Carbon coated stainless steel bipolar plates in polymer electrolyte membrane fuel cells

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The desirable properties of the metallic bipolar plates in polymer electrolyte membrane fuel cells (PEMFC) are good corrosion resistance, high electrical conductance, hydrophobicity, and low cost. In this study, carbon films are deposited on stainless steel 316L (SS316L) samples by close field unbalanced magnetron sputtering. The AFM, SEM, and Raman results show that the carbon film is dense, continuous, and amorphous. The corrosion resistance, hydrophobicity, and interfacial contact resistance (ICR) of the carbon coated steel are investigated and compared to those of uncoated SS316L. The deposited carbon film has high chemical inertness thereby significantly enhancing the corrosion resistance of the coated SS316L. Furthermore, the carbon coated SS316L is more hydrophobic and the resulting ICR is elevated to that of graphite. Our results indicate that the properties of the carbon coated SS316L are better than those of conventional graphite bipolar plates.

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1. Introduction

Environmental concerns and limited sources have spurred extensive research in alternative energy. The polymer electrolyte membrane fuel cell (PEMFC), which is one of the most promising power sources for transportation and distributed power generation, converts chemical energy of a fuel directly into electrical energy while permitting high efficiency, non-polluting operation, and low working temperature [1]. However, the high cost, large volume, and insufficient durability have prevented PEMFCs from more extensive commercialization. The bipolar plates which constitute one of the key multifunctional components in PEMFCs account for the large cost and volume in a typical fuel cell stack [2,3]. They serve to separate the individual fuel cell, connect adjacent cells with good conductivity, introduce the reactive gases, and remove the heat and reaction products (water) [4]. Therefore, good corrosion resistance, high electrical conductivity, good hydrophobicity, high gas impermeability, good mechanical performance, and low fabrication cost are some of the desired properties of bipolar plate materials.

Although graphite is commonly used in bipolar plates because of its excellent chemical inertness and conductivity [5–7], it is quite expensive to machine the gas flow channels. The graphite bipolar plate in the fuel cell stacks which is typically several millimeters thick is also quite brittle and has poor mechanical strength. In this respect, metallic bipolar plates are promising alternates due to their good mechanical strength, high gas impermeability, low cost, and easy manufacturing [8]. Among the various metals and alloys, stainless steel is desirable due to the low cost, high strength, and availability. For instance, it has been shown to deliver satisfactory performance for a thousand hours without obvious power density decline [9]. Nevertheless, the corrosion resistance, hydrophobicity, and interfacial contact resistance (ICR) must still be improved as these factors affect the performance and power output of the PEMFC. On account of its self-passivating nature, stainless steel is usually encapsulated by a passive film 1–3 nm thick which prevents the bulk from further corrosion, but this film significantly increases the ICR between the bipolar plate and carbon paper thereby compromising the cell performance due to ohmic loss. In addition, the passive film will dissolve and reform when the environment conditions change leading to leaching of metal ions. Therefore, alloys with special compositions and various surface modification techniques have been investigated in order to improve the performance of metallic bipolar plates [10,11]. One such candidate is a carbon film with a high sp² percentage which can serve as a coating on the bipolar plates due to its high electrical conductivity, hydrophobicity, and chemical inertness [12–14]. In our previous work [15], we prepared the carbon film by two steps. A Cr transition layer was first deposited followed by deposition of the amorphous carbon (a-C) film. In the work described here, the carbon
film was deposited onto stainless steel in one step without a catalytic layer, thereby making the process simpler and cheaper. Additionally, we studied the ICR and the contact angle of the carbon film systematically as well as the dissolved metal ions in the solution during the potentiostatic test. The carbon coated structure is demonstrated to be superior in terms of corrosion resistance, hydrophobicity, and ICR compared to bare SS316L.

2. Experimental details

2.1. Preparation and characterization of carbon film

Austenitic stainless steel 316L was purchased from Trinity Brand Industries, Inc. The chemical composition determined by XPS is shown in Table 1. SS316L samples were cut into dimensions of 15 mm × 15 mm × 4 mm and polished with No. 2000 SiC waterproof abrasive paper, cleaned with acetone and distilled water in an ultrasonic bath, and dried. The carbon film was deposited by close field unbalanced magnetron sputtering ion plating system consisting of two 99.99% pure graphite targets. This system was designed to be a low temperature coating system which worked well below 180 °C. The chamber was evacuated to a base pressure below 3 × 10⁻³ Pa using a turbo molecular pump. High purity argon (99.99%) and helium (99.99%) were bled into the chamber as the sputtering gases and the working pressure was maintained at 1.0 × 10⁻¹ Pa. Prior to deposition, the substrate was sputtered by 500 eV Ar ions to clean and remove the surface passive film and contamination. Afterwards, the carbon film was deposited using two carbon targets synchronously. The graphite target working current was 6 A and coating time was 4 h. Compared to our previous work [15], the target working current was lower in order to achieve a relatively low deposition rate.

Atomic force microscopy (AFM) and scanning electron microscope (SEM) were used to evaluate the surface morphology of the coated structure. Raman spectroscopy, being a very effective way to investigate the bonding type and domain size, was employed to determine the detailed bonding structure in the carbon film.

2.2. Electrochemical tests and ICP measurement

Corrosion resistance is an important factor in gauging bipolar plate materials. The potentiodynamic test, potentiostatic test, and inductively-coupled plasma atomic emission spectrometry (ICP-AES) were conducted to evaluate the corrosion resistance and corrosion rate of the bare and carbon coated samples. The samples were embedded in epoxy resin with an exposed surface of 10 × 10 mm² and the back side was connected to a copper wire by soldering. The three-electrode system comprised a platinum sheet as the counter electrode, a saturated calomel electrode (SCE) as the reference electrode, and the sample as the working electrode. The electrochemical tests were conducted in 0.5 M H₂SO₄ containing 2 ppm HF at 80 °C to simulate the aggressive PEMFC environment. The solution was purged with either air (to simulate cathode environment) or hydrogen gas (to simulate anode environment) prior to and during the electrochemical test. Before the electrochemical test, the open circuit potential (OCP) versus time was recorded for 1 h to ensure electrochemical stability. Potentiodynamic polarization was performed using a potential scanning rate of 1 mV s⁻¹. The potentiostatic test was carried out to investigate the performance and stability of the bare and carbon coated samples in the aggressive PEMFC environment. The potentiostatic test was conducted for 8.9 h at a potential of 0.6 V vs SCE while purging with air and −0.1 V vs SCE while purging with H₂ to simulate the cathode and anode operating environments in the PEMFC, respectively.

After the potentiostatic test, the solutions were collected and analyzed by inductively-coupled plasma atomic emission spectrometry (ICP-AES) to determine the amounts of Fe, Cr, Ni, and Mo ions dissolved in the solution.

2.3. Surface topography and contact angle

The samples after the potentiostatic test were carefully removed from the epoxy resin and cleaned with ethanol ultrasonically. Scanning electron microscopy (SEM) was performed on the samples before and after the electrochemical test in order to investigate the mechanism and extent of corrosion on both the bare and carbon coated SS316L.

Water flooding hinders the transport of oxygen by blocking the pores in the porous gas diffusion layer (GDL), covers active sites in the catalyst layer, and plugs the gas transport channels in the flow field [16]. In this respect, hydrophobic materials can effectively avoid water flooding in the PEMFC stack. Therefore, the water contact angle on the carbon coated SS316L was measured by a dataphysics OCA20 contact angle system. The volume of each water droplet was 4 μL and every sample was measured 5 times for better statistics.

2.4. Interfacial contact resistance (ICR)

The ICR between the metallic bipolar plate and gas diffusion layer (carbon paper) is a critical factor that affects the performance of the PEMFC stack. Therefore, the ICR values were determined from the bare and carbon coated SS316L by the method proposed by Davies’ [17] and Wang’s [18]. The schematic diagram of the measurement setup is illustrated in Fig. 1. Succinctly speaking, it consisted of a sandwiched structure using two pieces of conductive carbon paper (Toray TGP-H-090) sandwiched between the sample and two copper plates. A constant current (0.1 A) was applied via the two copper plates, and the variation in the total voltage was recorded with respect to the compaction force that was steadily increased. The area specific resistance (Rₛ) can be calculated by Eq. (1):

\[ Rₛ = \frac{V}{I} = \frac{V μ}{I} , \]

where \( V \) is the voltage drop, \( I \) is the total current applied, and \( Aₛ \) is the surface area of the sample.

The measured resistance of the system (\( Rₛ \) or \( R_{Total} \) mentioned below) can be divided into the bulk resistance (hereafter referred as \( R \))

![Fig. 1. Schematic diagram of the setup used to measure the ICR.](image-url)
and ICR (refered as r). Therefore, the measured resistance can be expressed as follows:

$$R_{total} = 2R_{copper-wire} + 2r_{copper-CP} + 2R_{CP} + 2R_{SS-CP} + R_{sample}$$  \hspace{1cm} (2)$$

where $R_{copper-wire}$, $R_{CP}$ and $R_{sample}$ are respectively, the sum of the bulk resistances of the copper plate and the wire, the bulk resistance of the carbon paper, and that of the sample, whereas $r_{copper-CP}$ and $r_{SS-CP}$ are the ICR between the copper plate and carbon paper and that between the SS316L sample and carbon paper, respectively. When one piece of carbon paper is sandwiched with two copper plates, the resistance of this new system is measured by

$$R_{base} = 2R_{copper-wire} + 2r_{copper-CP} + R_{CP}$$  \hspace{1cm} (3)$$

Since the bulk conductivity was excellent and $R_{sample}$ was much smaller than other resistances, it was assumed to be negligible and $R_{CP}$ was obtained from the supplier. Hence, the ICR between the SS316L and carbon paper ($r_{SS-CP}$) can be calculated from Eqs. (2) and (3) as below:

$$ICR(r_{SS-CP}) = \frac{R_{total} - R_{base} - R_{CP}}{2}$$  \hspace{1cm} (4)$$

3. Results and discussion

3.1. Characterizations of the carbon film

The surface topography and cross section of carbon film on the SS316L samples are observed by AFM and SEM and the results are shown in Figs. 2 and 3. The carbon film is observed to be composed of spherical carbon granules with diameters of 0.1 to 1 μm. The thickness of the carbon film is about 3.3 μm as shown in Fig. 3(b). Based on a coating time of 4 h, the carbon film deposition rate is estimated to be about 0.8 μm/h. The carbon film which is formed by accumulation of carbon spheres is compact and dense covering the entire stainless steel substrate. Consequently, the carbon film is expected to prevent the substrate from direct corrosion.

The Raman spectrum of the carbon coated SS316L is shown in Fig. 4. By means of deconvolution based on Gaussian fits, the G-band at approximately 1568 cm$^{-1}$ and D-band at 1390 cm$^{-1}$ correspond to the graphite band (originating from graphite lattice) and disordered band (originating from the defects in the graphite crystal), respectively [19]. It is suggested that the intensity of the D peak to that of the G peak ($I_D/I_G$) has a linear correlation with the graphite crystallite size [20–22]. As shown in Fig. 4, the intensity of the D-band is higher than that of the G-band, indicating that the size of the graphitic crystallites is small and the number of disordered bands is large. Ferrari and Robertson [20,23,24] have suggested that the G and D peaks are due to sp$^2$ only. The G peak arises from stretching of sp$^2$ atoms in both rings and chains, whereas the D peak is due to the breathing modes of A$_{1g}$.
involving phonons near the K zone boundary. This mode only becomes active in the presence of disorder.

3.2. Electrochemical test and ICP results

The potentiodynamic polarization behavior of the bare and carbon coated SS316L in 0.5 M H₂SO₄ with 2 ppm HF at 80 °C is shown in Fig. 5. The corrosion potentials of the SS316L in the cathode and anode environments are −0.286 V vs SCE and −0.307 V vs SCE, respectively. The corrosion potential of SS316L in the simulated cathode environment is higher than that in the anode due to the higher half-cell potential of $\text{e} (\text{O}_2/\text{H}_2\text{O}) [\text{O}_2 + 4\text{H}^+ + 4\text{e}^− \leftrightarrow 2\text{H}_2\text{O}]$ compared to that of $\text{e} (\text{H}^+/\text{H}_2) [2\text{H}^+ + 2\text{e}^− \leftrightarrow \text{H}_2]$ [25] as well as the formation of the passive film which is more noble. The carbon coated SS316L shows a more noble corrosion potential compared to the bare SS316L, being 247 mV vs SCE and 192 mV vs SCE in the simulated cathode and anode environments, respectively. Thermodynamically, a higher corrosion potential means higher chemical inertness and better corrosion resistance. The bare SS316L exhibits a polarization curve typical of austenitic stainless steel that can be divided into three regions, namely the active region, passive region, and transpassive region. A very high peak passivating current of about 1 mA cm⁻² can be observed from Fig. 5 due to active dissolution and oxidation of the metal. In the passive region, the passivation current density maintains its value regardless of potential increase. When the potential is increased to about 0.9 V vs SCE, the current density increases dramatically due to oxidation of $\text{Cr}^{3+} (\text{Cr}^{3+} \rightarrow \text{Cr}^{6+})$. As for the carbon coated SS316L, the major different in the polarization curve is that the carbon coated SS316L does not show a passive region. Fig. 5(a) shows the potentiodynamic polarization results when the solution is purged with air to simulate the aggressive PEMFC cathode environment. The cathode operation potential at 0.6 V vs SCE is marked by the red dotted line and the passivation current density of SS316L at the cathode operation potential is 11.26 μA cm⁻². In contrast, the current density of the carbon coated SS316L is significantly reduced to 1.85 μA cm⁻². The smaller anodic current density usually implies better durability and lower corrosion rate. Fig. 5(b) displays the potentiodynamic polarization curves in the simulated anode (bubbled with $\text{H}_2$) environment with the anode potential at −0.1 V vs SCE. The anode potential is in the active region of the SS316L but the corrosion potential of the carbon coated SS316L shifts towards the positive direction reaching about 0.2 V vs SCE. This is more positive than the operating potential in the PEMFC anode environment. Hence, the anode operation potential is cathodic to the carbon coated SS316L and corrosion in the simulated anode environment is retarded by the carbon film.

The bipolar plate under the real PEMFC working conditions undergoes corrosion at an applied potential (cathode 0.6 V vs SCE; anode −0.1 V vs SCE) which is different from the free corrosion potential. Therefore, the potentiostatic test is conducted to benchmark the corrosion resistance in the aggressive PEMFC environment. In the cathode environment, 0.6 V vs SCE is applied during the potentiostatic test while purging with air whereas in the anode environment, −0.1 V vs SCE is applied while purging with $\text{H}_2$. Fig. 6(a) shows the potentiostatic curves at 0.6 V vs SCE determined from the bare and carbon coated SS316L samples in the simulated PEMFC cathode environment. The current density of the bare SS316L decreases rapidly in the beginning and then gradually stabilizes at about 1.4 μA cm⁻². The fast drop in the current density is due to the reformation of the passive film. As soon as the whole surface is covered by a new passive film, the current density needed to maintain passivation is relatively low. This is also believed to be the reason why the current density in the potentiostatic test is much lower than that in the potentiodynamic test. The insert in Fig. 6(a) gives the accumulated charge of the bare SS316L and carbon coated SS316L during the test. There is a fast increase in the beginning followed by a linear relationship between charge and time. The change between the two parts is approximately 1 h. Thus, it is reasonable to consider that the time needed for the current to stabilize (totally passivated) is about 1 h. For the carbon coated SS316L, the current density stabilizes rapidly and then is maintained at around 2.4 μA cm⁻². The charge-time curve exhibits a linear relationship during the entire testing time, indicating that the carbon coating on SS316L prevents the substrate from passivation rendering it to be stable in the simulated PEMFC cathode environment. It is interesting to find that the current density of the SS316L is lower than that of the carbon coated SS316L in the potentiostatic test. As aforementioned, the passive film formed on the stainless steel surface greatly diminishes the corrosion rate. Since it is important to understand the mechanism and determine the extent of metal dissolution during the process, a post corrosion analysis of the test solution for the metal ions is performed.

Fig. 6(b) presents the potentiostatic curves at −0.1 V vs SCE acquired from the bare and carbon coated SS316L samples in the anode environment. The current density of SS316L decays dramatically, undergoes a positive-negative switch, and then gradually heads to the positive direction finally stabilizing at a relatively low negative level. This result indicates that the native passive film on the bare SS316L is not stable in the simulated PEMFC anode environment and it quickly dissolves followed by the formation of a passive film with a
different composition. In comparison, the current measured from the carbon coated SS316L does not exhibit such a switch. It increases in the beginning and gradually stabilizes at a lower current density. The cathodic current discloses that the sample is cathodically protected and the active dissolution rate is low in the anode environment.

The metal ions in the solution arising from corrosion are very important in gauging the performance of the bipolar plates. Here, the dissolved metal ions are determined by ICP and the results are summarized in Table 2. It is apparent that the Fe concentration is the highest in all cases, implying that Fe is selectively dissolved. The total metal ion concentrations leached from the uncoated SS316L are 10.08 ppm and 7.13 ppm after the potentiostatic test in the simulated cathode environment. More metal ions are released in the cathode environment than the anode environment, indicating that the cathode environment is more corrosive. This is reasonable since the negative current in the simulated PEMFC anode environment provides partial cathodic protection on the bare SS316L. In contrast, the metal ion concentrations leached from the carbon coated SS316L is found to be greatly reduced, being 1.99 ppm and 0.92 ppm after the cathode and anode potentiostatic test. This impressive reduction reveals that the carbon

![Fig. 6. Potentiostatic curves of the bare and carbon coated SS316L in the (a) simulated cathode (0.6 V vs SCE purged with air) and (b) anode environment (−0.1 V vs SCE purged with H2).](image)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ion concentration in PEMFC cathode environment after 8.9 h (ppm)</th>
<th>Ion concentration in PEMFC anode environment after 8.9 h (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe   Cr Ni Mo Total</td>
<td>Fe   Cr Ni Mo Total</td>
</tr>
<tr>
<td>Uncoated</td>
<td>8.53 0.86 0.62 0.07 10.08</td>
<td>5.63 0.78 0.59 0.13 7.13</td>
</tr>
<tr>
<td>Coated</td>
<td>1.86 0.06 0.05 0.02 1.99</td>
<td>0.87 0.04 0.01 0.92</td>
</tr>
</tbody>
</table>
coating gives rise to excellent chemical stability and durability in the simulated PEMFC environment.

3.3. Surface topography and contact angle

The bare and carbon coated SS316L samples are observed after the potentiostatic test by scanning electron microscopy (SEM) in order to investigate the mechanism and extent of corrosion. The micrographs are depicted in Fig. 7. The bare SS316L undergoes serious corrosion in both the cathode and anode PEMFC environments. There are many corrosion cells on the surface of the SS316L after polarization in the cathode environment [shown in Fig. 7(b)], whereas a porous corrosion layer is formed on the surface after polarization in the anode environment as shown in Fig. 7(c). The surface topography observed from the carbon coated SS316L does not show an appreciable difference with the exception of some adsorbed white corrosion byproducts on the surface, as shown in Fig. 7(d) to (f). This indicates that the carbon film is stable and can provide a protective barrier against direct corrosion of the stainless steel substrate in the simulated PEMFC environment.

Water is produced during the operation of the fuel cell. If the water removal rate does not keep up with the generation rate (at the cathode environment [shown in Fig. 7(b)])
cathode in particular), excess water will accumulate to degrade the cell performance [16]. Furthermore, water adhesion on the surface accelerates corrosion of the bipolar plate and so surface hydrophobicity is preferred. The average water contact angles measured from the bare and carbon coated SS316L samples are listed in Table 3 (each value representing the average of 5 measurements). The contact angle of the uncoated SS316L is rather small, being 48.1° because its large surface free energy attracts polar water molecules onto the surface. In contrast, the carbon coated SS316L sample has a much larger contact angle (85.5°) than SS316L. This is because that the carbon structure is non-polar consequently repelling polar water droplets (forming a spherical shape to lessen the contact area with the carbon surface) [12]. The contact angles on the carbon coated SS316L polarized in the cathode and anode environments are also measured to investigate the hydrophobicity of carbon film under the real working conditions. As shown in Table 3, the contact angles decrease after polarization. The electrochemical reaction on the carbon surface decreases the hydrophobic properties of the carbon film. The contact angle in the cathode (77.0°) is bigger than that in the anode (70.8°) probably due to the different environment being purged with gas.

3.4. Interfacial contact resistance (ICR)

The ICR between the bipolar plates and gas diffusion layer (carbon paper) is a critical factor in the internal resistance of the PEMFC stack. Therefore, the ICR values of the bare and carbon coated SS316L samples before and after the potentiostatic test are investigated as a function of compaction force (Fig. 8) with graphite as the reference. The actual ICR, which is influenced by the contact area between the sample and carbon paper as well as the conductivity of the sample surface, is further affected by the surface composition. Fig. 8 shows that the ICR decreases with compaction force exponentially [26], implying that the compaction force is dominant in the reduction of the ICR in the region with a small compaction force while the surface composition is the predominant factor in the region with a large compact force. The uncoated SS316L sample exhibits higher ICR values of 664.3–255.4 mΩ cm² at compaction force from 90 to 210 N cm⁻² due to the surface passivation layer acting as a semiconductor [27,28]. Compared to the bare SS316L, the ICR of the carbon coated sample is much lower throughout the whole range of applied compaction force and even lower than that of graphite. In particular, the ICR value of the carbon coated SS316L sample is 10.2–5.2 mΩ cm² and that of graphite is 17.8–5.4 mΩ cm² under 90–210 N cm⁻². Based on these results, the carbon film is electrically conducting. In the working PEMFC environment, the bipolar plates undergo corrosion and the surface composition may change leading to changes in the ICR. Therefore, the ICR values of the bare and carbon coated SS316L samples after the potentiostatic test are measured to evaluate the effects of corrosion. As shown in Fig. 8(a), the ICR values of the bare SS316L increase after polarization. According to our previous study [29], this is likely due to the change in the composition and thickness of the passive film formed during the potentiostatic test. Fig. 8(b) shows that the ICR of the carbon coated SS316L specimen also increases after the potentiostatic test. It is noted that the ICR values of the carbon coated SS316L sample in the cathode and anode environments are almost the same at about 46.0–18.4 mΩ cm² under 90–210 N cm⁻². Hence, the increase in the ICR values is not caused by purging with gas (air or H₂) but rather changes in the carbon bonding or adsorption of ions during polarization.

![Fig. 8. Variation in ICR values against the compaction force determined from (a) bare and carbon coated SS316L (b) carbon coated SS316L after the potentiostatic test.](image)

4. Conclusion

Carbon films are deposited SS316L samples by close field unbalanced magnetron sputtering. The AFM and SEM results show that the 3 μm thick carbon film is dense enough to form a continuous and compact film. The carbon film which evolves by aggregation of spherical carbon granules has an amorphous structure as indicated by SEM and Raman. The electrochemical behavior, hydrophobicity, and ICR values are determined. Results obtained from the potentiodynamic test, potentiostatic test, ICP measurement, and SEM are consistent demonstrating that the corrosion resistance is significantly improved by the carbon film. In particular, the current densities under the cathode operation potential are reduced from 11.26 μA cm⁻² to 1.85 μA cm⁻². The corrosion potentials diminish from about −307 mV vs SCE to 192 mV vs SCE in the simulated anode environment and are nobler than the anode operating potential. The potentiostatic and ICP results show that the carbon film is stable and greatly reduces the corrosion rate of the SS316L. The contact angle on the uncoated SS316L is much smaller than that of the carbon coated one suggesting that the carbon film is more hydrophobic. The ICR of the carbon coated SS316L is 10.2–5.2 mΩ cm² under 90–210 N cm⁻² whereas that of the bare SS316L is much higher because of the passive
film on the surface. Our results indicate that the carbon coated SS316L combines the advantages of graphite and stainless steel and is expected to reduce the volume and cost while improving the performance of bipolar plates in PEMFCs.

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