

Article

Experimental Study on the Effect of Carbon Graphitization Degree and Pore Structure on the Electrochemical Durability of Gas Diffusion Layers

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Abstract: Gas diffusion layers (GDLs) in high-temperature, high-humidity, and high-electric-potential environments can be affected by the carbon corrosion and degradation of Polytetrafluoroethylene (PTFE) network structures, resulting in reduced reliability and hydrophobicity. By using cyclic voltammetry and offline characterization, a high-potential scanning of 1–1.5 V is applied to the GDL in the three-electrode system, considering the role of gradient graphitization degree and pore size structure in corrosion. Accelerating the electrochemical corrosion process of carbon and PTFE allows the identification of corrosion location, extent, and determinants. The results indicate that after 800 cycles of high-potential triangulation scanning, the graphitization of gas diffusion base has the most significant impact on the GDL's durability. On the other hand, the durability of the GDL's microporous layer is influenced by its small pore size structure rather than its graphitization degree. Furthermore, the corrosion process of GDLs with a small pore size structure tends to be relatively slow, providing a basis for GDL selection and durability prediction.

Keywords: proton-exchange membrane fuel cell; gas diffusion layer; durability; degree of graphitization; pore structure; electrochemical corrosion



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1. Introduction

The Proton Exchange Membrane (PEM) fuel cell is a clean energy conversion device that is highly efficient, clean, and operates quietly [1]. It has gained significant attention for its promising potential. By utilizing a catalyst, the PEM fuel cell generates current and heat through the electrochemical reaction of hydrogen and oxygen.

The gas diffusion layer (GDL) facilitates the transfer of reaction gases, conducting heat, and transmitting electrons and water [2,3]. The GDL consists of a gas diffusion base (GDB) and microporous layer (MPL). The GDB is made up of carbon fibers and a PTFE network, offering the passage of gas diffusion, backing membrane electrode assembly and conducting electrons [4]. The MPL is made up of carbon particles and PTFE. The shape of MPL is quite different than the GDB. The denser structure of the MPL serves as a transition between the catalytic layer and the carbon paper, reducing the overall contact resistance and providing channels for drainage and gas transfer [5].

The GDL possesses a porous structure that is hydrophobic and exhibits good thermal and electrical conductivity [6]. During operation, the reactant gas enters the catalyst layer through the pores of the GDL from the flow channels in the bipolar plate. Meanwhile, water in the electrode diffuses through the pores into the flow channels for discharge. Electron transfer occurs through the carbon fibers and carbon particles in the GDL during the electrochemical reaction [7]. The GDL also dissipates reaction heat through conduction, resulting in a higher potential at the cathode.

However, like all components in a PEM fuel cell, the GDL is subjected to durability loss during prolonged operation. Research on the durability of GDLs has shown complexities and multiple effects, considering different scenarios and specific cases [8,9]. There are three main categories of damage: potential oxidation during shutdown, long-time running stage oxidation at the cathode, and electrochemical corrosion under extreme anodic conditions. From an electrochemical point of view, the carbon electrode potential typically falls within the range of 0.4 V–1.2 V [10]. Therefore, understanding the mechanisms behind these forms of degradation is crucial for advancing research aimed at enhancing the functional reliability of GDLs.

The corrosion difficulties in the GDL vary depending on the location: GDB or MPL. The corrosion process is controlled by the structural and material characteristics [11]. Corrosion in the GDLs not only affects carbon materials but also PTFE materials coated on carbon fibers and particles, resulting in the loss of hydrophobicity due to the rupture and detachment of the molecular chains [12]. Therefore, it is important to understand the failure mechanisms of the corrosion process, design simulation accelerated experiments, conduct single-factor corrosion experiments, determine the influencing factors, and explore improvement directions [13].

There is limited research on the durability of the GDL. Some studies have focused on adding iridium or titanium oxide to the GDL and observing durability changes after fuel cell testing. However, these studies mainly address the trade-off between performance changes and durability improvement without delving into the structural- and material-level mechanisms of the GDL durability failure [14].

Offline accelerated durability experiments are more suitable for GDL durability research and development [15,16]. Electrochemical testing methods can be utilized to conduct these experiments, simplify operation procedures, improve the completeness of GDLs before and after durability testing, obtain electrochemical performance data, and facilitate offline characterization of key parameters to assess the degree of corrosion.

Some offline research has attempted to modify the MPL structure to mitigate carbon corrosion under the non-steady-state operating condition of PEMFC. By introducing a wavy micro-porous structure to promote corrosion mitigation, a GDL with enhanced corrosion resistance can be achieved [17]. However, the details of corrosion mechanisms, locations, and degrees are not extensively discussed. Research on simulation of GDL corrosion is even scarcer.

This setup facilitated the investigation of the corrosion potential of carbon and the simulation of offline accelerated corrosion of carbon and PTFE. This study revealed the locations, degrees, and determining factors of electrochemical corrosion, with a particular focus on the degradation mechanism of PTFE chains under electrochemical corrosion. The contact angle of the GDL was tested before and after the corrosion experiment, providing insights into the selection of GDL and predictions regarding its durability.

2. Materials and Methods

The experiment in this paper uses cyclic voltammetry combined with contact angle testing and other offline characterization methods to develop an offline electrochemical accelerated corrosion simulation method. Researchers here conducted high-potential corrosion experiments on commercial GDL samples with varying degrees of graphitization and pore structures within the voltage range of 1–1.5 V.

2.1. Accelerated Electrochemical Corrosion

To carry out the corrosion simulation, a three-electrode system was employed, comprising a platinum rod as the counter electrode, a GDL sample as the working electrode, and a calomel electrode as reference electrode. A 0.5 M H_2SO_4 solution served as the electrolyte. The Zahner electrochemical workstation was used to apply a scan voltage of 1–1.5 V, simulating the accelerated electrochemical corrosion of the GDL (Figure 1).

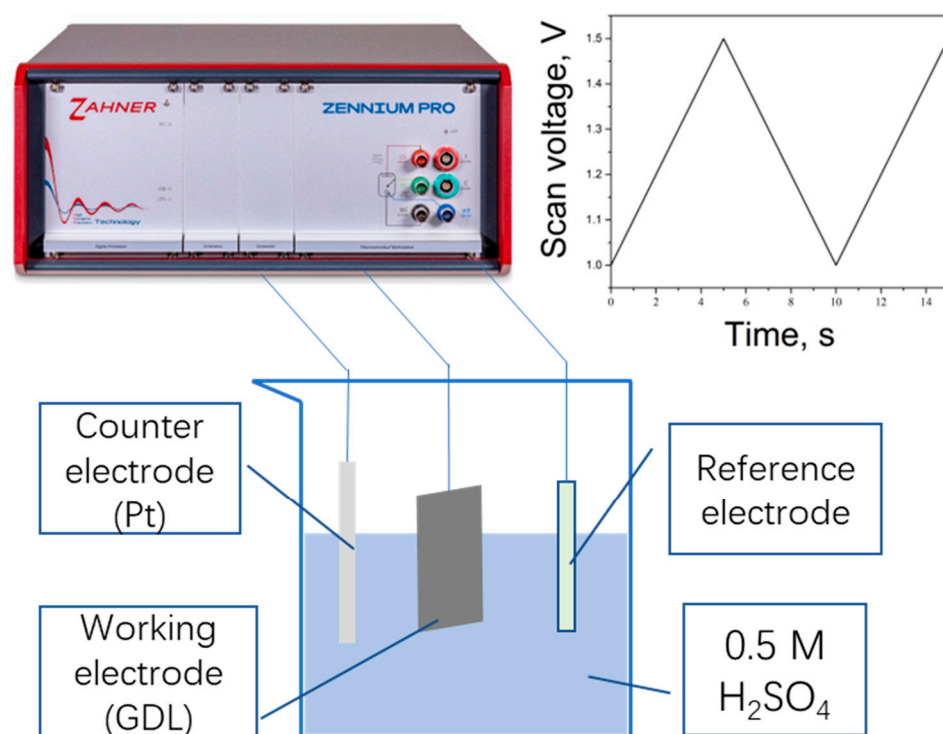


Figure 1. Electrochemical corrosion testing bench. Electrochemical workstation applied cyclic voltage scan to whole system in 0.5 M H₂SO₄ electrolyte, where the Pt rod worked as counter electrode, GDL samples worked as working electrode, and calomel electrode worked as reference electrode.

Experimental conditions are as shown in Table 1.

Table 1. Electrochemical corrosion parameter settings. A Zahner electrochemical workstation was used to apply cyclic voltage scan.

| Parameter | Value | Description |
|-----------------|--------------------------------------|------------------------------|
| Electrolyte | 0.5 M H ₂ SO ₄ | |
| Potential | 1–1.5 V | Carbon corrodes at 1.2 V |
| Scanning cycles | 800 | |
| Scanning rate | 100 mV/s | |
| Sample size | 4 cm × 6 cm | Area immersed by 4 cm × 4 cm |

After scanning 800 circles, the response current and contact angle before and after corrosion is compared to confirm the corrosion status.

2.2. Graphitization and X-ray Diffraction

The graphitization process of GDL includes GDB graphitization and MPL graphitization. For GDB that mainly consists of carbon fibers and MPL that mainly consists of carbon particles, graphitization degrees depend on the heat treatment under inert gas protection atmosphere. By setting the temperature gradient, carbon atoms are arranged more orderly at different degrees than that originally scattered and disorderly. The process of graphitization is the transformation of carbon materials from a two-dimensional structure of a carbon mesh to a three-dimensional ordered structure through “microcrystalline” growth under high temperatures. The closer the carbon structure approaches the ideal graphite, the higher the degree of graphitization is. When the temperature of GDB and MPL heat treatment process is set 4 degrees, different graphitization degrees are acquired.

X-ray diffraction (XRD) is a commonly used method to test the degree of graphitization in carbon materials, which reflects the ordering of carbon atoms and their electrical

conductivity. In this paper, a Smartlab 9 kW XRD test machine is used from Rigaku, Japan. For GDL carbon materials, due to the complex multiple elements of different graphitization degrees, the diffraction rays result in significant asymmetry. Grind and sieve the carbon fibers of GDL. Weigh 2 g of the sample and place it in two glass sheets for compaction. Place the pattern in the center of the test bench to ensure that all X-rays are irradiated on the sample. The X-ray tube is subjected to a 45 kV voltage then emits X-rays. It is controlled to rotate around the sample at a speed of $0.02^\circ/\text{min}$, from 0° (parallel) to 90° (vertical), to collect diffraction intensity and draw diffraction curves. By separately collecting carbon material samples from GDB and MPL, their full spectrum X-ray diffraction patterns can be tested, and the numerical values of graphitization can be quantitatively compared by Bragg's law:

$$d = \lambda / 2\sin\theta \quad (1)$$

where d is the spacing between crystal planes, θ is the angle between the incident X-ray and the corresponding crystal plane tested, λ is the wavelength of the X-ray which is 0.15406 nm here.

There are two extreme sides when it comes to the graphitization of carbon materials. When carbon materials are not subjected to graphitization treatment at all, their crystal plane spacing is 0.3440 nm, which can be expressed as 0%. When the carbon material is completely graphitized carbon, its crystal plane spacing is 0.3354 nm, which can be expressed as 100% degree. Therefore, the degree of graphitization of carbon materials with crystal plane spacing between the two can be expressed as a percentage.

By comparing with the planar interlayer spacing of standard graphitized carbon (0.3354 nm), graphitization degree g is acquired by Mering–Maire equation:

$$d = 0.3354g + 0.3440(1 - g) \quad (2)$$

g (%) is the GDL sample's graphitization degree.

2.3. Mercury Intrusion and Thermogravimetry Method

The mercury intrusion method is a technique used to assess the pore size distribution of a material by pressurizing mercury into the pores and measuring the pressure and amount of mercury consumed. A poremaster33 from Quantachrome is used to obtain a relationship curve between the volume of mercury and pore size, revealing the pore structure of the material.

Thermogravimetry method can be used to detect the PTFE loading of GDL sample, further investigating the hydrophobic ability. Heat up the samples in a protective atmosphere, and the PTFE component with low boiling point will melt and evaporate. By comparing the weight difference, the PTFE component in the GDL sample can be obtained in Table 2.

Table 2. PTFE content which is a factor in the contact angle performance and porosity of GDL samples.

| GDL ID | GDB PTFE Content | MPL PTFE Content | Porosity |
|--------|---------------------|---------------------|----------|
| #1 | 15 | 33 | 89.85% |
| #2 | 5 | 30 | 87.41% |
| #3 | 10 | 33 | 93.11% |
| #4 | 5 | 20 | 81.90% |
| #5 | 5 | 20 | 76.09% |
| #6 | 5 | 30 | 85.57% |

3. Results

3.1. Influence of GDB Carbon Graphitization Degree on Corrosion

To investigate the influence of the GDB carbon graphitization degree on the corrosion process, GDLs with similar pore size structure and MPL carbon graphitization degrees are

used. Controlling the carbon graphitization degree of the GDB (Figure 2), the influence of the GDB carbon graphitization degree on the corrosion process is investigated. The samples are #1 (GDB-XRD-3.15%), #2 (GDB-XRD-74.69%), and #3 (GDB-XRD-89.18%) in Table 3. In terms of pore structure, their average pore size is around 2 μm , and the MPL carbon graphitization degree is evaluated to be around 20%.

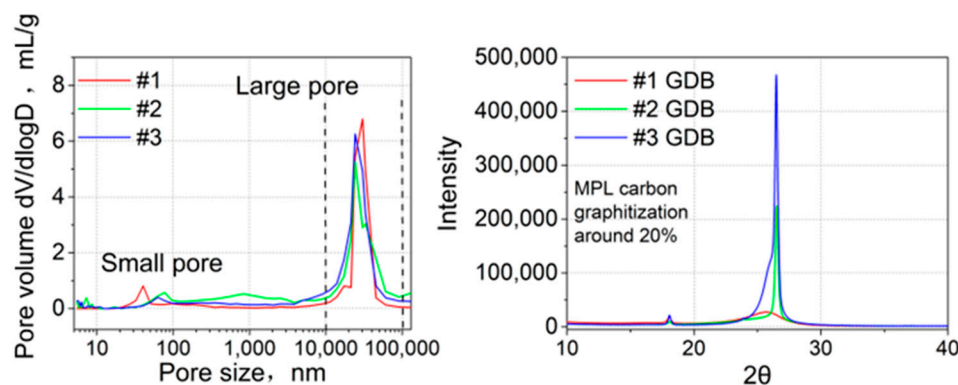


Figure 2. Same pore structure and MPL carbon graphitization but different GDB carbon graphitization degree in samples #1, #2, #3, by which the influence of GDB carbon graphitization degree on corrosion is determined under CV test.

Table 3. GDL samples involved and key parameters. Samples of #1, #2, and #3, which have similar MPL carbon graphitization degrees and pore structure, are used to determine the influence of GDB carbon graphitization degree on corrosion. Samples of #4 and #5, which have similar GDB carbon graphitization and pore structure, are used to determine the influence of MPL carbon graphitization degree on corrosion. And samples of #1, #4 and #6, which have similar GDB and MPL carbon graphitization, are used to determine the influence of pore size on corrosion.

| GDL ID | GDB 2 θ (°) | GDB Carbon Graphitization Degree (%) | MPL 2 θ (°) | MPL Carbon Graphitization Degree (%) | Pore Structure | Description |
|--------|--------------------|--------------------------------------|--------------------|--------------------------------------|----------------|--|
| #1 | 25.9 | 3.15% | 26 | 18.26% | Large | Influence of GDB carbon graphitization degree on corrosion |
| #2 | 26.36 | 71.69% | 25.96 | 12.23% | Large | |
| #3 | 26.48 | 89.18% | 26.08 | 30.26% | Large | |
| #4 | 26.08 | 30.26% | 25.9 | 3.15% | Small | Influence of MPL carbon graphitization degree on corrosion |
| #5 | 26.12 | 36.23% | 26.5 | 92.08% | Small | |
| #6 | 26.16 | 42.19% | 26.2 | 48.12% | Middle | Influence of pore size on corrosion |

With similar degrees of MPL carbon graphitization degrees and pore size structures, the initial response current decreases as the GDB carbon graphitization degree increases (Figure 3a). In fact, carbon with a higher graphitization degree exhibits superior conductivity and durability. For sample #1 (Figure 3b), the corrosion current reached 10 mA after 800 cycles, and the response current doubled. For sample #2 (Figure 3c), although the initial response current is low, it still increased as corrosion progressed, although it remained lower than the corrosion response current of sample #1. Sample #3 (Figure 3d) had the highest graphitization degree among all samples, and its initial response current and 800-cycle corrosion response current were the smallest, resulting in the highest durability.

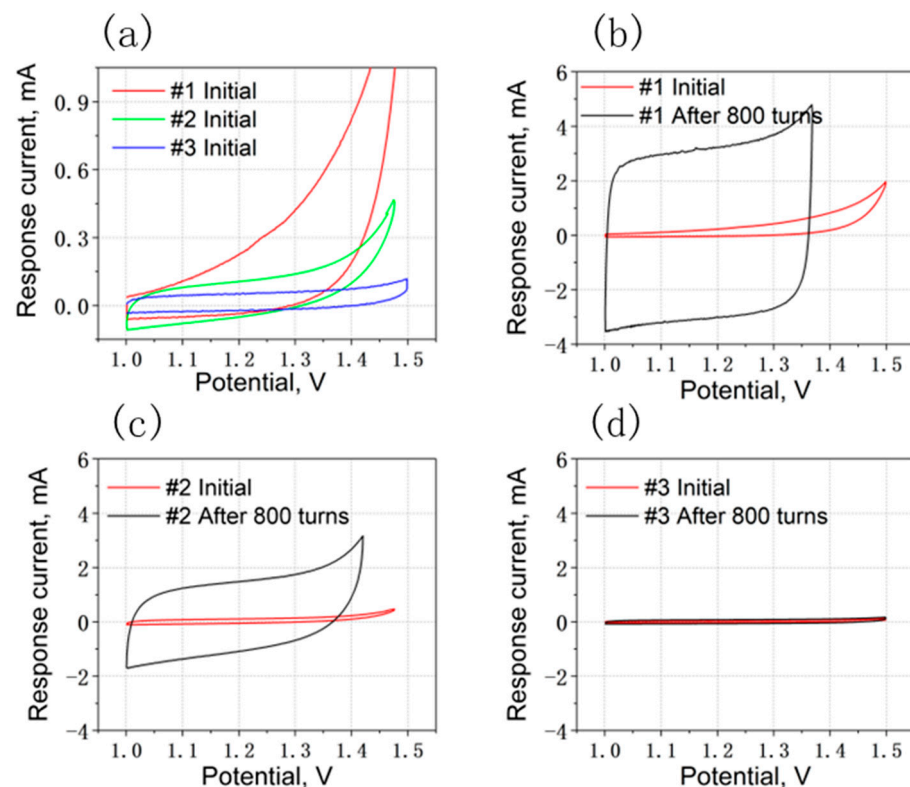


Figure 3. The effect of GDB graphitization degree on corrosion response current. (a) Different initial response currents under gradient GDB carbon graphitization degrees. The higher the graphitization is, the smaller the initial response current is. (b–d) Response current before and after the 800 turns scan. The most severe change occurs in #1, which has lowest GDB carbon graphitization degree. And the most moderate change occurs in #3, which has highest GDB carbon graphitization degree.

The change in response current after electrochemical corrosion follows the trend of the initial response current, indicating that the electrochemical durability of the GDL is related to the material's graphitization degree. Graphitization affects the chemical bond energy of the carbon atoms, which in turn affects the electrochemical corrosion process. Graphite, being a polymorph of elemental carbon, has a different molecular crystal structure compared to regular carbon. The carbon atoms in graphite have stronger binding forces, making it more resistant to corrosion. In 0.5 M H_2SO_4 solution, after an external potential scan of 1–1.5 V, the outermost carbon atom forms a covalent bond with three other carbon atoms, making it less prone to corrosion and detachment, resulting in a lower initial response current. Therefore, by detecting the GDB carbon graphitization degree, one can simply predict its durability.

Additionally, after high-potential electrochemical corrosion, the GDL not only experiences a degradation in its response current properties but also undergoes significant changes in its physical characteristics, specifically its hydrophobicity. It is demonstrated that the GDB and the MPL become more hydrophilic after corrosion in Figures 4 and 5.

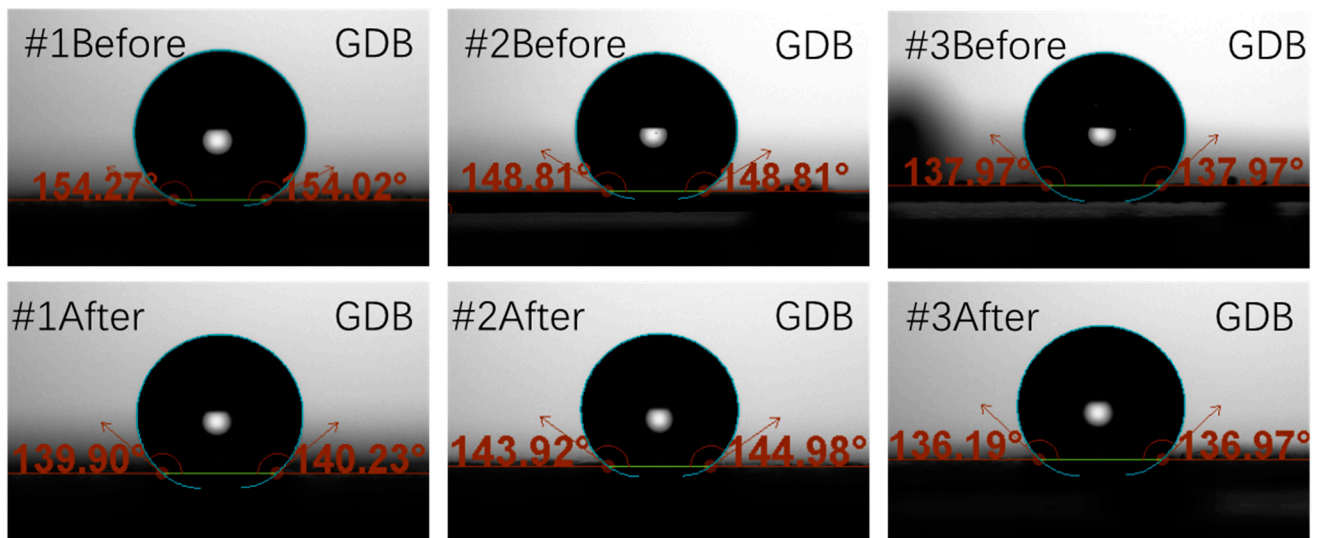


Figure 4. Changes in GDB contact angle before and after corrosion. For #1 GDB, nearly 15° decrease happened for it has poor resistance on corrosion. And for #3, it shows great resistance on CV scan.

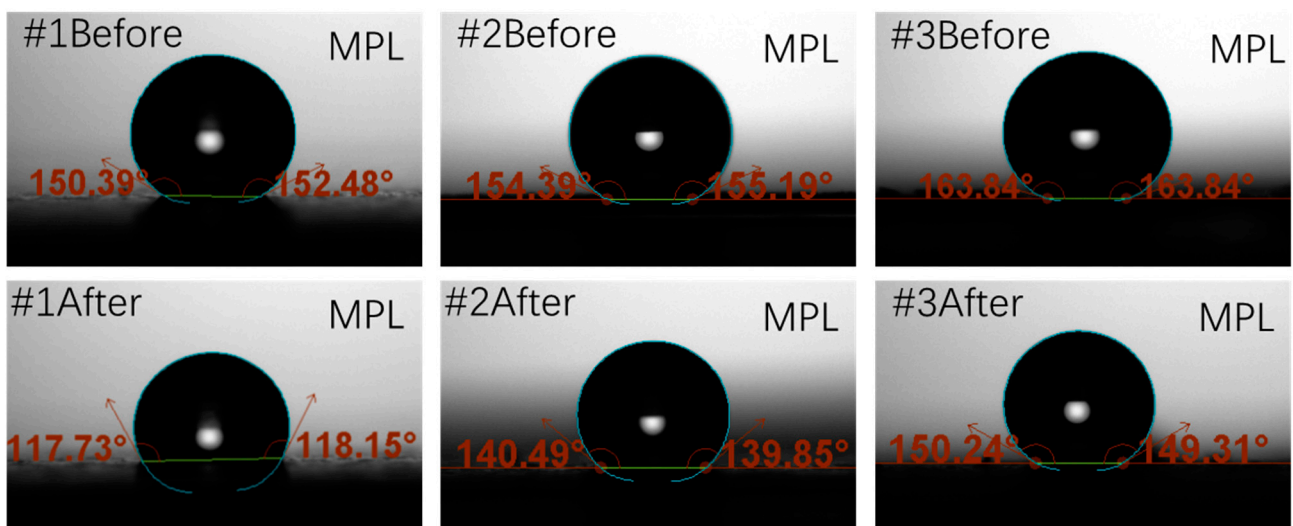


Figure 5. Changes in MPL contact angle before and after corrosion. All of the GDL samples occurred with decrease in contact angle.

For GDB, #1 drops by 15%, #2 drops by 5%, and #3 shows little drop. For MPL, #1 drops by 30%, #2 drops by 15%, and #3 drops by 10%. This phenomenon indicates that long-term high-potential electrochemical corrosion affects the hydrophobicity of the GDL, leading to abnormal water management in the fuel cell and promoting flooding at the MPL-CL interface.

The hydrophobicity of the GDL primarily relies on its internal network structure composed of polytetrafluoroethylene (PTFE). The different response currents observed during corrosion indicate varying degrees of carbon detachment. Consequently, the degree of graphitization and corrosion response current correspond with the deterioration of the contact angle.

3.2. Influence of MPL Carbon Graphitization Degree on Corrosion

To investigate the influence of MPL carbon graphitization degree on the corrosion process, GDLs with similar pore size structure and GDL carbon graphitization degrees are used. Controlling the carbon graphitization degree of MPL (Figure 6), the influence of

MPL carbon graphitization degree on the corrosion process is determined. The samples are #4 (MPL-XRD-3.15%) and #5 (MPL-XRD-92.08%) in Table 3.

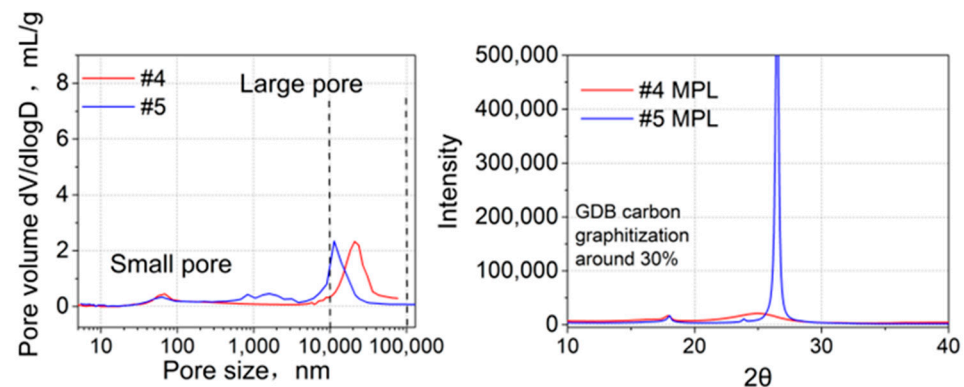


Figure 6. Same pore structure and GDB carbon graphitization but different MPL carbon graphitization degree in samples #4 and #5, by which the influence of MPL carbon graphitization degree on corrosion is determined under CV test.

In terms of pore size structure, their average pore size is $0.5\ \mu\text{m}$, and the degree of GDB carbon graphitization is evaluated as 30%.

With similar GDB carbon graphitization degrees and pore size structures, the initial response current shows similarity (Figure 7a) even though the MPL carbon graphitization degree is different. After a prolonged period of high-level current corrosion, the degree of deterioration is small and similar for all samples. However, in the case of #5 (Figure 7b), which has the highest graphitization degree among all the samples, it exhibits strong corrosion resistance.

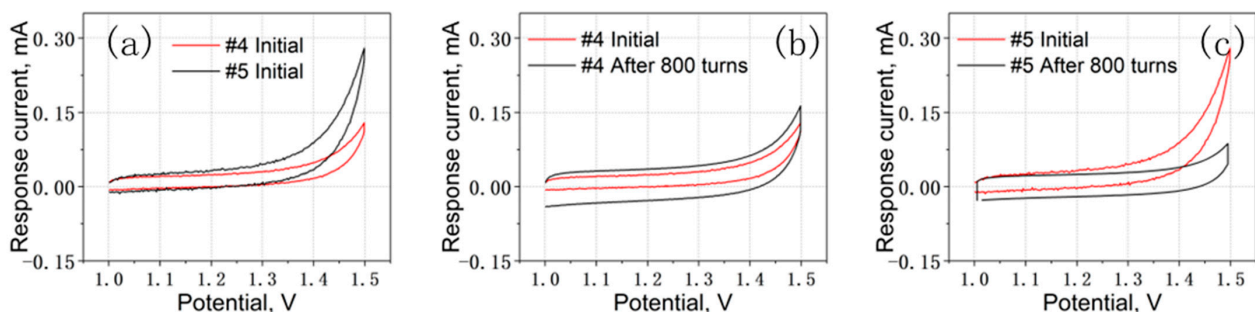


Figure 7. The effect of MPL graphitization degree on corrosion response current. (a) Different initial response currents under gradient MPL carbon graphitization degrees. There is little difference between two samples. (b,c) Response current before and after the 800 turns scan. Both changes are small indicating that the MPL carbon graphitization has little change with corrosion.

On the other hand, according to theoretical expectations, the trend of corrosion response current should have deteriorated greatly for both #4 and #5, as their GDB carbon graphitization degrees are low. However, the change in corrosion response current is small before and after 800 turns of electrochemical accelerated corrosion. This suggests that the influence of pore size structure on the electrochemical corrosion process cannot be ignored, as #1, #2, and #3 have larger pore structures compared to #4 and #5.

3.3. Influence of GDL Pore Structure on Corrosion

From the results of the last section, it is necessary to verify the influence of the pore size structure factor on electrochemical durability. By adopting GDLs with similar carbon graphitization degree, and controlling pore size structure of the GDL (Figure 8), the influence of pore size distribution on the corrosion process is discussed. The samples are

#4 (average pore size 0.5 μm), #6 (average pore size 2 μm), and #1 (average pore size 5 μm) in Table 3. Their graphitization degree is 30–42%, which is at a similar level.

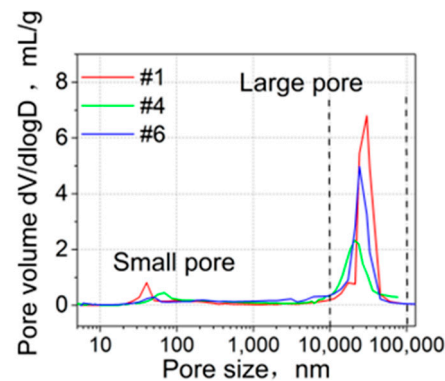


Figure 8. Similar GDB and MPL carbon graphitization but gradient pore structure in samples #1, #4, and #6, by which the influence of pore size on corrosion is determined under CV test.

With similar GDB and MPL carbon graphitization degrees and different pore size structures, the initial response current increases as the GDL pore size increases. The response current correspondingly deteriorates after 800 turns of corrosion related to the initial trend. For #4, although the degree of graphitization of MPL and GDB is not high, it shows strong corrosion resistance due to its small pore size. In contrast, for #1 and #6, the pore size is relatively large, thus durability is poor. Combined with the phenomenon above, it can be concluded that the durability of the GDL is not only affected by the degree of graphitization of the material, but also determined by the size of the pore structure. When the degree of carbon graphitization is similar, the durability is determined by the size of the internal pore, and the pore structure has a more profound impact on durability (Figure 9).

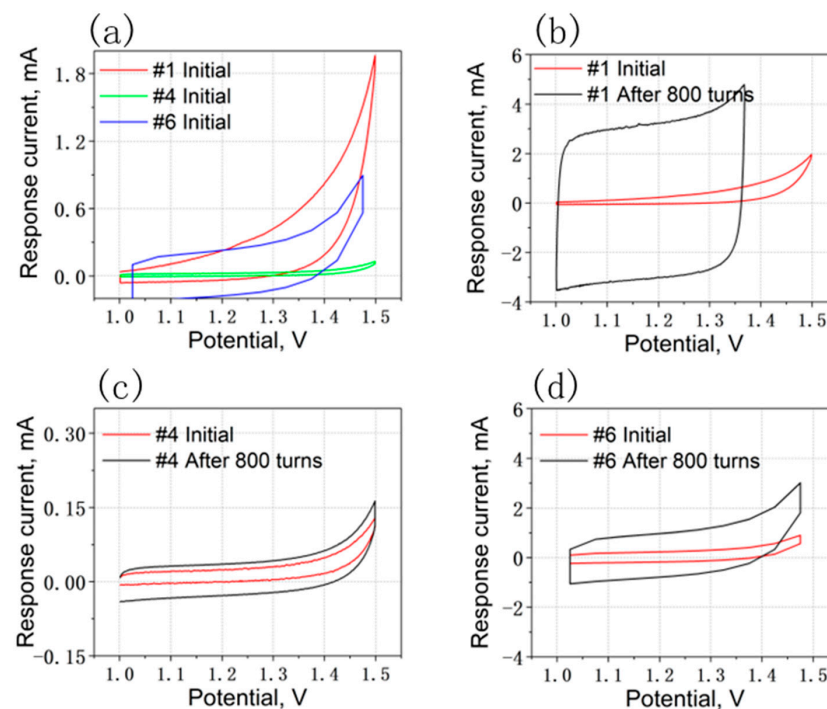


Figure 9. The effect of GDL pore structure on corrosion response current. (a) Different initial response currents under gradient pore sizes. The larger the pore size is, the bigger the initial response current is. (b–d) Response current before and after the 800 turns scan. The most severe change occurs in #1, which has largest pore size. And the most moderate change occurs in #4, which has smallest pore size.

The pore size reflects the state of the pore volume to the specific surface area of the GDL to a certain extent. Combined with the hydrophobic properties of GDL, GDL with larger pore sizes is exposed to more electrolyte in the three-electrode system, so the electrochemical reaction process is richer, leading to an increase in response current. Through the above conclusion, the durability of the GDL can be preliminarily predicted by confirming the pore size structure.

4. Conclusions

By conducting cyclic voltammetry and contact angle tests, high-potential triangulation wave scanning is applied to GDL samples. The corrosion response current under different GDB carbon graphitization degrees, MPL carbon graphitization degrees, and pore size structures are tested and compared. Effects on the electrochemical durability of the GDL are analyzed.

Based on appropriate standards, the initial corrosion response current of accelerated experiments is primarily determined by the degree of GDB carbon graphitization and is proportional to the electrochemical corrosion characteristics. After corrosion, samples with a poorer initial response current exhibit lower corrosion resistance ability.

The degree of MPL carbon graphitization and pore size structure have a synergistic effect on the electrochemical durability. A higher degree of MPL carbon graphitization provides better resistance to the electrochemical corrosion process. Additionally, a smaller pore structure offers good durability to the MPL.

Pore size strongly influences the durability of the GDL. In practical applications, a smaller pore size structure increases the breakthrough pressure for liquid water. This reduces the likelihood of forming strands of liquid water inside the GDL, thereby limiting the location of electrochemical corrosion.

According to the above conclusions, the offline test of carbon graphitization degree and pore size structure can predict the durability performance of the GDL, providing guidance for the selection and development of GDL materials.

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