Performance of a proton exchange membrane fuel cell stack using conductive amorphous carbon-coated 304 stainless steel bipolar plates

Peiyun Yi[a], Linfa Peng[a,*], Lizhong Feng[b], Pin Gan[b], Xinmin Lai[a]

[a] *State Key Laboratory of Mechanical System and Vibration, Shanghai Jiao Tong University, Shanghai 200240, PR China*
[b] *SAIC Motor Corporation Ltd., Shanghai 200041, PR China*

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ABSTRACT

In this study, 304 stainless steel (SS) bipolar plates are fabricated by flexible forming process and an amorphous carbon (a-C) film is coated by closed field unbalanced magnetron sputter ion plating (CFUBMSIP). The interfacial contact resistance (ICR), in-plane conductivity and surface energy of the a-C coated 304SS samples are investigated. The initial performance of the single cell with a-C coated bipolar plates is 923.9 mW cm^{-2} at a cell voltage of 0.6 V, and the peak power density is 1150.6 mW cm^{-2} at a current density of 2573.2 mA cm^{-2}. Performance comparison experiments between a-C coated and bare 304SS bipolar plates show that the single cell performance is greatly improved by the a-C coating. Lifetime test of the single cell over 200 h and contamination analysis of the tested membrane electrode assemble (MEA) indicate that the a-C coating has excellent chemical stability. A 100 W-class proton exchange membrane fuel cell (PEMFC) short stack with a-C coated bipolar plates is assembled and shows exciting initial performance. The stack also exhibits uniform voltage distribution, good short-term lifetime performance, and high volumetric power density and specific power. Therefore, a-C coated 304SS bipolar plates may be practically applied for commercialization of PEMFC technology.

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1. Introduction

Proton exchange membrane fuel cells (PEMFCs) are considered to be promising power sources for automotive, stationary and portable equipments due to their high efficiency, high power density and low environmental impact [1]. As one of the most important components in PEMFCs, bipolar plates constitute the backbone of stack, conduct current between cells, facilitate water and thermal management through the stack, and provide conduits for reactant gases [2]. Typically, the bipolar plates account for approximately 80% of the stack volume, 70% of the stack weight and as much as 60% of the stack cost [3,4]. Graphite composites are currently employed to fabricate bipolar plates in PEMFCs due to their excellent chemical stability and electrical conductivity. Nevertheless, the costs of graphite composite bipolar plates in material and machining flow field are very high and a sufficient thickness is necessary for redeeming the poor mechanical strength of graphite composites so as to prevent crush during the PEMFCs assembly process [5]. Stainless steel is considered to be a promising candidate for bipolar plates in PEMFCs because of cost effectiveness, higher mechanical strength, better durability to shock and vibration, no

permeability, and superior manufacturability compared to graphite composites. Since the bipolar plates work in the harsh acidic and humid environment in PEMFCs stemming from perfluorosulfonic acid membrane, the primary challenge of stainless steel bipolar plates is surface corrosion. The corrosion behavior of the bipolar plates leads to a release of metal ions, e.g. Fe, Cr, Ni, etc. which can contaminate the electrolyte membrane and poison the electrode catalysts. Furthermore, the formation of a passivating oxide or oxhydroxide layer on the surface of stainless steel will increase the interfacial contact resistance (ICR) between bipolar plate and gas diffusion layer (GDL) [6–8]. Both conditions can significantly degrade stack performance [9]. Therefore, great efforts are still required to combat metallic corrosion and eliminate the passive layer.

A promising approach is to coat a highly conductive layer which is both chemically and mechanically stable on stainless steel substrate. Carbon film which may integrate the merits of graphite composites into metallic bipolar plates is a potential candidate. Fu et al. [10] coated carbon-based films by pulsed bias arc ion plating (PBAIP) on 316L stainless steel substrate to obtain protecting layers. These results indicated that the interfacial conductivity of the bipolar plates with pure C film or with C–Cr–N film was not satisfied, but the bipolar plates with C–Cr film exhibited a very low ICR. Fukutsuka et al. [11] prepared a carbon coating on SUS304 using plasma-assisted chemical vapor deposition. The corrosion

* Corresponding author. Tel.: +86 21 34206302; fax: +86 21 34204542.
E-mail address: penglinfa@sjtu.edu.cn (L. Peng).

resistance of the bipolar plates was improved in simulated PEM-FCs environments, and the ICR was also greatly reduced. Chung et al. [5] developed a continuous carbon film on AISI 304 stainless steel by thermal chemical vapor deposition. Both the corrosion tests and PEMFCs operation indicated that the carbon film had excellent chemical stability similar to that of high purity graphite plates. Show [12] prepared an amorphous carbon (a-C) film on titanium bipolar plates at various temperatures by using the radio frequency plasma enhanced chemical vapor deposition (RF-PECVD) method. The results revealed that the a-C film coated titanium bipolar plates had lower ICR and the stack with the a-C coated Ti bipolar plates showed performance improvement. Feng et al. [13] coated 316L stainless steel samples with an a-C film about 3 μm in thickness by closed field unbalanced magnetron sputter ion plating (CFUBMSIP). Testing results indicated that the ICR of the a-C coated SS316L samples was 8.3–5.2 mΩ cm^2 under compaction pressures of 1.2–2.1 MPa and the passivation current density was reduced from 11.26 to 3.56 μA cm^{-2} with the a-C coating in the simulated cathode environment. However, most of the carbon coating performance was evaluated by small samples in simulated PEM-FCs environments. Carbon film coated stainless steel bipolar plates should be evaluated in a PEMFC stack and the lifetime of coating in the real fuel cell environment is still worth studying.

In this study, an a-C film was coated on 0.1 mm thick 304SS foil using the CFUBMSIP process. The ICR, in-plane conductivity and surface energy of the a-C coated 304SS were investigated as well. A single cell and a 100 W-class PEMFC short stack with a-C coated bipolar plates were assembled and evaluated. Lifetime test of the single cell was performed for 200 h of continuous operation and contamination of the tested membrane electrode assembly (MEA) was analyzed by inductively coupled plasma-mass spectrometry (ICP-MS).

2. Experimental

2.1. Design, fabrication and material tests for a-C coated 304SS bipolar plates

As shown in Fig. 1, the proposed bipolar plate design satisfies functional requirements of three inlets and three outlets: cathode reactant inlet/outlet, anode reactant inlet/outlet, and coolant water inlet/outlet [14]. Fourteen equal-length serpentine flow channels are used to distribute reactant gas uniformly on the anode and cathode. The land width, channel width and channel depth of flow channels are 0.9 mm, 1.1 mm and 0.4 mm, respectively. The bipolar plate is 100 mm $\times$ 100 mm.

0.1 mm thick 304SS foil was selected as the bipolar plate material due to its lower cost compared with 316L stainless steel.
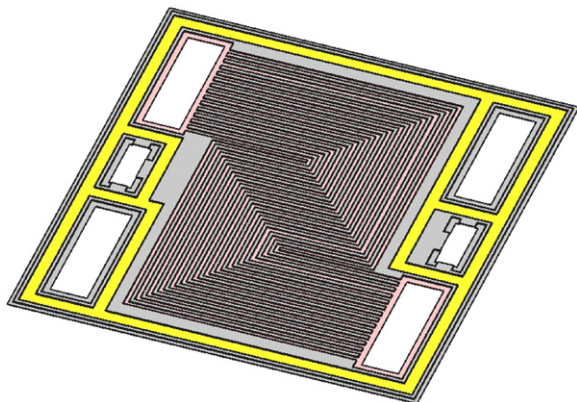
Flexible forming process, which utilizes a rigid die and a soft punch made of materials such as natural or synthetic rubbers [15], was adopted for bipolar plate fabrication. The forming experiments were conducted by a 500 tonnes hydraulic press machine and the cycle time for a single plate was less than 1 min. Two single plates were joined together using resistance spot welding process (Inverter Resistance Welding Machine IP/IT-3000). The margin outside of the effective area was cut out by wire Electrical Discharge Machining (EDM). Before coating, the bipolar plates were carefully cleaned by ultrasonic cleaning. The a-C film was deposited onto the bipolar plates using a CFUBMSIP system with 99.99% graphite and 99.99% Cr targets, respectively. High purity argon (99.99%) and helium (99.99%) were used as the sputtering gases [13].

The ICR of a-C coated 304SS samples with size of 20 mm $\times$ 20 mm $\times$ 0.1 mm was measured by techniques well documented in the literatures [16–18] and experimental setups developed by Lai et al. [19]. Toray TGP-H-060 carbon paper from Toray Industries, Inc. was adopted as simulated GDL. This carbon paper was the same as that in MEA used in fuel cell performance evaluation experiments. In-plane conductivity was measured by the well-known four-point probe technique. The probe diameter is 1.0 mm and contact pressure for each probe is 1.2 MPa. A constant current was applied through the two outermost probes, and the resulting voltage across the two innermost probes was measured. To evaluate the surface energy of the a-C coated 304SS, contact angle was measured by Dataphysics OCA20 Optical Contact Angle Measuring Device. The electrical and physical properties of bare 304SS samples were also measured for comparison.

2.2. Single cell tests

A self-developed single cell was designed and fabricated in house. Commercial MEA with platinum loading of 0.5 mg cm^{-2} for the anode and cathode as well as an active electrode area of 40 cm^2 was adopted. 15 mm thick aluminum plates were used as the end plates and 2 mm thick brass plates were applied as the current collectors. Coated bipolar plates, MEAs, silicon seals and current collectors were clamped between the two end plates by eight M4 screw joints and each had a torque of about 5 N m. Cell temperature was kept at 60 °C by two heating plates attached to the aluminium end plates. Performance of the single cell was evaluated by measuring I–V curves with the NBT-100 W fuel cell test system. The testing experiments were carried out under scanning current mode and the results were recorded after 1 h of stable operation. Parameters for the single cell tests are shown in Table 1. A single cell with bare 304SS bipolar plates was also assembled and tested under the same conditions for comparison.

To investigate chemical stability of the a-C coating, the two single cells mentioned above were operated at a cell voltage of 0.6 V and current density was measured as a function of operating time. After 200 h of continuous operation, contamination (Fe, Cr, Ni) con-

Fig. 1. Schematic of the proposed 304SS bipolar plate design.

Table 1
Operating conditions for the single cell and 100 W-class short stack tests.

	Single cell	100 W-class short stack
Active area	40 cm^2	120 cm^2
Anode stoichiometric ratio	1.2	1.2
Cathode stoichiometric ratio	2.0	2.0
Anode pressure	0.3 MPa	0.3 MPa
Cathode pressure	0.3 MPa	0.3 MPa
Cell temperature	60 °C	60 °C
Reactants temperature	80 °C	80 °C
Anode back pressure	30 kPa	50 kPa
Cathode back pressure	50 kPa	100 kPa
Hydrogen humidification	30%	30%
Oxygen humidification	30%	30%
Heating/cooling type	Heating	Circulating water cooling
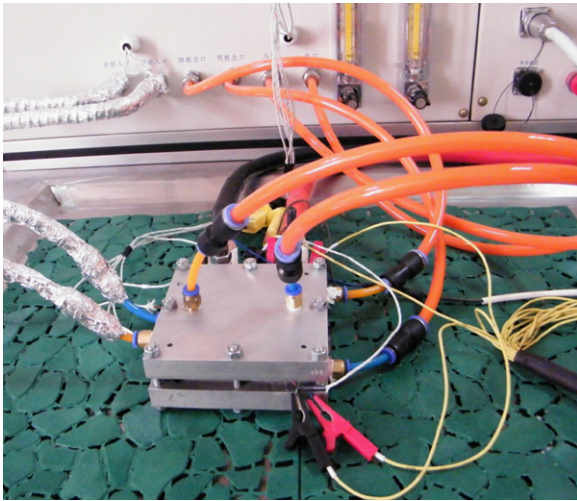
Fig. 2. Picture of the 100 W-class PEMFC short stack using a-C coated 304SS bipolar plates.

Fig. 3. Contact resistance of a-C coated and bare 304SS at various compaction pressures.

tent in the tested MEAs was analyzed using ICP-MS (Agilent 7500a, Agilent Technologies Inc., USA). For reference, the contamination of a fresh MEA was also analyzed.

2.3. 100 W-class PEMFC short stack tests

A 3-cell short stack was assembled using coated bipolar plates as shown in Fig. 2. Circulating water was used to cool the short stack with the deionised water going through the coolant channels in bipolar plates. Cell temperature was kept at 60 °C by adjusting the temperature and flow rate of coolant water. Parameters for the 100 W-class short stack tests are shown in Table 1. I–V curves were scanned with the NBT-1KW fuel cell test system after 2 h of stable operation. For performance comparison, a 3-cell short stack with bare 304SS bipolar plates was also assembled and evaluated under the same conditions.

Voltage distribution was monitored to analyze the uniformity of stack at various voltage levels. The 100 W-class PEMFC short stack was operated for 48 h at a constant voltage of 1.8 V and the current was recorded for short-term lifetime analysis. Volumetric power density and specific power were also investigated as feasibility analysis for commercialization of the fuel cell technology.

3. Results and discussion

3.1. Material characterization of the a-C coating

High in-plane conductivity and low ICR are essential features for PEMFC bipolar plates. Using four-point probe technique, the in-plane conductivity of a-C coated and bare 304SS samples was measured as shown in Table 2. The in-plane conductivity of a-C coated 304SS is slightly lower than that of bare 304SS. That is because the surface layer of the carbon film contains numerous disarranged graphite structures and the conductivity of graphite is lower than 304SS [5]. However, this value is much higher than the Department of Energy (DOE) target: >100 S cm^{-1} [20]. As a

matter of fact, contact resistance in an operating fuel cell has a much stronger effect on cell performance than in-plane conductivity [21]. The contact resistance of a-C coated and bare 304SS samples with Toray TGP-060 GDL was measured at various compaction pressures and the results are presented in Fig. 3. With an increase in compaction pressure, the contact resistance decreases rapidly at low compaction pressures and then decreases gradually at high compaction pressures. At 1.5 MPa, the contact resistance of a-C coated 304SS is 5.4 mΩ cm^{-2} which is lower than the DOE target of 10 mΩ cm^{-2} [20], while the contact resistance of bare 304SS is 93.3 mΩ cm^{-2}.

Wettability has a strong effect on cell performance particularly at high current densities [10,22]. The lower the surface energy of the bipolar plates, the more easily the flooding can occur at the cathode. Therefore, bipolar plates with high surface energy would be helpful for water removal in the stack and beneficial to the simplification of water management [21]. A high contact angle implies a high surface energy and low surface wettability of a material. Fig. 4 shows that the contact angle of a-C coated 304SS is 78.8° while that of bare 304SS is 73.2°. These results demonstrate that the surface energy of a-C coated 304SS is improved by the a-C coating. However, great efforts should still be made to increase the surface tension since the contact angle of a-C coated 304SS is still lower than 90° which is considered to be an ideal value [2].

Table 2
In-plane conductivity of a-C coated and bare 304SS.

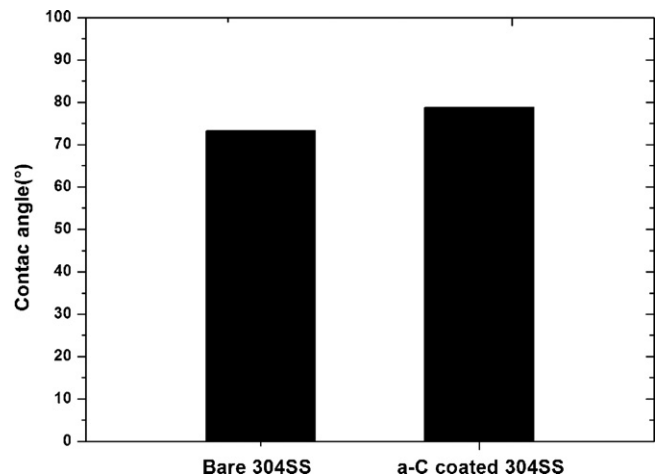| Bipolar plates	In-plane conductivity (S cm^{-1})
a-C coated 304SS	7955.4
Bare 304SS	8326.4

Fig. 4. Static water contact angle of a-C coated and bare 304SS.
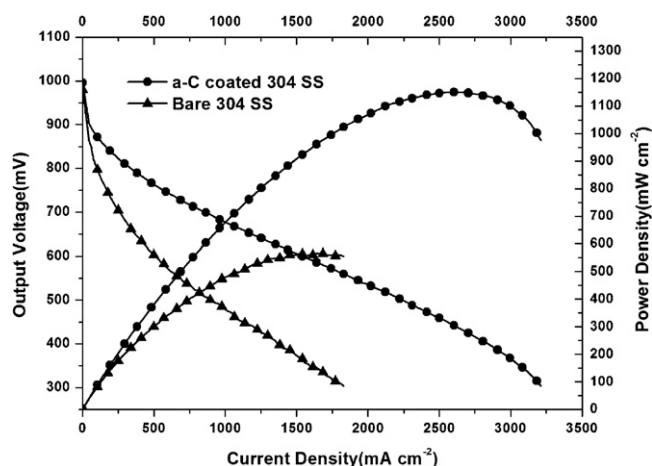
Fig. 5. Initial performance of the single cells with a-C coated and bare 304SS bipolar plates.

Fig. 6. Lifetime test of the single cells with a-C coated and bare 304SS bipolar plates at a cell voltage of 0.6 V during 200 h of continuous operation.

3.2. Performance of single cell

Fig. 5 characterizes the initial performance of the single cells assembled with a-C coated and bare 304SS bipolar plates, respectively. The open circuit voltage (OCV) of the single cell with a-C coated 304SS bipolar plates is 1.01 V which is almost the same as that with bare 304SS bipolar plates. The peak power density of the single cell assembled with a-C coated 304SS bipolar plates is 1150.6 mW cm^{-2} at a current density of 2573.2 mA cm^{-2}, while the power density is 923.8 mW cm^{-2} at 0.6 V. The peak power density of the single cell assembled with bare 304SS bipolar plates is 562.6 mW cm^{-2} and the power density at 0.6 V is 306.3 mW cm^{-2}. It is obvious that the cell performance is greatly improved by the a-C coating on 304SS bipolar plates.

The performance of PEMFCs is affected by resistance over-potential, activation over-potential, and concentration over-potential. In this study, resistance over-potential is the major cause that the single cell performance with bare 304SS bipolar plates is significantly lower than that with a-C coated bipolar plates because assembling conditions and testing conditions are the same. Furthermore, the ICR plays a dominant role in resistance over-potential since the membrane resistance is identical due to the same operation conditions [23] and in-plane conductivity of bipolar plates is similar as discussed above. With an increase in current density, the output voltage of the single cell with bare 304SS bipolar plates decreases faster than the output voltage of the single cell with a-C coated 304SS. This trend occurs because the ICR of bare 304SS bipolar plates is much higher than a-C coated 304SS bipolar plates as shown in Fig. 3, hence the resistance over-potential is higher.

3.3. Lifetime analysis of single cell

In developing metallic bipolar plates, lifetime extension is another challenge in addition to the improvement of initial performance since metal ions would be liberated to contaminate Pt catalysts and proton-conductive membrane if corrosion of the bipolar plates occurs. To evaluate the lifetime performance, the single cell was operated at a constant cell voltage of 0.6 V and the current density was recorded as a function of operation time. Fig. 6 compares the performance of the single cells with a-C coated and bare 304SS bipolar plates during 200 h of continuous operation.

To characterize the performance degradation, least squares regression is used to obtain the lines which are the best fit to the experimental data. The current density decay of the single cell with a-C coated bipolar plates can be described by:
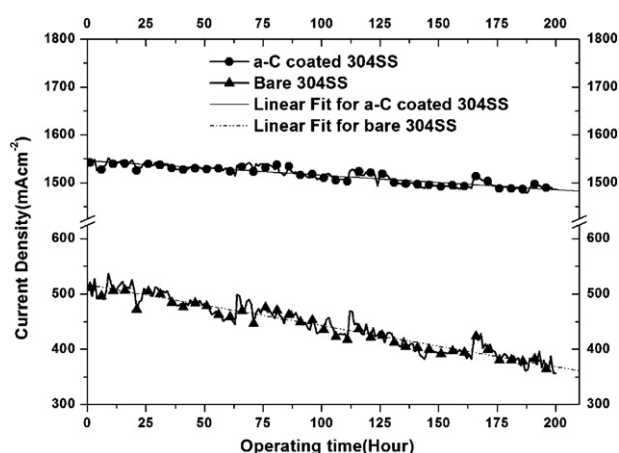
$$\rho_A(t) = 1545.96 - 0.30t \tag{1}$$

where ρ_A is the current density (mA cm^{-2}) and t is the operating time (h).

From (1), the performance degradation (PD) of the single cell using a-C coated 304SS bipolar plates after 200 h of continuous operation can be defined as:

$$PD = \frac{\rho_A(0) - \rho_A(200)}{\rho_A(0)} = 3.9\% \tag{2}$$

For the single cell using bare 304SS bipolar plates, the current density decay and the PD can be expressed in Eqs. (3) and (4), respectively.

$$\rho'_A(t) = 516.89 - 0.74t \tag{3}$$

$$PD = \frac{\rho'_A(0) - \rho'_A(200)}{\rho'_A(0)} = 28.7\% \tag{4}$$

Chung et al. [5] investigated the performance degradation of a single cell with bare 304SS bipolar plates and found that the PD was 16% after 100 h of continuous operation at 0.6 V and 40 °C. Besides, Cho et al. [22] tested a single cell with bare AISI316 bipolar plates and showed that the PD was 50.4% after 200 h of continuous operation at 0.6 V and 80 °C. Although the testing conditions are not exactly identical, a similar result is still found in this study that the performance of the single cell with bare 304SS bipolar plates degrades very rapidly. That is because corrosion of bare 304SS bipolar plates occurs in the acidic PEMFCs environment and causes MEA contamination. This MEA contamination leads to a decrease in ionic conductivity of the electrolyte membrane and an increase in charge transfer resistance by poisoning of the catalytic layers [24].

The lifetime performance of the single cell is greatly improved by a-C coating. However, performance degradation of 3.9% after 200 h of continuous operation is unacceptable for commercialization of fuel cell according to the DOE target [20]. It should be pointed out that the cause of performance degradation is not only due to bipolar plate corrosion but also thanks to the degradation of MEA itself including membrane, catalyst layer and GDL layer degradation [25,26]. Therefore, it is necessary to further investigate how much bipolar plate corrosion contributes to the cell performance degradation by analyzing the concentration of metal elements in fresh and tested MEAs.

Contamination of the MEA was investigated using ICP-MS analysis and the results are presented in Table 3 for the major elements

Table 3
Concentration of the metallic elements observed in the fresh and tested MEAs (wt.%).

Element	Contamination		
	Fresh MEA	a-C coated 304SS	Bare 304SS
Fe	0.000822649	0.0104142	0.918472
Cr	0.000876068	0.0028516	0.340177
Ni	0.000172721	0.0006946	0.106531

of 304SS (Fe, Cr, Ni). It is obvious that dissolution of Fe is dominant because it accounts for the largest weight percentage of 304SS. The concentration of the metallic elements in the fresh MEA is very low and can be neglected. The MEA assembled with bare 304SS bipolar plates has a significantly higher concentration of metallic elements than the MEA assembled with a-C coated 304SS bipolar plates. Hence, a-C coating protects 304SS bipolar plates well and reduces MEA contamination greatly.

3.4. Performance of 100 W-class PEMFC short stack

Material properties and single cell performance have revealed a potential of the a-C coated 304SS bipolar plates. Based on the good results above, a 100 W-class PEMFC short stack using a-C coated 304SS bipolar plates was assembled and evaluated. As shown in Fig. 7, the OCV of the short stack is 3.03 V. With lowering the voltage to 2.1 V, 1.8 V, and 1.5 V, the current is increased to 31.3 A, 58.1 A and 82.0 A, respectively. The peak output power is 124.8 W at the current of 83.4 A. Since the active area for each MEA is 40 cm^2, the peak power density is 1040.1 mW cm^{-2} at a current density of 2085.9 mA cm^{-2} and output power density at a voltage of 1.8 V is 873.3 mW cm^{-2}. In contrast, the peak output power for the 3-cell short stack with bare 304SS bipolar plates is 59.6 W and the output power density at 1.8 V is 263.2 mW cm^{-2}.

In a fuel cell stack, uniform performance of each unit cell is preferable so that voltage distribution was monitored to assess the uniformity of stack. The histogram in Fig. 8 shows the voltage distribution of each unit cell in the 100 W-class PEMFC short stack when the stack was operated at OCV, 2.1 V, 1.8 V and 1.5 V. The performance of each unit cell in this short stack is considerable uniform and the difference between the maximum and the minimum voltages of the single cells among the stack is 23 mV at 1.8 V [27].

The 100 W-class PEMFC short stack was operated for 48 h at a constant voltage of 1.8 V and the current was recorded for short-term lifetime analysis. As shown in Fig. 9, obvious performance degradation is not observed in this study but there is a slight perfor-
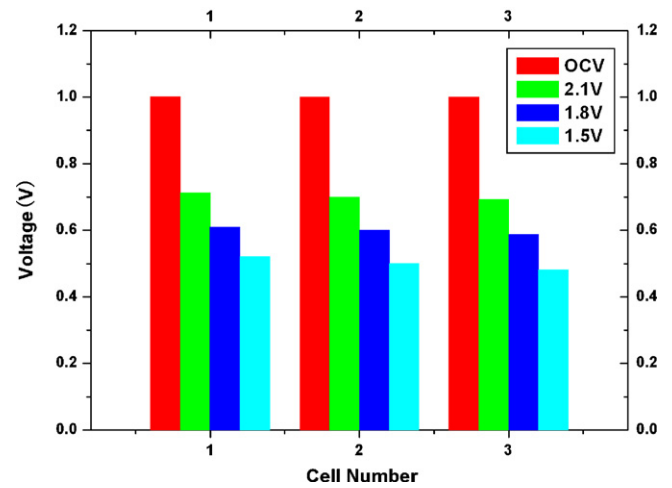

Fig. 8. Voltage distribution in the 100 W-class PEMFC short stack using a-C coated 304SS bipolar plates.

mance fluctuation. That is because cell temperature was controlled by adjusting coolant water temperature and flow rate. It is not sensitive enough to change the temperature of coolant water due to its high heat capacity and flow rate cannot be controlled very precisely.

High volumetric power density and specific power are needed for PEMFCs to compete with traditional energy conversion devices. Volumetric power density and specific power of the 100 W-class short stack are analyzed using a repeating MEA-seal-bipolar plate-seal unit which is 18.788 g in weight and 1.368 mm in thickness after assembly. As shown in Fig. 10, the peak volumetric power density and specific power are 3040.9 W L^{-1} and 2214.2 W kg^{-1}, respectively. It should be pointed out that the volumetric power density and specific power would be lower when considering the contributions of the end plates and bolts. However, this effect can be compensated by increasing the number of cells and enlarging the flow field area since the effective flow field area in this study is only 40 cm^2 on 100 cm^2 bipolar plates. As a result, a-C coated 304SS bipolar plate fabricated by flexible forming process might be a feasible approach to meet the DOE target that the stack volumetric power density is 2000 W L^{-1} and the stack specific power is 2000 W kg^{-1} for automotive fuel cell power systems operating on direct hydrogen [20].
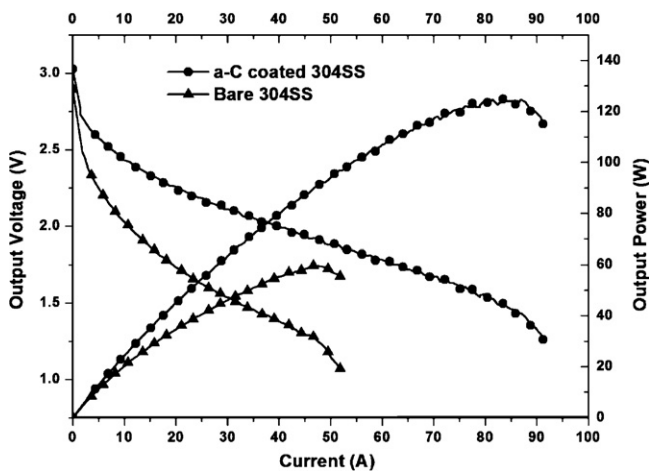

Fig. 7. Polarization curves of the short stacks with a-C coated and bare bipolar plates.
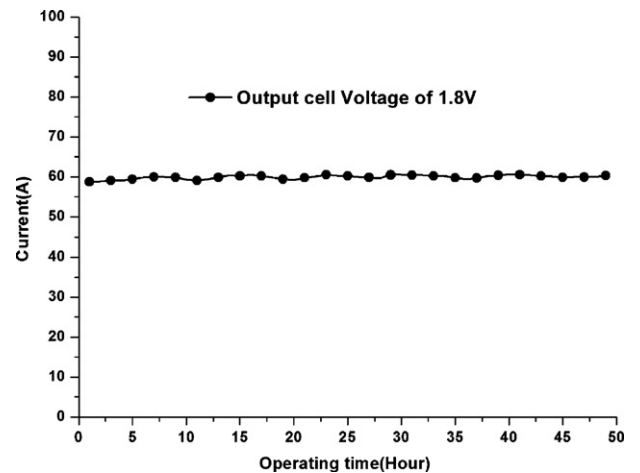

Fig. 9. Short-term lifetime test of the 100 W-class short stack with a-C coated 304SS bipolar plates at a voltage of 1.8 V during 48 h of continuous operation.
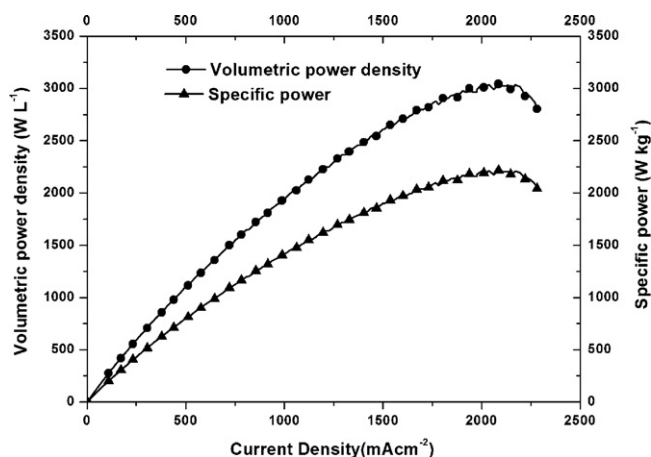
Fig. 10. Volumetric power density and specific power of the 100 W-class short stack with a-C coated 304SS bipolar plates.

4. Conclusion

Low-cost 304SS foil was fabricated to bipolar plates for PEMFCs by flexible forming process and an amorphous carbon (a-C) film was coated by CFUBMSIP. The in-plane conductivity of the a-C coated 304SS is slightly increased and contact resistance is remarkably reduced to $5.4\,m\Omega\,cm^{-2}$ which meets the DOE target of $10\,m\Omega\,cm^{-2}$ at a compaction pressure of 1.5 MPa. The surface tension of a-C coated 304SS is also higher than that of bare 304SS. Increasing the surface energy further without contact and anti-corrosion performance loss remains the focus of ongoing efforts and future work.

Single cell test results show that the peak power density is $1150.6\,mW\,cm^{-2}$ and the output power density is $923.9\,mW\,cm^{-2}$ at a cell voltage of 0.6 V. Performance comparison experiments show that the power density of the single cell with a-C coated 304SS bipolar plates is 3.02 times higher than the power density of the single cell with bare 304SS bipolar plates at 0.6 V. The lifetime test results indicate that the performance degradation over 200 h of continuous operation is decreased from 28.7% to 3.9% by the a-C coating. The contamination analysis results of the tested MEAs reveal that the chemical stability of 304SS bipolar plates is greatly improved by the a-C coating.

The peak power density of a short stack assembled with a-C coated bipolar plates is $1040.1\,mW\,cm^{-2}$ at a current density of $2085.9\,mA\,cm^{-2}$ and output power density is $873.3\,mW\,cm^{-2}$ at 1.8 V. The stack also exhibits a uniform voltage distribution and good short-term lifetime performance over 48 h of continuous operation. The peak volumetric power density and specific power of a repeating MEA-seal-bipolar plate-seal unit are $3040.9\,W\,L^{-1}$ and $2214.2\,W\,kg^{-1}$. Overall, a-C coated 304SS fabricated by mass-production forming process is a promising roadmap for PEMFCs commercialization development.

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