the elimination of non-carbon elements. The carbonization rate will affect how much time is available for the release of non-carbon elements. Gas desorption causes the release of non-carbon elements during the thermal decomposition of carbon-containing matter during the carbonization process, and ladder polymer cross-linking causes the fibers to gradually form basal planes. The carbon gradually assumes a layered structure as carbon fiber undergoes carbonization. The desorption of hydrogen, water, nitrogen, and other small molecules from the fibers allows the fibers to gradually achieve a tighter arrangement of carbon layers. In general, the initial stacking of the carbon in carbon fiber occurs when the heat treatment temperature is 25–1000 °C. The formation of ladder structures and lateral linkage creates localized planar structures from partial benzene rings. Carbon basal planes begin to form as the desorption of non-carbon elements gradually occurs. Less than 7% of the non-carbon elements in carbon fiber remain after the temperature exceeds 1000 °C; these elements chiefly consist of structural nitrogen that has not yet desorbed.

It can be seen from XRD patterns, such as the pattern shown in Fig. 1, which as the carbonization rate increased, the carbon layer stacking thickness (L_c) decreased. It can further be seen from Table 1 that carbon fiber cloth produced via conventional carbonization had an L_c of 25.69 Å, and that L_c did not change significantly when the carbonization rate was increased to 190 or 220 °C min⁻¹, remaining approximately 25.5 Å. However, when the carbonization rate was increased to 250 and 280 °C min⁻¹, L_c fell slightly to 25.3 Å, and L_c fell to only 19.3 Å at a carbonization rate of 310 °C min⁻¹. This effect is due to the desorption of non-carbon elements from the carbon fiber as temperature increases during carbonization, which causes the carbon basal plane stacking thickness (L_c) to gradually increase. In contrast, because rapid carbonization greatly shortens the time during which non-carbon elements can desorb from the carbon fiber, basal planes cannot properly form, and the L_c value will be low. This phenomenon is very apparent when the carbonization rate is increased to 310 °C min⁻¹.

Table 1 shows the measured thickness, air permeability, and L_c values for carbon fiber cloth produced using different carbonization rates. The thickness of carbon fiber cloth chiefly depends on the cloth's original thickness and final treatment temperature, and the original thickness has the greatest effect. The final treatment temperature (1300 °C) was not changed in this study, and all test pieces were fabricated from LOW6713 material. The chief experimental parameter that was changed was the carbonization rate. It can be seen from Table 1 that the thickness of carbon fiber cloth does not change due to carbonization rate. The thickness does not change significantly even in comparison with carbon fiber cloth made using conventional carbonization. It has been shown elsewhere [20] that the greater the thickness of carbon fiber cloth, the lower the air permeability. The thickness varied little in this study; however, permeability of the cloth was consistent with the results obtained by Liu et al. In conclusion, there is no significant difference between the air permeability of carbon fiber cloth made using conventional carbonization and that of cloth made using rapid carbonization.

Table 1
Thickness, air permeability, and Lc of carbon fiber cloth prepared using different carbonization rates.

Carbonization rate ($^{\circ}\text{C min}^{-1}$)	Thickness (mm)	Air permeability ($\text{cm}^3 \text{cm}^{-2} \text{s}^{-1}$)	Lc (\AA)
Traditional carbonization	0.57	43.2	25.69
$190^{\circ}\text{C min}^{-1}$	0.58	42.7	25.52
$220^{\circ}\text{C min}^{-1}$	0.60	41.5	25.56
$250^{\circ}\text{C min}^{-1}$	0.57	45.6	25.31
$280^{\circ}\text{C min}^{-1}$	0.61	42.6	25.37
$310^{\circ}\text{C min}^{-1}$	0.59	42.3	19.35

Fig. 2 shows the relationship between surface resistivity and Lc for carbon fiber cloth samples prepared using different carbonization rates. The \blacktriangle curve is the surface resistivity curve, and the \blacktriangledown curve is the Lc curve for carbon fiber cloth produced using different carbonization rates. The chart shows that while the Lc decreases as the carbonization rate increases, the surface resistivity of the cloth displays a significant rising trend. In other words, conductivity decreases as the carbonization rate increases. The surface resistivity of carbon fiber cloth prepared via conventional carbonization was $0.3 \Omega \text{sq}^{-1}$, and the surface resistivity of samples prepared using carbonization rates of 190, 220, and $250^{\circ}\text{C min}^{-1}$ were all approximately $0.33 \Omega \text{sq}^{-1}$. However, when the carbonization rate was increased to $280^{\circ}\text{C min}^{-1}$, the surface resistivity of the carbon fiber cloth rose slightly to $0.36 \Omega \text{sq}^{-1}$. The surface resistivity rose to $0.58 \Omega \text{sq}^{-1}$ when the carbonization rate was $250^{\circ}\text{C min}^{-1}$, and

this surface resistivity value was roughly 75% greater than that of cloth prepared using a carbonization rate of $190^{\circ}\text{C min}^{-1}$. This can be attributed to the low Lc and relative irregularity of the carbon layers in the carbon fibers when carbon fiber cloth is prepared using a high carbonization rate. In contrast, when the carbonization rate is low, the carbon layer structure of fibers in the carbon fiber cloth tends to be more regular and the Lc will be larger. Resistivity will be low when the electron-conducting layered carbon structure is regular. The irregular carbon layers of carbon fiber cloth prepared using a fast carbonization rate tend to obstruct the transmission of electrons, which reduces the conductivity and increases the surface resistivity of the cloth. This trend can be seen from the figure. If it is desired to achieve a relatively high conductivity with a short carbonization time, the carbon fiber cloth can be prepared using a carbonization rate of $250^{\circ}\text{C min}^{-1}$.

Fig. 3 is a bar chart of the carbon and nitrogen content of carbon fiber cloth prepared using different carbonization rates. It can be seen that the nitrogen content of the cloth tends to increase as the carbonization rate increases. The nitrogen content of carbon fiber cloth prepared using carbonization rates of 190, 220, and $250^{\circ}\text{C min}^{-1}$ was approximately 13.5%, but rose significantly to roughly 16% when the carbonization rate was increased to $280^{\circ}\text{C min}^{-1}$, and rose further to 16.5% when the carbonization rate reached $310^{\circ}\text{C min}^{-1}$. The carbon content of the carbon fiber cloth was approximately 72% when the carbonization rate was 190 and $220^{\circ}\text{C min}^{-1}$, and rose slightly to 74% when the carbonization rate was 250 and $280^{\circ}\text{C min}^{-1}$. The carbon content of the carbon fiber cloth fell significantly to 69% when the carbonization rate was $310^{\circ}\text{C min}^{-1}$, however. The carbon content of the original oxidized fibers was approximately 50%. Dehydrogenation occurs after the temperature reaches $400\text{--}600^{\circ}\text{C}$ during carbonization, and denitrogenation occurs at a temperature of $600\text{--}1300^{\circ}\text{C}$ [21]. When the carbonization rate is high – such as $310^{\circ}\text{C min}^{-1}$ – the carbonization time is relatively short, and the dehydrogenation and denitrogenation of the resulting carbon fiber cloth will be incomplete. The carbon content will thus tend to be low, and the nitrogen

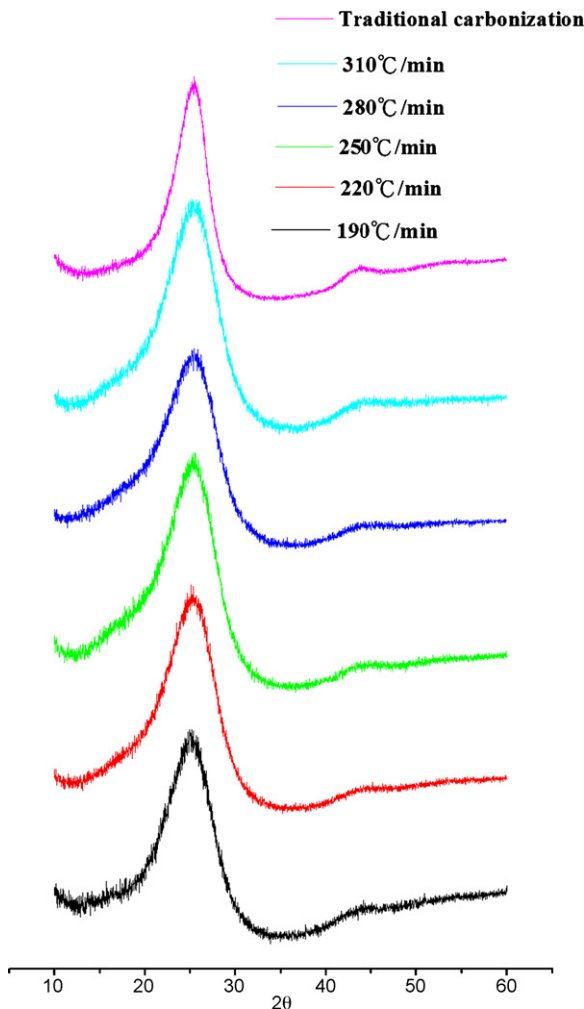


Fig. 1. XRD patterns of carbon fiber cloth prepared using different carbonization rates.

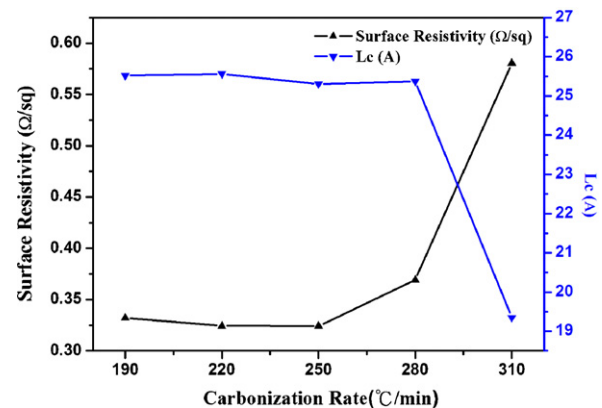


Fig. 2. Surface resistivity versus Lc for carbon fiber cloth prepared using different carbonization rates.

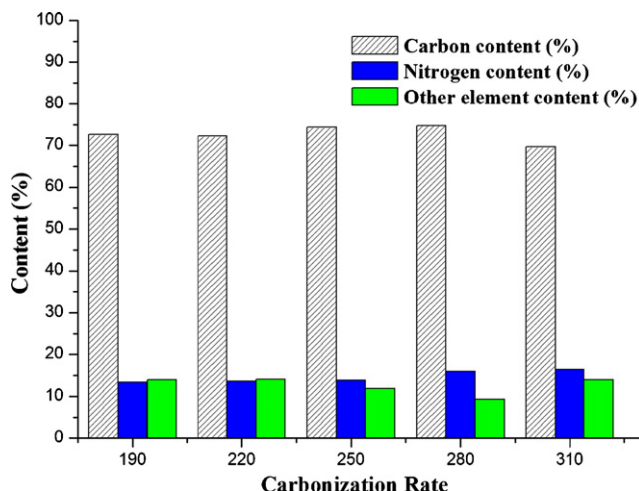


Fig. 3. Carbon and nitrogen content of carbon fiber cloth prepared using different carbonization rates.

content will tend to be high. In contrast, when the carbonization rate is relatively low, the carbonization, and denitrogenation of the carbon fiber cloth will be more complete. This will result in cloth with a high carbon content and low nitrogen content. This figure shows that it is desired to prepare carbon fiber cloth with a relatively high carbon content using a short carbonization time, a carbonization rate of $280^{\circ}\text{C min}^{-1}$ can be employed.

Table 2 shows the carbon content, nitrogen content, carbon/nitrogen ratio, and Lc of carbon fiber cloth prepared using different carbonization rates. It can be clearly seen that while cloth made with a conventional carbonization rate has a carbon content of as high as 98.07%, carbon fiber cloth made using rapid carbonization has a carbon content of roughly $70 \pm 5\%$. While rapid carbonization at a rate of $280^{\circ}\text{C min}^{-1}$ yields carbon fiber cloth with a relatively high carbon content, nitrogen content increases with the carbonization rate. A carbonization rate of $280^{\circ}\text{C min}^{-1}$ can be used to produce carbon fiber cloth with a low content of other elements. When the carbonization rate is increased to $310^{\circ}\text{C min}^{-1}$, however, the amount of other elements increases significantly, and the carbon content decreases significantly. This is because the carbon fiber cloth does not have sufficient time to complete dehydrogenation and denitrogenation when the carbonization rate when the carbonization rate is $310^{\circ}\text{C min}^{-1}$ or above. In addition, a high carbonization rate does not provide sufficient time for the carbon layers to rearrange themselves, which yields a carbon fiber cloth with a low Lc value. As a consequence, the carbon content and Lc of carbon fiber cloth prepared using a carbonization rate of $310^{\circ}\text{C min}^{-1}$ or above are both low.

Prior research [20] indicates that the thickness of the gasket and GDL may affect fuel cell performance. This study chose to use a gasket a thickness of 0.36 mm in fuel cell tests. The carbon fiber cloth pieces prepared using different carbonization rates were tested in single cells at a reaction temperature of 40°C . The resulting polarization curves are as shown in Fig. 4(a) and (b).

Table 2

Carbon content, nitrogen content, C/N ratio, and Lc of carbon fiber cloth prepared using different carbonization rates.

Carbonization rate ($^{\circ}\text{C min}^{-1}$)	Carbon content (%)	Nitrogen content (%)	Other element content (%)	C/N ratio	Lc (\AA)
Traditional carbonization	98.07	1.351	0.579	72.59	25.69
$190^{\circ}\text{C min}^{-1}$	72.59	13.42	13.99	5.41	25.52
$220^{\circ}\text{C min}^{-1}$	72.23	13.65	14.12	5.29	25.56
$250^{\circ}\text{C min}^{-1}$	74.35	13.81	11.84	5.38	25.31
$280^{\circ}\text{C min}^{-1}$	74.72	15.98	9.3	4.68	25.37
$310^{\circ}\text{C min}^{-1}$	69.63	16.44	13.93	4.24	19.35

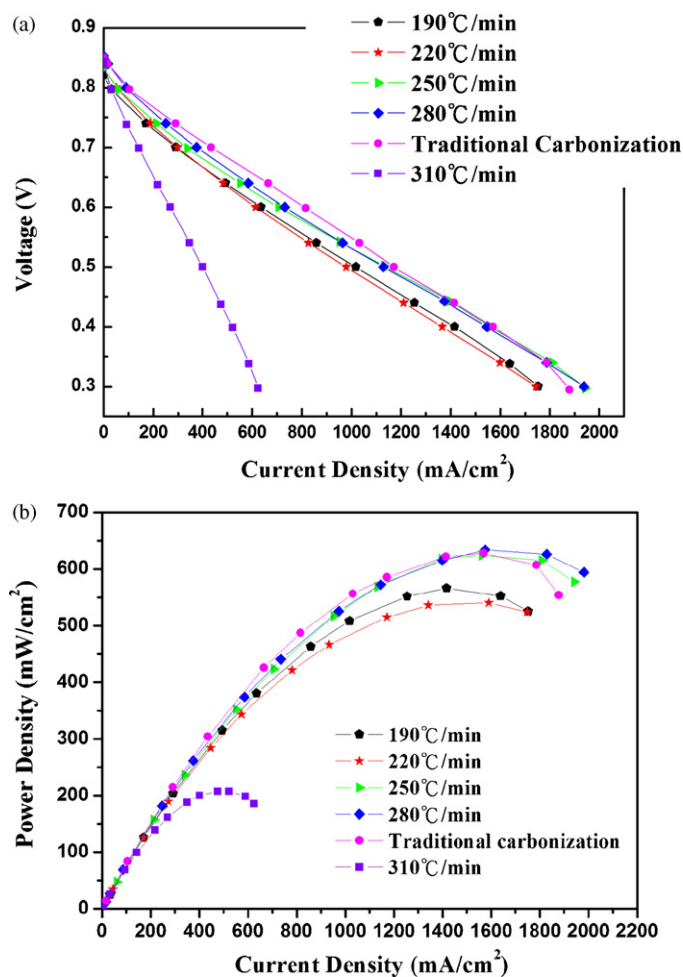


Fig. 4. (a) Current density versus operating voltage at 40°C for GDLs made from carbon fiber cloth prepared using different carbonization rates. (b) Current density versus power density at 40°C or GDLs made from carbon fiber cloth prepared using different carbonization rates.

The carbon fiber cloth used to fabricate the GDLs was not subjected to hydrophobic treatment, and no micro-electrode layer was applied. It can be seen from Fig. 4(a) and (b) that GDLs fabricated from carbon fiber cloth prepared using carbonization rates of 250 and $280^{\circ}\text{C min}^{-1}$ both yielded relatively good cell performance. When the load was 0.3 V, the current densities of these cells were 1942 and 1938 mA cm^{-2} respectively, while the maximum power densities were respectively 625 and 633 mW cm^{-2} . When the carbon fiber cloth was subjected to a carbonization rate of $310^{\circ}\text{C min}^{-1}$, however, the current density was 625 mW cm^{-2} and the maximum power density was 208 mW cm^{-2} . This performance was only roughly 30% that of cells employing carbon fiber cloth prepared using a carbonization rate of $280^{\circ}\text{C min}^{-1}$. In contrast, carbon fiber cloth prepared using conventional carbonization yield a current density of 1898 mA cm^{-2} and a maximum power density

of 628 mW cm^{-2} with a load of 0.3 V . The preparation of carbon fiber cloth using a carbonization rate of $310^\circ\text{C min}^{-1}$ thus results in relatively poor cell performance, but carbon fiber cloth prepared using a carbonization rate of $280^\circ\text{C min}^{-1}$ yields performance on a par with that obtained from carbon fiber cloth prepared using a conventional carbonization rate. The chief reason for the poor performance of carbon fiber cloth prepared using a carbonization rate of $310^\circ\text{C min}^{-1}$ is the low L_c , only 19.3 \AA , which causes surface resistivity to increase to $0.58 \Omega \text{ sq}^{-1}$, and a carbon content of only 69.9%. Although the L_c of carbon fiber cloth prepared using a carbonization rate of $280^\circ\text{C min}^{-1}$ was slightly low, it was still 25.3 \AA . In addition, the surface resistivity did not increase significantly, remaining at $0.36 \Omega \text{ sq}^{-1}$, and the carbon content was 74.7%. As a result, carbon fiber cloth prepared using a carbonization rate of $280^\circ\text{C min}^{-1}$ yields relatively good cell performance.

4. Conclusions

Carbon fiber cloth was prepared using carbonization rates of 190, 220, 250, 280, and $310^\circ\text{C min}^{-1}$ respectively. The maximum carbonization temperature was 1300°C . The carbon fiber cloth was used to fabricate fuel cell GDLs. This paper found that the carbonization rate affects cell performance by via the internal structure of the carbon fibers. While the L_c of the carbon fiber cloth was 25.52 \AA when the carbonization rate was $190^\circ\text{C min}^{-1}$, the L_c fell to 19.35 \AA when the carbonization rate was increased to $310^\circ\text{C min}^{-1}$. The faster the carbonization rate, the smaller the L_c of the carbon fiber cloth. Surface resistivity fell as carbonization rate was increased: The surface resistivity was $0.33 \Omega \text{ sq}^{-1}$ when the carbonization rate was $190^\circ\text{C min}^{-1}$, and rose to $0.58 \Omega \text{ sq}^{-1}$ when the carbonization rate was increased to $310^\circ\text{C min}^{-1}$. As for elemental composition, the carbon fiber cloth had a nitrogen content of 13.42% and a carbon content of 72.59% when the carbonization rate was $190^\circ\text{C min}^{-1}$, and a nitrogen content of 16.44% and a carbon

content of 69.63% when the carbonization rate was $310^\circ\text{C min}^{-1}$. The nitrogen content of the carbon fiber cloth rose as the carbonization rate was increased, and the carbon content also increased to a certain extent. The carbon content began to decrease when the carbonization rate reached $310^\circ\text{C min}^{-1}$, however. A gasket with a thickness of 0.36 mm was used during fuel cell performance testing. It was found that carbon fiber cloth prepared using a carbonization rate of $280^\circ\text{C min}^{-1}$ yielded relatively optimal performance. Taking carbon fiber cloth prepared with this carbonization rate as an example, the current density was 1938 mA cm^{-2} when the load was 0.3 V , and the maximum power density was 633 mW cm^{-2} .

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